Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Quality Assurance Project Plan

Title: Collection and Analysis of Water from Innovative/Alternative Onsite Wastewater Treatment Systems in the Shubael Pond Area of Barnstable, Massachusetts

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QUALITY ASSURANCE PROJECT PLAN
FOR
COLLECTION AND ANALYSIS OF WATER FROM INNOVATIVE/ALTERNATIVE ONSITE WASTEWATER TREATMENT SYSTEMS IN THE SHUBAEL POND AREA OF BARNSTABLE, MASSACHUSETTS

MASSTC Document ID#: MASSTC-QAP-003, Revision #000

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1. Project Management

1.1 Title and Approval Page (EPA QA/R-5 A1)

See page 1.

1.2 Table of Contents (EPA QA/R-5 A2)

See pages 2-4.

1.3 Distribution List (EPA QA/R-5 A3)

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1.4 Project Organization (EPA QA/R-5 A4)

The responsible agency for this program is the Massachusetts Alternative Septic System Test Center (MASSTC). The participating agency is the U.S. Environmental Protection Agency (USEPA). Barnstable County Water Quality Laboratory is the Massachusetts state certified laboratory that will be performing the chemical and microbiological analyses for the monitoring program following commonly used analytical methods.

The roles and responsibilities of those involved in the implementation of the surface water monitoring program are described below. An organization chart for the project is shown in Figure 1: Project organization chart.

Project Manager is the responsible official for this project overseeing the overall project and budget, as well as tasking contractors and staff with work required to complete this project.

Project Assistant is responsible for assisting in the completion of project milestones as directed by the Project Manager. They will be responsible for sampling, field measurements, and recording of data. They may also supervise subordinate staff and subcontractors in completion of project tasks.

Quality Assurance Manager (QAM) is responsible for reviewing and approving the Quality Assurance Project Plan (QAPP). They may provide technical input on proposed sampling design, analytical methodologies, and data review. They may assist with coordinating laboratory services. The QAM is also responsible for transferring and reviewing all data prior to its inclusion into the project datasets. This includes the acquisition of laboratory reports, inspection of data derived from laboratory notes, downloading and inspection of data from the field instrumentation and assisting in the correction of conditions that prevent quality data from being attained and used toward project objectives. The QAM is responsible for writing, reviewing, and making recommendations to the Project Manager in relation to changes in Standard Operating Procedures as they apply to the maintenance of quality data.

Barnstable County Laboratory Director is responsible for oversight of all analytical laboratory operations including review and integration of all data originating from the occasional subcontracting of assays through a non-county laboratory.

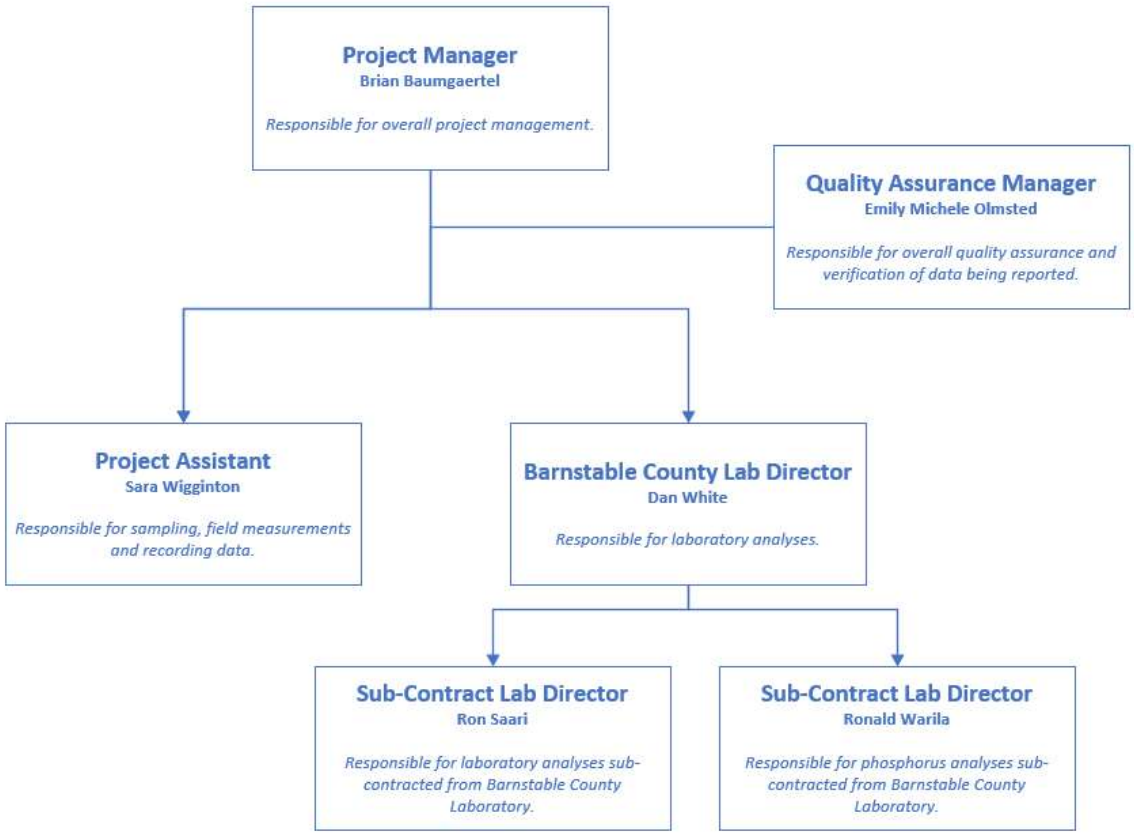


Figure 1: Project organization chart.

1.5 Problem Definition/Background (EPA QA/R-5 A5)

Onsite wastewater treatment systems (OWTS; i.e. septic systems) are a major contributor of nitrogen to waterways in areas, such as Barnstable County, MA, where most residences rely on OWTS to treat and disperse wastewater. Conventional OWTS do not remove adequate nitrogen from wastewater before it is recharged to groundwater where it then flows to surrounding sensitive ecosystems like estuaries and freshwater ponds. Excess nitrogen in waterways lead to eutrophication and toxic algal blooms which can cause fish kills and beach/pond closures.

Shubael Pond, located in the Three Bays watershed in Massachusetts, has been experiencing repeated annual toxic algal blooms and pond closures. Preliminary groundwater monitoring performed by the United States Geological Survey (USGS) indicate that nitrogen loading from onsite septic systems is likely fueling these algal blooms. The project endeavors to reduce the nitrogen loading to the watershed by employing the use of various alternative onsite septic system technologies with efficacy for removing nitrogen from wastewater, confirmed through testing by MASSTC.

1.6 Project/Task Description and Schedule

The Project involves the sampling of up to 20 alternative onsite septic systems in a neighborhood near Shubael Pond, to be located based on homeowner preference and suitability of the site as determined by the design engineer. The goal is to determine the efficacy of these technologies for reducing nitrogen inputs from wastewater to the Three Bays watershed. Additionally, this project will serve as a pilot to inform whether nitrogen removing technologies, installed in neighborhood clusters, can improve water quality on a larger scale.

During the first year of the project, participants will be identified, and nitrogen-reducing technologies will be installed at up to 20 households. These installations will likely not coincide and will instead be distributed over several months. Two months after the first system has been installed, monthly sampling will begin and continue for 36 consecutive months. Each system will be sampled monthly for 36 months with system inspections being performed quarterly approximately two weeks after each sampling event. Each individual OWTS will be sampled at two to three locations: An "I/A influent" sample taken at the outlet of the septic tank (referred to as "influent"), an "I/A effluent" sample taken at the outlet of the I/A Technology (referred to as "effluent"), and in certain cases, a "soil absorption system effluent" taken using a pan lysimeter (referred to as "lysimeter").

Data synthesis and interpretation will be ongoing, but final analyses of data and write-up of results will occur in the final three months of the project.

MONITORING PARAMETERS

Monitoring parameters are detailed in Table 1. Monthly sampling will measure nitrate (NO_3^-), nitrite (NO_2^-), total Kjeldahl nitrogen (TKN), and total phosphorus (TP) at each of the two or three locations referenced above. In addition to monthly nutrient sampling, selected chemical parameters will be collected quarterly at all locations coincident with nutrient samples; these parameters include ammonia (NH_4^+), total suspended solids (TSS), alkalinity, and biochemical oxygen demand (BOD). During each inspection, the following field parameters will be collected at each sampling location: temperature, pH, conductivity, turbidity, and dissolved oxygen (DO). All methodology for analyses is presented in Appendix 1.

Table 1: Monitoring Parameters for the Shubael Pond Project

Variable	Method	Units	Preservation	Collection	Holding Time	Laboratory	Precision	Minimum Detection Limit	Minimum Reportable Limit
Alkalinity	SM 2320-B	mg/L as $CaCO_3$	Ice (<6 °C)	250-mL plastic	14 days	BCDHE	1.8% RSD	0.95 mg/L	2.0 mg/L
Ammonia	EPA 350.1	mg/L	Ice (<6 °C) H2SO4	250-mL plastic	28 days	BCDHE	0.60% RSD	0.15 mg/L	0.25 mg/L
BOD or cBOD	SM 5210 B	mg/L	Ice	1000-mL plastic	48 hours	Envirotech	See Appendix 1	See Appendix 1	See Appendix 1
Dissolved Oxygen	SM 4500-O H	mg/L	None	Grab (ProDSS Meter)	N/A	Field	±1%	N/A	N/A
Nitrate	EPA 300.0	mg/L	Ice	250-mL plastic	48 hours	BCDHE	0.033% RSD	0.019 mg/L	0.10 mg/L
Nitrite	EPA 300.0	mg/L	Ice	250-mL plastic	48 hours	BCDHE	0.037% RSD	0.005 mg/L	0.05 mg/L
pH	SM 4500-H^+	Standard Units	None	Grab (ProDSS Meter)	N/A	Field	±0.1 units	N/A	N/A
Specific Conductance	SM 2510	µS/cm	None	Grab (ProDSS Meter)	N/A	Field	±1%	N/A	N/A
Temperature	SM 2550	°C	None	Grab (ProDSS Meter)	N/A	Field	±0.1°C	N/A	N/A
Total Kjeldahl Nitrogen	EPA 350.2	mg/L	Ice (<6 °C) H2SO4	250-mL plastic	28 days	BCDHE	1.9% RSD	0.10 mg/L	0.25 mg/L
Total Phosphorus	EPA 365.1	mg/L	Ice	250-mL plastic	28 days	Microbac		0.01 mg/L	0.01 mg/L
Total Suspended Solids	SM 2540 D	mg/L	Ice (<4 °C)	250-mL plastic	7 days	BCDHE	See Appendix 1	See Appendix 1	See Appendix 1
Turbidity	ISO 7027	FNU	None	Grab (ProDSS Meter)	N/A	Field	±2%	N/A	N/A

RSD – Relative Standard Deviation

1.7 Quality Objectives and Criteria for Measurement Data (EPA QA/R-5 A7)

The general quality objectives of MASSTC for any study include the production of quality data for the assessment of wastewater treatment technologies for the range of contaminants present. This includes the use of approved methods of analysis, the integrity of the chain of custody, a continuous assessment of the quality of the data produced, the maintenance of accurate records, and the continuous vigilance to adherence of standard operating procedures (SOP) and principles of good test center and laboratory practice. High quality data is the goal of all sample collection, field analysis, and laboratory analysis procedures. Specific data quality objectives have been set for analytical procedures on a method basis for method detection limits (MDL), precision, accuracy, comparability, and completeness.

1.7.1 Objectives and Project Decisions

The objective of this project is to determine the N removal performance of up to 20 innovative/alternative OWTS located in a watershed experiencing eutrophication. The results will help determine if nitrogen-reducing OWTS are a lower cost alternative to sewering or an effective stopgap while sewers slowly expand. Nitrogen removal performance will be expressed as percent removal of total nitrogen (TN) using the following formula:

$$\%\, TN\ removal = \left(\frac{Effluent\ TN\ Concentration}{Influent\ TN\ Concentration}\right) \times 100$$

The TN concentration will be calculated using the following formula:

$$TN = NO_3^- + NO_2^- + TKN$$

As referenced above, this study involves collection of monthly influent and treated effluent samples from various nitrogen-reducing OWTS. These samples will be collected by the Project Manager and/or Project Assistant and transported to the Barnstable County Laboratory for analysis of NO_3^-, NO_2^-, TKN, NH_4^+, TP, BOD, TSS, and alkalinity. During each inspection pH, conductivity, turbidity, and DO measurements will be collected by the Project Manager and/or Project Assistant using properly calibrated field instruments.

- If total nitrogen exceeds the concentration set out in the technology approval, then the manufacture will be notified.
- If percent removal is determined to be <75%, then the manufacturer will be notified.
- If total nitrogen values exceed those set out in the technology approval for five or more consecutive sampling events, then daily nitrogen load will be calculated using flow data and total annual loading will be compared to other systems to determine if low water use is contributing to high concentrations.

1.7.2 Action Limits/Levels

Due to the exploratory nature of this project and because these system upgrades and replacements were not mandated by any entity, there are no project action limits and no regulatory action limits. If nitrogen levels exceed those set out in the technology approval, the manufacturer will be notified. Table

1 provides a list of the parameters of interest and their associated precision, detection limits, and reporting limits.

1.7.3 Measurement Performance Criteria/Acceptance Criteria

To determine and define acceptable data quality for this project we will use data quality indicators. Data quality indicators will include precision, data representativeness, data comparability, and data completeness. The general approach to assessing these data quality indicators is detailed below. Some of the data quality indicators will be assessed quantitatively while others will be qualitative. Quantitative data quality indicators are detailed in Table 1.

1.7.3.1 General Approach

Precision

Precision is the degree of agreement among repeated measurements of the same characteristic, or parameter, and gives information about the consistency of methods. In context of this project, precision of measurements is a prime objective. It will be evaluated by comparison with laboratory precision objectives referenced in Table 1. Laboratory methods for measurement of precision are included in respective Standard Operating Procedures.

Data Representativeness

Data Representativeness is the extent to which measurements actually represent the true environmental condition. It is the degree to which data from the project accurately represent a particular technology being tested. Representativeness of samples is ensured by adherence to standard field sampling, measurement and laboratory protocols. The design of the sampling scheme and number of samples for this project provide representativeness of the performance of the technology being monitored. As a whole, representativeness of the data will be determined during data assessment and data interpretation phase.

Data Comparability

Data comparability is the degree to which data can be compared directly to similar studies. Comparability is ensured by using standardized sampling protocols and the same or comparable analytical methods and reporting with comparable sensitivity. Protocols used in this project are similar to those used in many other pilot projects and approval procedures for alternative septic system treatment performance.

Data Completeness

Data completeness is the comparison between the amounts of usable data collected versus the amount of data called for in the sampling plan. Completeness is the percentage of valid results obtained compared to the total number of samples taken for a parameter. The goal of this project is to meet or exceed 90% usable data.

Sensitivity

Sensitivity refers to the method minimum detection limits and minimum reporting limits. These limits

are defined in Table 1 and, for laboratory samples, are determined by the laboratory analyzing each parameter. For field measurements, sensitivity is determined by the instrument manufacturer.

1.8 Special Training Requirements/Certification (EPA QA/R-5 A8)

The Barnstable County Laboratory is certified by the Commonwealth of Massachusetts Department of Environmental Protection (Mass DEP) for analytes listed in Table 1. The Project Manager of this project has a Massachusetts Wastewater Operator's License of Grade 4. All technical assistants will be required to complete the employee training checklist under direct supervision of the Project Manager (MASSTC-FRM-011 – Training Log, Appendix 2).

For each task relating to the collection and processing of sampling and the collection and recording of field (in situ) data, trainees will accompany the trainer personnel during a normal task-run. The trainer personnel will then observe the trainee during independent completion of a normal task-run to verify that all tasks have been performed correctly. The trainer personnel will then sign the training checklist record to verify that the trained personnel are able to perform the task correctly. Quarterly field audits will be conducted to verify proper taking and handling of samples as well as use of field instruments. All training records and audits will be maintained and included in appendices of the final report.

1.9 Documents and Records (EPA QA/R-5 A9)

MASSTC employs a Document Management System compliant with ISO/IEC 17025 (2017) to ensure that appropriate staff have access to quality management documents where and when they need it and to ensure that no unauthorized or unrecorded changes are made to the contents of documents under control (MASSTC-SOP-001 – Document Control Procedure, Appendix 3). Documents under this system include forms and datasheets used to collect critical data and information on testing. A list of documents under the Controlled Document System (as of November 2021) is provided in Appendix C. The Director of MASSTC is the only person allowed to approve and make changes to any document in this control system and all previous versions of any document are maintained indefinitely in the event of a need for future reference.

For procedures relating to the maintenance of records and archives see Appendix 4 – MASSTC-SOP-003 – Data and Records Management. Upon approval, this QAPP will be entered into the document control system as MASSTC-QAP-003 and all project participants will be supplied copies and be required to sign off as having read and be willing to adhere to it.

1.9.1 QA Project Plan Distribution

The Shubael Pond Project Manager/QAM will be responsible for preparing and maintaining amended versions of the QAPP and for distributing amended QAPPs to the individuals listed in the Distribution List (Section 1.3 of this document).

1.9.2 Field Documentation and Records

Field records will be documented in field logbooks, chain of custody forms, site maps, and preprinted labels. Field notebooks will be used to record field measurements, site conditions, and other on-site observations. Field books will be kept in a permanent file at MASSTC and will include sampling

information such as sampling date/time, sample location, sampler's name(s), field measurement instrument readings, any deviation from QAPP or SOPs, arrival/departure times, other personnel on site, and other field observations (weather, odors, system alarms, etc.).

All samples will be labeled in a clear and precise way for identification in the field and for identification by laboratory personnel. Sample labels will contain unique ID numbers, a site-specific ID, sampling location (i.e. "I/A influent", "I/A effluent", or "Lysimeter"), sample description (i.e. grab or composite), time/date of collection, analytical parameters to be measured, and method of preservation.

Chain of custody forms will be printed ahead of sampling, taken to the field, and provided to the laboratory. Chain of custody forms will be used to document collection and shipping of samples to the BCDHE Laboratory. Each site will have a chain of custody form, even if samples are collected on the same day. If multiple coolers are used to transport samples, each cooler will have a chain of custody specific to the samples within that cooler. The original form will be sent to the BCDHE Laboratory and copies will be retained by the Shubael Pond Project Manager/QAM.

All field activities will be conducted according to the appropriate SOPs (Appendix 1). Samples will be collected in accordance with MASSTC-SOP-037 – Sample Collection (Appendix 5) and MASSTC-FRM-040 – Sampling Plan (Appendix 6) will be completed during each sampling event. The Shubael Pond Project Manager/QAM are responsible for maintaining updated SOPs and to distribute updated SOPs to sampling personnel. All documentation generated during field sampling will be kept in a permanent file at MASSTC.

1.9.3 Laboratory Documentation and Records

The Barnstable County Department of Health and Environment Laboratory will keep a log of samples received and all chain of custody forms submitted for this project. The BCDHE Laboratory will also keep a record of all analysis performed and all associated quality control information. Data from analyses performed at or by MASSTC are directly accessed weekly from the Laboratory Information Management System (LIMS) of BCDHE Laboratory. Data from BCDHE Laboratory are stored and maintained as described in the QAP for the laboratory (Appendix 7). All data from analyses on this project will be directly accessed from the laboratory information management system (LIMS) by the Quality Assurance Manager. These data are then stored in a Structured Query Language (SQL) file where they are manipulated to produce various internally developed outputs. Data are organized in the MASSTC Database by project to assist in graphing and charting and are capable of being exported as files compatible with Microsoft Excel™ and other character delimited files. If, during the access and integration process errors in the data are suspected, the Laboratory Director is contacted and asked to verify the data. Scanned copies of all laboratory reports are maintained in an organized database on the MASSTC server and are accessible by all staff. If upon inspection of the data by any staff working on a particular project there appears to be an error, the Laboratory Director is notified for comment and resolution.

1.9.4 Quarterly and/or Final Reports

The Shubael Pond Project Manager and Project Assistant are responsible for the preparation of quarterly and final reports which will also be reviewed by the QAM. Quarterly reports will be submitted within 30 days of the end of each quarter (March, June, September, December) and a final report will be

submitted within 60 days of completing the final sampling event. All quarterly and final reports will be submitted to the US EPA Grants Project Officer.

The quarterly reports shall include, at a minimum, a table summarizing results for field measurements and laboratory data, laboratory reports, a discussion of field activities, discussion of any issues with laboratory or field data, discussion of any data points exceeding those specified in the technology approval, and recommendations for the next sampling event(s).

The final report should include, at a minimum, a description of the project, a summary of all results and any trends observed during monitoring, a description of all field and laboratory activities, a description of any deviations or modifications from this document, copies of all documentation discussed in the above sections, an evaluation of the data in meeting the project objectives, and any recommendations for future project activities.

2 Data Generation and Acquisition

2.1 Sampling Design (EPA QA/R-5 B1)

A total of up to 20 Innovative/Alternative Onsite Wastewater Treatment Systems (I/A OWTS) will be sampled over the course of three years for this project. All OWTS are located in the vicinity of Shubael Pond in the town of Barnstable, Massachusetts.

Each individual OWTS will be sampled at two to three locations: An “I/A influent” sample taken at the outlet of the septic tank (referred to as “influent”), an “I/A effluent” sample taken at the outlet of the I/A Technology (referred to as “effluent”), and in certain cases, a “soil absorption system effluent” taken using a pan lysimeter (referred to as “lysimeter”).

See Table 2 for a listing of analytical parameters and their sampling frequency. Field measurements for pH, Dissolved Oxygen, temperature, and specific conductance will also be taken at each sampling location at the time of sampling.

Table 2: Sampling frequency and sample totals

Parameter	Maximum Number of Sites	Number of Locations[1]	Number of Samples/Year	Number of Years	Total Samples without Blanks or Duplicates		Total Number of Field Blanks[2]	Field Duplicates[3]	Total Samples
Nitrate	20	2-3	12	3	1,440 – 2,160		144	72 – 108	1,656 – 2,412
Nitrite	20	2-3	12	3	1,440 – 2,160		144	72 – 108	1,656 – 2,412
Total Kjeldahl Nitrogen	20	2-3	12	3	1,440 – 2,160		144	72 – 108	1,656 – 2,412
Total Phosphorus	20	2	12	3	1,440		144	72	1,656
Total Suspended Solids	20	2	4	3	480		48	24	552
Ammonia	20	2	4	3	480		48	24	552
Biochemical Oxygen Demand	20	2	4	3	480		48	24	552
Alkalinity	20	2	4	3	480		48	24	552
Total Samples					7,680 – 9,840		768	492	8,832 – 11,100

[1]Systems with new soil treatment areas will be instrumented with a pan lysimeter and have three sampling locations for most parameters.
[2]Field blanks collected during each sampling trip with five sites assumed for each sampling trip, yielding four blanks each month that all 20 sites are sampled
[3]Field duplicates collected at a 5% rate (every 20 samples)

2.2 Sampling Methods (EPA QA/R-5 B2)

Sampling methods for OWTS included in this study will be grab samples for influent and effluent locations. Sampling beneath soil treatment areas is accomplished by means of pan lysimeters placed beneath the areas as illustrated in Figure 2. See Appendix 5 – MASSTC-SOP-037 – Sample Collection.

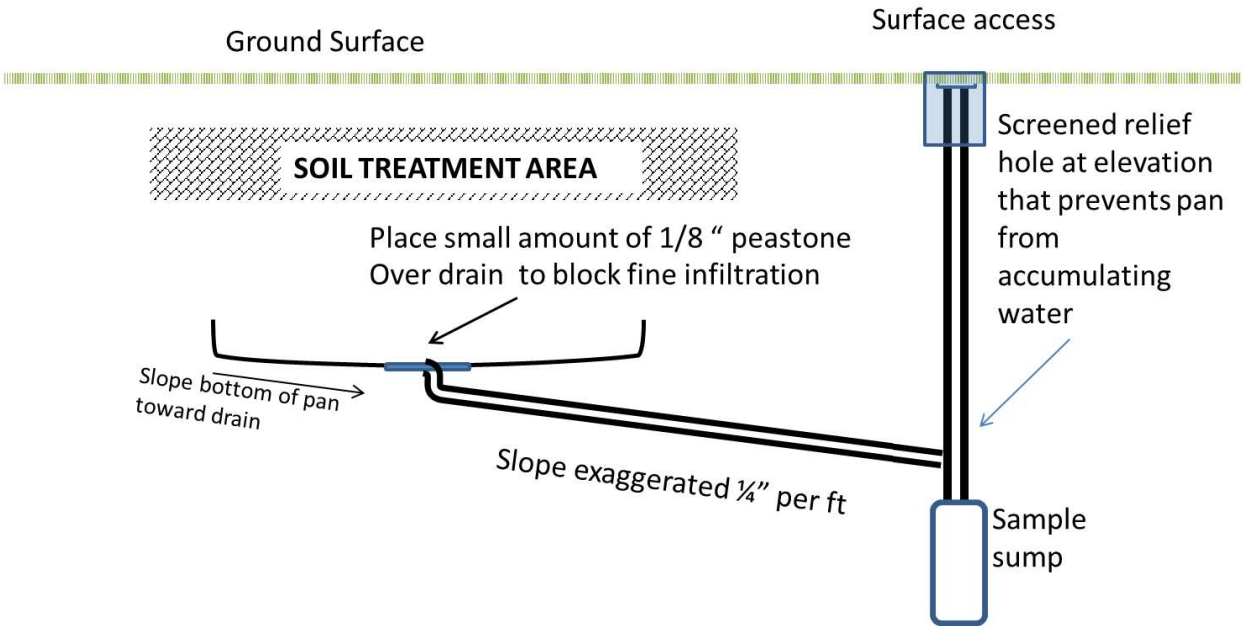


Figure 2: Schematic of a typical pan lysimeter placed beneath a soil treatment area for the purpose of collecting percolating wastewater. The relief hole allows for a constant refreshing of sample and is hence a type of composite sample that integrates the quality of the percolate over time.

When sampling from pan lysimeters, the collection of field parameters is accomplished by lowering the ProDSS™ field meter probe into the sampling sump. Following the collection of readings and withdrawal and rinsing of the probe with clean tap water, samples for laboratory analyses are withdrawn using either a 12-volt peristaltic (fashioned from the sampling module of an ISCO™ sampler) or a 12-volt submersible pump (Whale® whalepumps.com). Sample pumps are flushed with tap water and drained between samples. Subsequent samples drawn follow a flushing of the sample tubing with the liquid to be sampled.

Sampling at residential site septic tanks or pump chambers for purpose of determining influent contaminant levels are retrieved using the above referenced pumps.

2.3 Sample Handing and Custody (EPA QA/R-5 B3)

Sample preparation and transportation is described in MASSTC-SOP-015 – Sample Preparation and Transportation (Appendix 8). All samples collected are initially stored in a cooler on ice prior to transport to the primary certified laboratory at Barnstable County Department of Health and Environment. Chain of Custody Procedures and handling of samples at the laboratory are described in the Quality Assurance Plan of the laboratory provided in Appendix 7.

2.4 Analytical Methods (EPA QA/R-5 B4)

Most analyses are performed at the Barnstable County Department of Health and Environment Laboratory. Both MASSTC and the Barnstable County Department of Health and Environment Laboratory are divisions of the Barnstable County Department of Health and Environment. The analytical methods performed in the Barnstable County Department of Health and Environment Laboratory as well as the descriptions of the remaining Section 2 requirements are outlined in Appendix B titled "Quality Assurance Plan – Revision 26 – Barnstable County Department of Health and Environment" dated 29 December 2020. On occasion, selected analytes are assayed by Envirotech Laboratory (Certification Number M-MA063) located on Jan Sebastian Drive in Sandwich, MA and Microbac Laboratories located on Flanders Road in Westborough, MA. See Table 1 for a list of the analytes covered by this QAPP.

Tests conducted on samples collected at field sites include pH, dissolved oxygen, tubidity, specific conductance, and temperature using a YSI ProDSS™ field meter. Standard Operating Procedures for all analytes are presented in Appendix 1.

2.4.1 Field Measurements Methods

Field measurements that will be taken concurrently with all samples include pH, dissolved oxygen, temperature, turbidity, and specific conductance. Field measurements will be taken using a YSI ProDSS field meter using MASSTC-SOP-16 (Appendix 1) which also describes calibration and acceptance procedures.

2.4.2 Field Analysis Methods

Field analyses are not included as a part of this project.

2.4.3 Laboratory Analyses Methods

All chemical analyses will be performed at the Barnstable County Department of Health and Environment Laboratory using standard or EPA methods referenced in Table 1. SOPs for all chemical analytes for this study are presented in Appendix 1. These methods and SOP prescribe all aspects of the quality control including calibrations, QC acceptance criteria, corrective actions, etc.

2.5 Quality Control Requirements (EPA QA/R-5 B5)

2.5.1 Field Sampling Quality Control

To ensure quality control over field sampling activities two types of QC samples will be collected: field duplicates and field blanks.

Field duplicates will be collected each 20 samples, at a rate of 5%. Field duplicate samples will be homogenized in a single container and aliquoted to two sample bottles. Duplicates will be submitted blind with a numerical sample ID like all other samples so that they are not discernable as duplicates to laboratory personnel. Variance between field duplicates shall not exceed 20%. If this criterion is exceeded, field sampling and handling procedures will be evaluated and the laboratory running analyses will be informed. Project members will then work with the laboratory director to take appropriate

action to correct problems, such as increased training, instrument calibration, greater attention to detail, etc.

Field blanks will be collected for each analyte in Table 2 during each sampling event to evaluate whether contaminants have been introduced during collection or from sample bottles themselves. Field blank samples will be collected by pouring deionized or ultra-pure water into sample containers on the day of sampling. Field blanks will be preserved and handled in the same manner as all other samples. Field blanks will be submitted blind with a numerical sample ID like all other samples so that they are not discernable as blanks to laboratory personnel. If target analytes are found in field blanks over the method detection limit, sampling and handling procedures will be evaluated and corrective action will be taken. This may include communicating with laboratory personnel about potential contamination, increased training measures for laboratory or sampling personnel, investigating sample containers for possible contaminants, finding another source for sample containers, or increasing attention to detail during sampling.

2.5.2 Field Measurement/Analysis Quality Control

Continuing Calibration Verification (CCV) is performed every ten samples per Section 10.12 of MASSTC-SOP-016 – YSI ProDSS Field Meter (Appendix 1). This CCV is part of the permanent record of use and is verified daily by the MASSTC Operator upon the meter closeout procedure of instrument data download (Section 11.9 MASSTC-SOP-016 – YSI ProDSS Field Meter). Corrective actions and other aspects of calibration and meter use are contained in the SOP.

2.5.3 Laboratory Analysis Quality Control

Quality assurance measures for the Barnstable County Department of Health and Environment Laboratory are presented in Section 5 of the BCDHE Quality Assurance Plan (Appendix 7) and associated SOPs (Appendix 1).

2.6 Instrument/Equipment Testing, Inspection, and Maintenance (EPA QA/R-5 B6)

2.6.1 Field Measurement Instruments/Equipment

All field sampling equipment is inspected and cleaned following and before each outing. The field dissolved oxygen/pH/temperature/conductivity/turbidity meter is checked for calibration at the opening and closing of each day using three pH standards and air saturation for dissolved oxygen. A record is maintained for both the opening and closing of the meter as discussed in the SOP (Appendix 1 – MASSTC-SOP-016 – YSI ProDSS Field Meter, and opening and closing forms, Appendix 9 MASSTC-FRM-033 – ProDSS Calibration Checklist, Appendix 10 MASSTC-FRM-034 – ProDSS End of Day Checklist) used for verification of meter performance and maintenance, and these records are maintained for five years. As described in MASSTC-SOP-016 – YSI ProDSS Field Meter, field data and all confirming initial calibration verification (ICV) and continuing calibration verification (CCV) are directly uploaded at the end of each use and data are recorded as an Excel™ worksheet which is later correlated with any samples taken and reported through the laboratory information management system (LIMS). Anomalies in operation, when observed, are reported to the Quality Assurance Manager and, if necessary,

technical support is notified. The instrument is returned for service if problems are not rectified. MASSTC maintains a service contract of the instrument.

Small sampling pumps, and sampling receivers are regularly cleaned and checked for defects before and after each use. Conveyance and internal tubing (inside of peristaltic pumps) is changed regularly when showing any discoloration or indication of biofilm and growth.

See Table 3.

Table 3: Field Equipment/Instrument Calibration, Maintenance, Testing, and Inspection

Analytical Parameter	Field Equipment/ Instrument	Calibration Activity	Maintenance & Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action
Temperature	Multimeter, YSI ProDSS	See MASSTC-SOP-013 – Thermometer Calibration SOP	See MASSTC-SOP-016 - YSI ProDSS Field Meter.	Annual	Maximum correction factor of ±1.0°C of true value at both endpoints.	Remove thermometer from use.
Optical Dissolved Oxygen (ODO)	Multimeter, YSI ProDSS	See MASSTC-SOP-016 - YSI ProDSS Field Meter	See MASSTC-SOP-016 - YSI ProDSS Field Meter.	Every day that the meter is used.	Initial: one-point check using local ODO of water-saturated air ±1.0% from that day's calibration value. CCV: one-point check using local ODO of water-saturated air ±1.0% from that day's calibration value. Close: one-point check using local ODO of water-saturated air ±1.0% from that day's calibration value.	Initial: Recalibrate CCV: Recalibrate and re-take field measurements following last successful CCV. Close: Recalibrate and re-take field measurements following last successful CCV.
pH	Multimeter, YSI ProDSS	See MASSTC-SOP-016 - YSI ProDSS Field Meter	See MASSTC-SOP-016 - YSI ProDSS Field Meter.	Every day that the meter is used.	Initial: one-point check using 7.0 pH buffer ±0.2 pH units of true value.	Initial: Recalibrate

					CCV: one-point check using 7.0 pH buffer ±0.2 pH units of true value. Close: one-point check using 7.0 pH buffer ±0.2 pH units of true value.	CCV: Recalibrate and re-take field measurements following last successful CCV. Close: Recalibrate and re-take field measurements following last successful CCV.

2.6.2 Field Instruments/Equipment

Field analyses are not included as a part of this project.

2.6.3 Laboratory Analysis Instruments/Equipment

Inspection and maintenance of laboratory equipment is the responsibility of the Barnstable County Water Quality Laboratory and is described in the laboratory's QA Manual included as Appendix B.

2.7 Instrument/Equipment Calibration and Frequency (EPA QA/R-5 B7)

2.7.1 Field Measurement Instruments/Equipment

The field dissolved oxygen/pH/temperature/conductivity/turbidity meter is checked for calibration at the opening and closing of each day using three pH standards and air saturation for dissolved oxygen. A record is maintained for both the opening and closing of the meter as discussed in the SOP (Appendix 1). Data and notes related to opening the meter (Appendix 9- MASSTC-FRM-033 - ProDSS Calibration Checklist) and closing the meter (Appendix 10 - MASSTC-FRM-034 - ProDSS End of Day Checklist) are recorded and used for verification of meter performance and maintenance. Anomalies in operation, when observed are reported to the Quality Assurance Manager and, if necessary, technical support is notified. The instrument is removed from service if problems are not rectified. MASSTC maintains a service contract of the instrument. One instrument is dedicated to field activities while two identical instruments are used for onsite MASSTC activities that could be employed in the field in the event of a failure of the field instrument.

See Table 3.

2.7.2 Field Instruments/Equipment

Field analyses are not included as a part of this project.

2.7.3 Laboratory Analysis Instruments/Equipment

Laboratory instruments will be calibrated according to the appropriate analytical methods. Acceptance criteria for calibrations at Barnstable County Water Quality Laboratory are contained in their QA Manual included as Appendix 7.

2.8 Inspection/Acceptance Requirements for Supplies and Consumables (EPA QA/R-5 B8)

2.8.1 Field Sampling Supplies and Consumables

Sample containers will be provided by the Barnstable County Water Quality Laboratory. Containers requiring chemical preservatives are pre-prepared by the laboratory. Containers will be inspected for breakage and proper sealing of caps. Other supplies and consumable such as ice and sample coolers are acquired by MASSTC from reputable suppliers.

Any field supplies or consumables deemed to be in unacceptable condition will be replaced.

2.8.2 Field Measurement/Analyses Supplies and Consumables

Field measurement supplies, such as calibration solutions, will be acquired from standard sources, such as the instrument manufacturer or reputable suppliers. Chemical supplies will be American Chemical Society reagent grade or higher.

All consumables and supplies are logged in with information regarding receipt date, expiration date, lot number, and identification of the person logging it in. Prior to opening or using these materials, the expiration date is inspected, and the date opened is recorded on the registration sheet. MASSTC-FRM-014 - Chemical Receipt Log (Appendix 11) is used for this purpose and records are maintained indefinitely. Any staff member receiving or opening the material is responsible for recording the appropriate data.

Any field measurement supplies or consumables deemed to be in an unacceptable condition will be replaced.

Field analyses are not included as a part of this project.

2.8.3 Laboratory Analyses (Off-Site) Supplies and Consumables

The Barnstable County Water Quality Laboratory's requirements for supplies and consumables are described in its QA Manual which is provided in Appendix 7.

2.9 Data Acquisition Requirements (Non-Direct Measurements) (EPA QA/R-5 B9)

We anticipate no need for the use of external data sources.

2.10 Data Management (EPA QA/R-5 B10)

Data collected under this project meet three general descriptions: analytical laboratory data, field measurement data, and ancillary data including field observations, problems, photographs, etc.

2.10.1 Laboratory Data

Laboratory data are directly accessed from Barnstable County Water Quality Laboratory's Laboratory Information Management System (LIMS) on a weekly basis. These data are then stored in a Structured Query Language (SQL) file where they are manipulated to produce various internally developed outputs. These data are organized in the MASSTC Database to assist in graphing and charting. Data are capable of being exported as files compatible with Microsoft Excel™ and other character delimited files. This task is presently completed by the Quality Assurance Manager. Data are organized by OWTS installation address.

If, during the access and integration process, errors in the data are suspected the Laboratory Director is contacted and asked to verify the data. Scanned copies of all laboratory reports are maintained in a file directory on the Microsoft Sharepoint© platform. If upon inspection of the data by any staff working on a particular project there appears to be an error, the Laboratory Director is notified for comment and resolution.

2.10.2 Field Measurement Data

Field measurement data are downloaded from the field instrument daily and integrated with the laboratory data for the complete record. This is performed by the MASSTC Operator or the Quality

Assurance Manager. These records are also available for inspection by any staff in a shared file. The format of the downloaded file is compatible with Microsoft Excel™.

2.10.3 Ancillary Data

Field observation notes will be recorded in bound notebooks. Photographs, OWTS design drawings, and other electronic data are maintained in a file directory on the Microsoft Sharepoint© platform.

3 Assessment and Oversight

3.1 Assessments/Oversight and Response Actions (EPA QA/R-5 C1)

The Project Manager holds a weekly meeting with the Project Assistant on this project and, if required, the Quality Assurance Manager and other relevant staff. Regular discussions range from data reduction activities and quality assurance of data, chemical analyses update, sample collection scheduling, review of initial data. Any corrective actions or adjustments are discussed at these meetings and assignments are given to appropriate staff. These meetings are essentially audits of the project progress. Notes are taken and posted on the shared digital drive and preserved for future reference.

MASSTC also maintains an ISO-compliant Nonconformance (MASSTC-SOP-004, see Appendix 12) and Corrective/Preventive Action (MASSTC-SOP-005, see Appendix 13) process. Document-controlled forms are used to document nonconformances (MASSTC-FRM-001, see Appendix 14) and the associated corrective and preventive actions (MASSTC-FRM-002, see Appendix 15).

3.2 Reports to Management (EPA QA/R-5 C2)

Project reports are quarterly and contain information such as number of samples collected in reference to those projected in proposals, and the adherence or deviation from expected timelines. In some instances, preliminary data will be reported with limited interpretation. The contents of these reports have been detailed in Section 1.9.4 of this document. These reports will be prepared by the Project Manager and Project Assistant, reviewed by the QAM, and submitted to the US EPA Grants Project Officer.

The final report will report any data trends that emerged throughout the project and how actions taken during the project have affected the outcomes. The contents of these reports have been detailed in Section 1.9.4 of this document. This report will be prepared by the Project Manager and Project Assistant and submitted to the US EPA Grants Project Officer.

4 Data Review and Usability

4.1 Data Review, Verification, and Validation Requirements (EPA QA/R-5 D1)

Chemical data review begins in the BCDHE Laboratory and is conducted by the Laboratory Director. The process in that laboratory begins when analysts submit results through the Laboratory Information Management System (LIMS) and data awaits final approval by the Director. All data from external laboratories is incorporated into the BCDHE Laboratory report. Laboratory reports are generated for all samples associated with a Chain of Custody signed at laboratory reception. The analyst performing the analysis reviews the raw results for quality control, and when satisfied enters the results into the LIMS software. Raw data from any instruments and copies from original results recording logbooks are

attached to the original Chain of Custody, which is kept in a central area. Once all the data for that set of samples is entered, a report is generated and is attached to the original Chain of Custody and all of the instrument printouts, logbook copies, and subcontract laboratory reports into one package. That package is reviewed by an analyst who did not perform any of the associated analysis and by the Laboratory Director. Upon final review by the Laboratory Director the report is signed, dated, and delivered to the mailing desk for dissemination. The data review, verification, and validation requirements are presented in Section 13 of the QAP (Appendix 7).

Once approved at the analytical laboratory level, the MASSTC Quality Assurance Manager (QAM) has access to download these data into the data management system at MASSTC. The QAM checks at this point relate to ensuring proper connection with the appropriate project as well as timeliness of the received data. The laboratory is notified if the expected date of receipt of any data has exceeded five business days. Once these data are properly merged into the MASSTC Structured Query Language (SQL) setting, graphing and data analyses can be performed by any investigators that are required to report and interpret the data. The graphing routines allow the visual representation of trends, correlations, and outliers. Any suspect data at this point results in an inquiry back to the Laboratory Director who can verify all operational and analytical correctness down to an appropriate level. The QAM may ask for detailed description of the extent of the review.

The final steps in data review involve the staff investigators responsible for writing and interpreting the data. A number of tools facilitate the proper assessment of the data. Foremost, MASSTC has a database graphing program that allows for rapid visualization of data not only from the project at hand but from parallel or similar projects with similar data objectives or supportive comparisons. Graphic visualization often allows for the rapid spotting of outliers or anomalies in the data that might require investigation.

Field data review will be based upon the field QC methods; primarily instrument calibrations (see Appendix 9). In all cases, QC data as well as field calibrations and measurements will be recorded and supervised by the Project Manager. Furthermore, a record of all calibrations and closing verification is maintained along with each day's measurements. Initial review of field-collected data will occur in the field, at the time of measurement. Rejection of a measurement could be due to excessive instrument drift or instrument malfunction and would result in instrument re-calibration and re-measurement as necessary.

4.2 Verification and Validation Methods (EPA QA/R-5 D2)

Prior to submission to the Project Manager, laboratory data sets will have been reviewed first for completeness, errors in transcription, calculation, or computer input by the technical staff of the analytical labs. Laboratory analytical data will be submitted to the Project Manager in electronic and printed report format. Field and laboratory data will then be combined by the Project Manager in a master spreadsheet and reviewed for tabular errors. Data will subsequently be viewed in graphical form for further review of outliers as possible errors. The data will also be reviewed for technical reasonableness, which is defined as: whether the data fails within known or expected limits of data previously generated or by other similar wastewater systems.

If data appear to be suspect, verification and validation of data first proceeds with the assumptions that the laboratory analyses are correct. All the above conditions can then be reviewed for proper procedural

elements. Staff will ask the following questions of suspect data: Were all correct sampling techniques used? Do the field notes reveal any anomalies? Were there any operational anomalies on the day of sampling? Were the systems being tested receiving the correct doses? Were there any anomalous weather conditions? Were all field measuring instruments checked for operation? These and others can be verified either by inspection of hand-written records or the electronic retrieval of recorded operational elements.

In parallel, an inquiry is made to the Laboratory Director who can, in the instance of a suspect datapoint, inspect analysts' bench notes, calibration records, and instrument records. In some instances where samples are within holding time, the Director can have the suspect sample analyzed again.

Only data for which a definite miscalculation or transcription errors are documented will be changed. No data will be excluded or censored. A note will be placed on all reports indicating the reason for exclusion of any data point from graphic representation or statistical analyses.

4.3 Reconciliation with User Requirements (EPA QA/R-5 D3)

The purpose of the Shubael Pond Project is to determine the effect nitrogen-reducing onsite wastewater treatment systems will have on the quality of the groundwater when systems are installed throughout a community. In addition to determining the effect nitrogen removing systems can have at a neighborhood scale, we will be determining the nitrogen-removing performance of two technologies at the parcel scale (i.e. do systems meet effluent standards outlined in technology approvals). To be valuable to the project, data must fulfill the requirements of this Quality Assurance Project Plan. Decision making about the effectiveness of the project will be assessed using field data and laboratory data, as well as checklists and corrective action reports.

Prior to data summaries being incorporated into quarterly and final reports, the Project Manager will assess the final usability, and any limitations, of the data in meeting the project's objectives. This will include discussions with the Project Assistant and Quality Assurance Manager, a determination of whether potential deviations from the Quality Assurance Project Plan or SOPs impacted data quality, an evaluation of all results and quality control data, and an evaluation of the completeness of the goal of the Quality Assurance Project Plan.

APPENDICES

APPENDIX 1:
ANALYSIS METHODOLOGY

Barnstable County Department of Health and the Environment Laboratory

SM 2320B

STANDARD OPERATING PROCEDURE

For

Determination of Alkalinity in Aqueous Samples

(Revision 010)

June 16, 2020

		Signature	Date
Analyst:	Liping Xun	Liping Xun	9/20/21
Laboratory Director:	Dan White		21Sep21

5.1 PC Titrate (Man-Tech), Model PC -1000-1040
5.2 Man-Tech Autosampler, Model PC-1306-475

5.3 pH Electrode, Man-Tech, Model PCE 80-PH1200C

5.4 50 mL plastic vials, Fisherbrand #14-955-240

5.5 Reference filling solution, Mantech 4M KCL, PCE-R001013

5.6 Rinse solution, 200mL of pH 4.00 (SB101-4), Fisher Scientific, fill to 1000mL with tap water.

6. REAGENTS AND STANDARDS

6.1 Reagent water – Deionized water from Milli-Q Direct 8/16 System or Millipore Direct-Q 3 System,

6.2 Buffer Solution pH 4.00 (Red), Fisher Scientific, # SB101-4
Buffer Solution pH 7.00 (Yellow), Fisher Scientific, # SB107-4
Buffer Solution pH 10.0 (Blue), Fisher Scientific, # SB 115-4
Buffer Solution pH 7.00 (Clear), Fisher Scientific, # SB 108-1

6.3 0.02N Sulfuric Acid, Fisher Scientific SA 226-1or make from 98% H2So4(Fisher, #300-212)
6.4 QC standard Sodium Carbonate, Fisher Scientific S495-500.

6 SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1 Samples are collected in 500 mL plastic bottles. All bottles must be thoroughly cleaned and rinsed with reagent water.

6.2 Samples are stored at 4°C until analysis but must at room temperature when analyzed. Do not open sample bottle before analysis.

6.3 Holding time is 14 days.

7 QUALITY CONTROL

8.1 A duplicate sample is to be included in any set of 10 samples. The agreement between the sample and the duplicate must be ± 20%. If it is outside these limits, a third sample is analyzed. If this is outside the limits, a QC check is run to check

the calibration of the instrument itself. If the instrument is found to be out of calibration, it is to be recalibrated and all analyzes are repeated.

8.2 A Quality Control sample is to be run at the beginning. After every 10 samples, and at the end of the run, a CCV is to be run. Use pH yellow buffer (pH 7). The QC value is 25mg/L. It must fall within 10% of the range. If it is not within the range, a second analysis is performed to verify the measured value. Upon repeat failure, the results from this analysis will be considered unacceptable and the complete procedure will be repeated after the instrument is recalibrated.

8.2 1 To make the QC take 1.0g of Sodium Carbonate dilute to 1000mL with reagent water.

8.2 2 Take 25mL of the stock Sodium Carbonate solution and dilute to 1000mL for the QC 25 mg/L alkalinity Solution.

8.3 A Method Blank is run immediately following every QC sample. For the Blank, take 40mL of reagent grade water and place it in a 50mL plastic vial, and put it on the autosampler.

8.4 Method Detection Limit (MDL)- An MDL is established using reagent water fortified at 10 mg/L alkalinity, seven replicate aliquots of the fortified reagent water are run and processed through the entire analytical method. Then the MDL is calculated as follows:

$$MDL = (t) \times (S_{n-1})$$

Where,
t = student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.
S_{n-1} = sample standard deviation (n-1) of the seven replicate analyses.

8.3.1 The laboratory conducts MDL study annually. The MDL's must be run over a period of at least 3 days.

8.5 Accuracy and Precision- 4 replicates of 25 mg/L alkalinity are prepared and analyzed. The Accuracy and Precision study must be run over a period of at least 3 days. The mean measured concentration ($C_{\bar{x}}$) of the replicate values is calculated as follows

$$C_{\bar{x}} = \frac{(C_1 + C_2 + C_3 + C_4)}{n}$$

Where,

$C_{\bar{x}}$ = Mean recovered concentration of the replicate analysis.

$C_1, C_2, \ldots C_n$= Recovered concentrations of the replicate 1,2, …n.

The value derived for $C_{\bar{x}}$ must be within ± 10% of the true value.

The percent relative standard deviation (%RSD) of the replicate analysis as stated above is calculated using the following equation.

$$\%RSD = \frac{(S_{n-1})x100}{(C_{\bar{x}})}$$

Where,

S_{n-1} = sample standard deviation (n-1) of the replicate analysis.
$C_{\bar{x}}$ = mean recovered concentration of the replicate analysis.

9 CALIBRATION

9.1 In a 50 mL sample vial, place 40 mL of each of the 3 buffers. In position #1 on The autosampler place the pH 4.00, in position #2, put in pH 7.00 (yellow), and in position #3, put in pH 10.0.

9.2 A Quality Control Sample (QCS) is analyzed right after the initial calibration for the calibration verification. The acceptance limits for the recovery of the QCS must fall within 90-110%.

9.3 Continuing Calibration Verification (CCV): The QCS is also used as a CCV. The CCV must be analyzed at the beginning of the sequence and at the end of each ten samples as a closing CCV. The acceptance limits for the recovery of the CCV must fall within 90-110%.

10. OPERATIONAL PROCEDURE

10.1 Double click on PC Titration-

10.1 1 Main Menu will show up, Click on RUN Titration. Double Click on line 1, a menu will come up highlight pH CAL 4-7-10 and hit OK.
Under Order Number, highlight and click on Autogenerate.
Under Sample Name, type in pH cal
Under Vial#, type in #1
Hit Save As and type in the date, hit OK

10.1 2 Load the autosampler, #1 position is pH 4.00, # 2 is pH 7.00, and #3

is pH 10.0.

10.1 3 Hit START

10.1 4 The PC Titrate will perform the calibration and will say pH Calibration has passed.

10.2 SAMPLE ANALYSIS

10.2 1 Go to Titrator Main Menu- select RUN TITRATION

10.2 2 Double click on lst line- all the options will appear- select ALK QC-OK

10.2 3 Move the curser to order#, hit AUTO GENERATE, move to sample name type in ID of sample or QC, move to vial # and type that number in.

10.2 4 Go back to lst line-rt click- hit add a row or add rows
Double click on that next line, select pH QC-OK, move to order #, auto Generate, sample name, vial #, Repeat these steps for all samples, Selecting the appropriate analysis –pH-Alk or Cond-pH-Alk options.

10.2 5 After all samples have been typed in and orders hit SAVE, type in current date, OK, then START.

NOTE: *There are two methods set up internally in the Man-Tech instrument: One is for "low" alkalinity lower than 20 mg/L (, and the other one for "regular" alkalinity equal to or above 20 mg/L. In the schedule itself there is a "linked" titration where it will run the method based on the titration, and all samples start in the "low" method and move to regular if they are above the 20 mg/L.*

11. DATA ANALYSIS, CALCULATIONS AND REPORTS

11.1 Calculation:

The PC Titrate automatically calculates the titration.

11.2 Reports

All data, including the date, Lab ID, Client ID, the concentration (mg/L), and analyst initials are placed in the 3ring binder PC Titrate logbook.

12. POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The laboratory waste management practices are conducted consistent with all Applicable rules and regulations as stated in the laboratory's Sample and Waste Disposal (Revision 005) on December 15, 2017. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner

in this SOP.

13. REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D-1067, p13 Method B (1976)

2. American Public Health Association (APHA)
American Water Works Association (AWWA)
Water Environment Federation (WGI)
Standard Methods (SM) for the Examination of Water and Wastewater, 22nd edition SM2320B, 2012.

Barnstable County Department of Health and the
Environment Laboratory

EPA Method 350.1

STANDARD OPERATING PROCEDURE

For

Determination of Ammonia Nitrogen in Aqueous Samples by Semi-Automated Colorimetry Gas Diffusion Separation Method

(Revision 006)

March 5, 2019

		Signature	Date
Analyst:	Andrew Barker	Andrew L Barker	9/28/2021
Laboratory Director:	Dan White		28SEP21

STANDARD OPERATING PROCEDURE (SOP)

For

Determination of Ammonia Nitrogen in Aqueous Samples
by Semi-Automated Colorimetry
Gas Diffusion Separation Method
Salicylate Method

1.0 SCOPE AND APPLICATION

1.1 This SOP covers the determination of ammonia in drinking, ground, and surface waters, domestic and industrial wastes.

1.2 The applicable range is 0.10 – 20 mg/L NH_3 as N. The range may be extended with sample dilution.

2.0 SUMMARY OF METHOD

2.1 The sample containing ammonium is injected into a continuously flowing carrier stream by means of an injection valve, and mixed with a continuously flowing stream of an alkaline solution. The ammonia is separated from the matrix in a diffusion cell across a hydrophobic semi-permeable membrane and absorbed by a flowing acceptor stream. When ammonia in the acceptor is heated with salicylate and hypochlorite in an alkaline phosphate buffer an emerald green color is produced which is proportional to the ammonia concentration. The color is intensified by the addition is sodium nitroprusside. DCIC is used as the hypochlorite source in this method. Heat is used to aid ammonia from the donor in passing into the acceptor, in particular for the low ranges.

3.0 DEFINITIONS

3.1 Calibration Blank (CB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analyte.

3.2 Calibration Standard (CAL) – A solution prepared from the primary dilution standard or stock standard solutions.

3.3 Instrument Performance Check Solution (IPC) – A Solution of one or more method analytes or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 Laboratory Fortified Blank (LFB) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are

added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 Laboratory Fortified Sample Matrix (LFM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7 Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

3.8 Safety Data Sheets (SDS) [Used to be called as Material Safety Data Sheet (MSDS)] – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 Quality Control Sample (QCS) – A solution of method analytes of known concentrations that is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.11 Stock Standard Solution (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4.0 INTERFERENCES

4.1 In alkaline solution, calcium and magnesium will interfere by forming a precipitate. EDTA is added to the Alkaline Donor to prevent this interference

4.2 Lauryl sulfate and detergents can cause low ammonia recoveries, by wetting the membrane.

4.3 Oil and grease will also wet the membrane.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Barnstable County Health Laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Reference files of Safety Data Sheets (SDS) are available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult SDS.

5.3.1 Sulfuric acid.

5.3.2 Sodium nitroprusside.

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance – Analytical, capable of accurately weighing to the nearest 0.0001g (Fisher Scientific,Model ACCU-124D).

6.2 Glassware – Class A volumetric flasks and pipets as required.

6.3 Automated Continuous Flow Analysis Equipment – QuickChem 8500 Series 2 Flow Injection Analysis System (LACHAT Instruments, A Hach Company Brand)

6.3.1 LACHAT XYZ Autosampler.

7.0 REAGENTS, CHEMICALS AND STANDARDS

7.1 Sulfuric Acid (H_2SO_4), Fisher, Cat No. A300-212

7.2 Sodium Thiosulfate Pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$), Fisher, Cat No. RDC50930-500B1

7.3 Sodium Sulfite Anhydrous (Na_2SO_3), Fisher, Cat No. RDC50870-500B1

7.4 Sodium Tetraborate Decahydrate ($Na_2B_4O_7 \cdot 10H_2O$), Fisher, Cat No. AA4011436

7.5 Sodium Hydroxide (NaOH), Fisher, Cat No. S613-3

7.6 Sodium Hypochlorite (NaClO), Fisher, Cat No. 19-546-929

7.7 Disodium EDTA, (Ethylenediamine Tetraacetic Acid Dihydrate) ($Na_2EDTA \cdot H_2O$), Fisher, Cat No. BP120500

7.8 Sodium Nitroprusside (Sodium Nitroferricyanide Dihydrate) [$Na_2Fe(CN)_5NO \cdot H_2O$], Fisher, Cat No. AC21164-1000

7.9 Ammonium Chloride (NH_4Cl), Fisher, Cat No. AC199975000 and A661-3

7.10 Dichloroisocyanuric (DCIC) Acid Sodium Salt ($C_3Cl_2N_3NaO_3$), Fisher, Cat No. AAB2350436

7.11 **Reagent Water**: Ammonia free deionized water produced from Millipore Milli-Q Water Purification System.

7.12 **Degassing with Helium**:

7.12.1 To prevent bubble formation, degas the carrier and buffer with helium. Use He at 140 kPa (20 lb/in2) through a helium degassing tube. Bubble helium through one liter of solution for one minute.

7.12.2 All reagents used in heated chemistry must be degassed.

7.13 **Reagent 1: Alkaline Donor**

In a 1 L volumetric flask, add approximately 800 mL reagent water and 30.0 g ethylenediaminetetraacetic acid, disodium salt (EDTA)
Mix with a magnetic stirrer. Add 12.4 g boric acid. While mixing, add 40 g of sodium hydroxide (NaOH). Dilute to the mark with with reagent water. Degas this solution with helium. The pH of this solution will be approximately 13. This solution is stable for one month.

7.15 **Reagent 2: Buffer**

In a 2 L volumetric flask containing about 1 L reagent water, dissolve 30.0 g sodium hydroxide (NaOH), 25.0 g EDTA, and 67 g sodium phosphate dibasic heptahydrate $Na_2HPO_4 \cdot 7H_2O$ in about 900 mL reagent water. Dilute to the mark with reagent water and invert three times

7.16 Reagent 3: Salicylate Nitroprusside Color Reagent

In a 1 L volumetric flask, dissolve 350 g sodium salicylate $C_6H_4(OH)(COO)Na$ and 3.5 g sodium nitroprusside $Na_2Fe(CN)_5NO \cdot 2H_2O$ in about 800 mL reagent water. Dilute to the mark and invert and mix. Store in a light proof bottle.

7.17 Reagent 4: DCIC Reagent (Hypochlorite generator)

In a 500 mL volumetric flask, dissolve 2.5 g of sodium hydroxide NaOH and 2.5 g sodium dichloroisocyanurate dihydrate, in about 300 mL reagent water stir and bring to volume, this reagent may be degassed.

7.18 Reagent 5: Carrier / Diluent for Preserved Samples (Ammonia)

To a 2 L volumetric flask containing about 1 L reagent water, dilute 4 mL concentrated sulfuric acid H_2SO_4. Dilute to the mark with reagent water. This solution is used as the diluent for standards and over-range samples.

7.19 Calibration Standards

7.19.1 Standard 1 (S1): Stock Standard: 1000 mg/L

In a 1.0 L volumetric flask, dissolve 3.819 ammonia chloride (NH_4Cl) that has been dried for two hours at 110°C in about 800 mL reagent water. Dilute to the mark with reagent water and invert to mix.

7.19.2 Standard 2 (S2): Intermediate Stock Standard: 20.0 mg N/L in 0.04N H_2SO_4

In a 1 Liter volumetric flask, add 20.0 mL of the stock standard (**Standard 1**) to approximately 900 mL reagent water and then 1.099 mL of concentrated sulfuric acid. Dilute the mark with reagent water, and invert to mix.

7.19.3 Calibration Standards: Using Standard 1 (S1) and 2 (S2) (Section 7.20.1, Section 7.20.2) to have the autodilutor prepare the series of standards, as shown below, covering the desired range and a blank

by diluting suitable volumes of standard solution with Reagent 5, all done through the autodilutor (Section 7.18).

Initial Calibration Standard (ICS)	Concentration (mg/L)	Auto Dilution Factor
Level 1	20	1
Level 2	10	2
Level 3	5	4
Level 4	1.0	20
Level 5	0.25	80
Level 6	0	

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples are collected in disposable plastic. Volume collected must be sufficient to insure a representative sample, allow for replicate analysis, and minimize waste disposal.

8.2 Samples must be preserved with H_2SO_4 to a pH<2 and cooled to 4°C at the time of collection.

8.3 Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held for up to 28 days.

9.0 QUALITY CONTROL

9.1 Barnstable County Health Laboratory operates a formal quality control (QC) program. The QC program for this method consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks and other laboratory solutions as a continuing check on performance. The laboratory maintains performance records that define the quality of the data that are generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibration ranges and analysis of QCS) and laboratory performance (determination of MDL) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) – The LCR is determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected. The initial

demonstration of linearity uses a blank and five calibration standards. If any verification data exceeds the initial values by ±10%, linearity will be reestablished.

9.2.3 Quality Control Sample (QCS) – The QCS is analyzed right after initial calibration (Section 9.2.2) to verify the calibration standards and acceptable instrument performance with preparation and analysis of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL) – MDL must be established using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, seven replicate aliquots of the fortified reagent water are taken, processed and analyzed over a period of a minimum of three days. The spiking level is 0.10 mg/L which is the same as Level 6 in Section 7.19.3. The following equation is used to calculate the MDL:

$$MDL = (t) \times (S) \qquad (1)$$

Where

t = Student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates, and if more replicates are used, use the corresponding t-value].

S = Standard deviation of the replicate analyses.

9.2.4.1 The Standard deviation (S) can be calculated using the following equation:

$$S = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}} \qquad (2)$$

Where, n = number of samples;
x = concentration in each sample.

9.2.4.2 MDLs must be determined every six months, when a new operator begins work, or whenever there is a significant change in the background or instrument response.

9.2.4.3 One set of MDLs is listed as follows:

Spiking Level =0.10 mg/L							Unit: mg/L			
	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MEAN	STDEV	MDL
DATE	10/4/2017	10/4/2017	10/4/2017	10/11/2017	10/13/2017	10/13/2017	10/13/2017			
Conc	0.108	0.084	0.102	0.135	0.077	0.087	0.145	0.1054	0.026	0.0818

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) – The laboratory analyzes at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination must be suspected and corrective actions must be taken before continuing the analysis.

9.3.2 Laboratory Fortified Blank (LFB) – The laboratory analyzes at least one LFB with each batch of samples. Calculate accuracy as percent recovery as follows:

$$R = \frac{C_s - C}{S} \times 100 \qquad (3)$$

Where,
R = percent recovery;
Cs = recovered fortified blank concentration;
C = blank background concentration;
S = concentration equivalent of analyte added to blank.

9.3.2.1 If the recovery of any analyte falls outside the required control limits of 90-110%, the result is judged out of control, and the source of the problem must be identified and resolved before continuing analysis.

9.3.3 The laboratory also uses LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 25 analyses), optional control limits and control charts can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x – 3S

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to ten new

recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also the standard deviation (S) data must be used to establish an on-going precision statement for the level of concentration included in the LFB. These data are kept on file and be available for review.

9.3.4 Instrument Performance Check Solution (IPC) – For all determinations the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every 10th sample (or more frequently, if required), and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, the IPC solution is reanalyzed. If the second analysis of the IPC solution confirms calibration to be outside the limits, the sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution are kept on file with the sample analysis data.

9.4 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

9.4.1 Laboratory Fortified Sample Matrix (LFM)

9.4.1.1 The laboratory adds a known amount of analyte to a minimum of 20% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. The added analyte concentration should be the same as that used in the laboratory fortified blank.

9.4.1.2 The percent recovery for ammonia is calculated and corrected for concentration measured in the unfortified sample using the following equation:

$$R = \frac{C_s - C}{S} \times 100 \qquad (4)$$

Where, R = percent recovery;
Cs = fortified sample concentration;
C = sample background concentration;

S = concentration equivalent of analyte added to sample.

Acceptable range of R is 90-110%.

9.4.1.3 If the recovery falls outside the designated LFM recovery range (90-110%) and the laboratory performance is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be matrix related, not system related.

9.4.2 Laboratory Duplicate Sample

9.4.2.1 Duplicate samples are analyzed to demonstrate the precision of an analytical system. The duplicate analyses are performed on each batch of samples analyzed at a frequency of 10% of all samples in the batch or at least one sample if less than 10 samples are analyzed.

9.4.2.2 Relative Percent Difference (RPD): The relative percent difference is used to evaluate precision for the duplicate analyses, and RPD is calculated as follows:

$$RPD\ (\%) = \frac{|C_1 - C_2|}{C_{AVG}} \times 100 \qquad (5)$$

Where: C_1 = original sample concentration;
C_2 = duplicate sample concentration;
C_{AVG} = average of the two samples.

9.4.2.3 RPD Acceptable Limits: Acceptable limits of RPD for ammonia as nitrogen are ≤10%. If the recovery falls outside the designated duplicate recovery range and the laboratory performance is shown to be in control (Section 9.3), the recovery problem encountered with the duplicate analysis is judged to be matrix related, not system related.

10 CALIBRATION AND STANDARDIZATION

10.1 Prepare reagent and standards as described in Section 7.

10.2 Set up the Ammonia manifold as shown in Section 17.4 (Ammonia) of the Lachat Instruments Methods Manual (Section 14.2)

10.3 Input data system parameters as shown in Section 17.1 (Ammonia) of the Lachat Instruments Methods Manual (Section 14.2.) Also, see figure 2.

attached here at the end for Data System parameters and figure 3. for the manifold diagram.

10.4 Pump reagent water through all reagent lines and check for leaks and smooth flow. In order to avoid precipitate forming in the manifold tubing: Add the **Buffer Line First** and allow to pump through manifold for at least 5 minutes. Then the Carrier and other reagent lines one by one, ending with the nitroprusside added last. For removal after analysis, reverse this order with the nitroprusside line disconnected first, and the buffer line last. When finished, place all respective reagent lines into water and allow to pump through manifold for ten minutes.

10.5 Place standards in the sampler and sequence the required information in the data system.

10.6 Calibrate the instrument by injecting the standards. The system will then associate the concentrations with the peak area for each standard to determine the calibration curve.

10.7 The initial calibration is deemed acceptable if the following criteria are met:

10.7.1 $R \geq 0.995$

10.7.2 Quality Control Sample (QCS) standard is run right after the initial calibration. The concentration of the QCS is 5.0 mg/L. This standard (Ammonium chloride) is ordered from Fisher Scientific, Acros Organics, ACS reagent grade. The procedure for making the QCS is similar to the one for ICS 2 of the calibration standards described in (Section 7.20.1, 7.20.2), but having a final concentration of 5.0 mg/L The QCS concentration must fall within ± 10% of the stated value.

10.7.3 Instrument Performance Check (IPC) refer to (Section 9.3.4).

10.8 Figure 1. Lists a set of initial calibration peaks and a linear calibration curve

11 PROCEDURE

11.1 FLOW INJECTION SYSTEM START-UP PROCEDURE

11.1.1 Prepare reagents and standards as described in section 7.

11.1.2 Set up manifold as shown in Section 17.4 of the Lachat Instruments Methods Manual.

11.2.3 Input peak timing and integration window parameters as specified in section 17.1 of the Lachat Instruments Methods Manual.

11.2.4 Pump reagent water through all the reagent lines and check for leaks and smooth flow. Switch to reagent lines - add buffer first and pump through the system for 5 minutes, followed by the other reagents, adding salicylate nitroprusside last – and allow the system to equilibrate until a stable baseline is achieved.

11.2.5 Place the standards in the autosampler, and fill the sample tray. Input the information required by the data system, such as concentration, replicates and QC scheme.

11.2.6 Calibrate the instrument by injecting the standards with the autodilutor. The data system will then associate the concentrations with responses for each standard.

11.2.7 After a stable baseline has been obtained, start the sampler and perform the analysis.

11.3 ANALYTICAL SEQUENCE

Please see Table 1 for analytical sequence.

11.4 TROUBLESHOOTING AND SYSTEM NOTES

11.4.1 Allow at least 15 minutes for the heating unit to warm up to 60°C.

11.4.2 If phosphorus is also determined with the Lachat System, a second helium degassing tube should be used and segregated for the individual chemistries.

11.4.3 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

11.4.3.1 Place transmission lines in water and pump to clear reagents first.

11.4.3.2 Place reagent lines in 1M HCl and pump for several minutes

11.4.3.3 Place all lines back into water and pump out HCl.

12 POLLUTION PREVENTION

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous

opportunities for pollution prevention exist in the laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

12.2 Quantity of the chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

13 WASTE MANAGEMENT

13.1 The laboratory waste management practices are conducted consistent with all applicable rules and regulations as stated in the laboratory's Sample and Waste Disposal (Revision 001) on February 25, 2004. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner in this SOP.

14 REFERENCES

14.1 U.S. Environmental Protection Agency, **Methods for Chemical Analysis of Water and Wastes**, EPA-600/4-79-020, Revised March 1993, Method 350.1

14.2 Lachat Instruments Methods Manual, QuikChem Method 10-107-06-5-J Rev 2.0, Revision Date, 16 January 2015.

Figure 1.

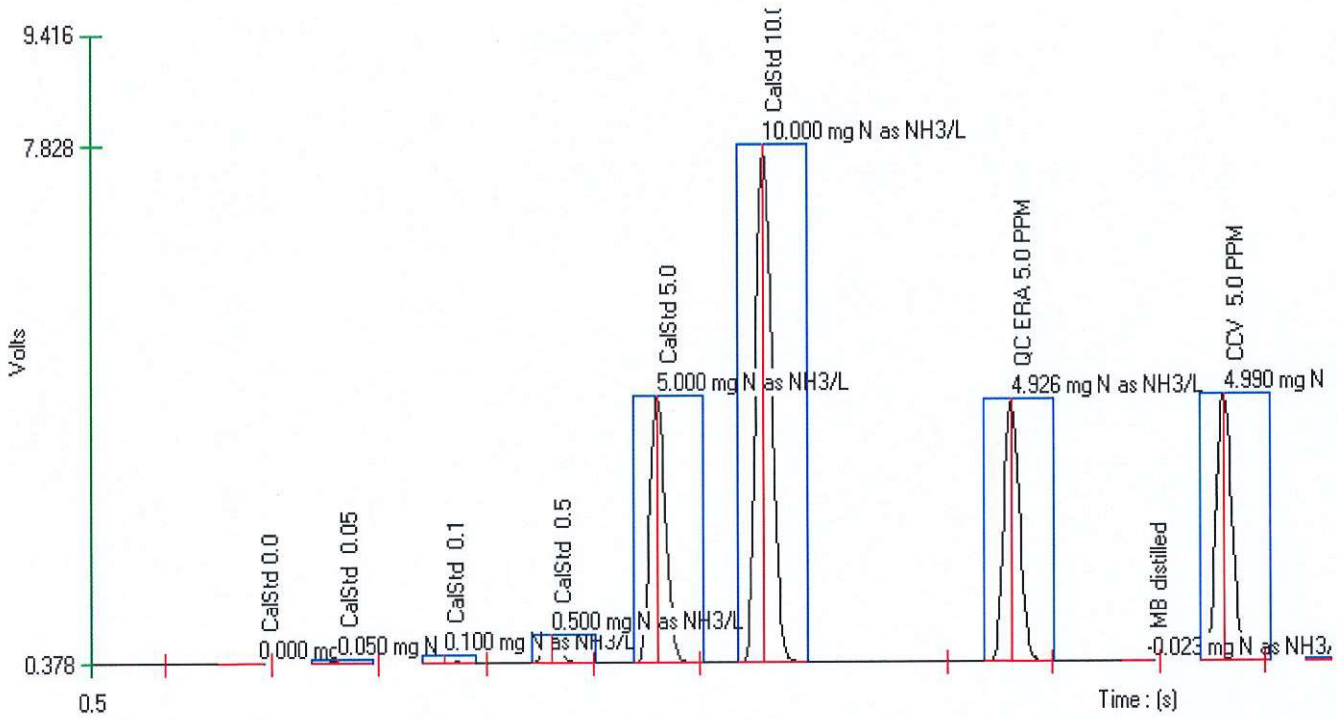
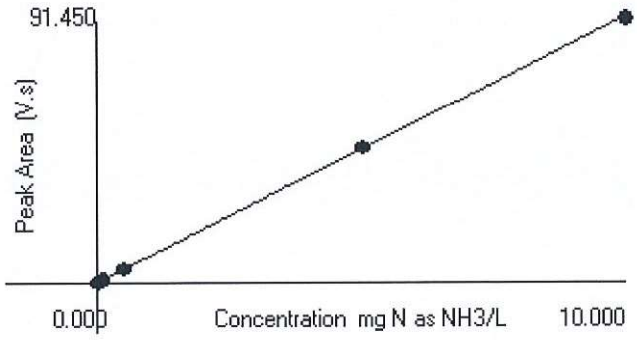


Figure : 1 (Ammonia)



Area = 9.205 * Conc + 0.207
Conc = 0.109 * Area - 0.022
Correlation Coefficient (r) = 0.99998
Weighting : 1/x

Figure 2.

17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1. DATA SYSTEM PARAMETERS FOR THE QUIKCHEM 8000/8500 FOR AMMONIA

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

Sample throughput: (low):	36 samples/h, 110 s/sample
Sample Throughput (high)	45 samples per hour, 80s/sample
Pump Speed:	35
Cycle Period Low:	110s
Cycle Period High:	80s

Analyte Data:

Concentration Units:	mg N/L or µg N/L
Chemistry High Range:	Direct/Bipolar
Peak Base Width:	
Inject to Peak Start:	
Chemistry Low Range:	Brackish
Inject to peak start:	81s
Peak base width:	85s
Brackish shutter offset:	21.4
Brackish shutter width:	30.5

Calibration Data:

Low Range Ammonia

Level	1	2	3	4	5	6	7	8
Concentration µg N/L as NH_3	1000	500	250	100	50	25	10	0.0

High Range Ammonia

Level	1	2	3	4	5	6	7	8	9
Concentration mg N/L as NH_3	20	10	5	2.5	1	0.5	0.25	0.1	0.00

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

Sampler Timing:

Min. Probe in Wash Period:	5 s
Probe in Sample Period:	39 s

Valve Timing:

Load Period:	32 s
Inject Period:	20 s

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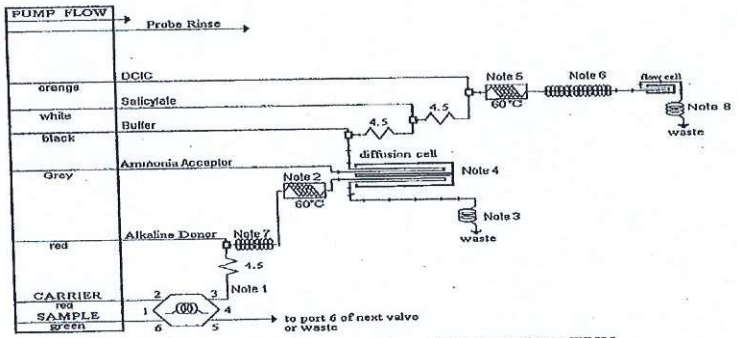
Figure 3.

17.4. AMMONIA MANIFOLD DIAGRAM (REV. 2.0)



Carrier: DI water for ammonia or Reagent 5 for TKN
Acceptor: DI water
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
QC8000/8500 Sample Loop: 350 cm Low Range Ammonia
80 cm High Range Ammonia and TKN
Interference Filter: 660 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required. The ⊣▨⊢ shows 175 or 650 cm of tubing wrapped around the heater block at the specified temperature.

4.5: 70 cm of tubing on a 4.5 cm coil support
⊣▦⊢ tubing wrapped in a figure 8 around 2, 7 or 22 cm coil supports (see below)
PVC PUMP TUBES MUST BE USED WITH THIS METHOD.

Note 1: 30 cm of manifold tubing is used to connect Port 3 to the first tee.
Note 2: 175 cm of tubing on the heater
Note 3 400 cm of 0.022" i.d. tubing backpressure loop, then to waste. **Waste container for the diffusion block should be on the bench with the instrument.** Placing this on the floor can lead to poor precision due to cavitation/vapor lock.
Note 4: Diffusion block with membrane PN 5033101-10 (Pack of 10 3.5 x 8.6 mm). Flow is **concurrent.** Be sure to place a membrane in the block before beginning. **Run**

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Table 1. A Typical Analytical Sequence with Quality Control Requirements

Injection #	Description of Quality Control Standards and Samples	Acceptance Criteria
1	Level 1 (20 mg/L) of Initial Calibration	R ≥ 0.995
2	Level 2 (10 mg/L) of Initial Calibration	
3	Level 3 (5.0 mg/L) of Initial Calibration	
4	Level 4 (1.0 mg/L) of Initial Calibration	
5	Level 5 (0.25 mg/L) of Initial Calibration	
6	Level 6 (0 mg/L) of Initial Calibration	
7	QCS at 5.0 mg/L	90-110%
8	Blank	≤ 0.10 mg/L
9	CCV at 5.0 mg/L	90-110%
10	MB	≤ 0.10 mg/L
11	LFB at 5.0 mg/L	90-110%
12	Sample 1	
13	Sample 1 – Laboratory Duplicate	≤ 10%
14	Sample 1 - Matrix Spike	90% – 110%
15	Sample 2	
16	Sample 3	
17	Sample 4	
18	Sample 5	
19	Sample 6	
20	Sample 7	
21	Sample 8	
22	Sample 9	
23	Sample 10	
24	Blank	
25	CCV	90-110%
26	MB	≤ 0.10 mg/L
28-35	Sample 11	
36	Sample 11 - Duplicate	≤ 20%
37	Sample 11 - Matrix Spike	90% – 110%
38	Sample 12 to Sample 20	
39	Blank	≤ 0.125 mg/L
40	CCV at 5.0 mg/L	90-110%
41	MB	≤ 0.125 mg/L
42	LFB at 5.0 mg/L	90-110%

STANDARD OPERATING PROCEDURE

For Method SM 5210 B

Determination of Biological Oxygen Demand (BOD, BOD5,c-BOD) in Aqueous Samples Using Dissolved Oxygen Meter (Method SM 4500-O-G)

Reference Standard Methods 22nd Edition, 2017

Method revision 2011

		Signature	Date
Analyst:	Maria Cathcart	electronically signed	01/22/19
Laboratory Director:	Ron Saari	electronically signed	01/22/19

STANDARD OPERATING PROCEDURE FOR BIOCHEMICAL OXYGEN DEMAND METHOD SM 5210 B (5-DAY)

1.0 SCOPE AND APPLICATION:

1.1 The biological oxygen demand (BOD) test is used to determine the oxygen requirement of municipal and industrial wastewaters.

1.2 Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen supply of the receiving waters. Data from BOD tests are used in engineering calculations in designing wastewater treatment plants.

2.0 SUMMARY OF METHOD:

2.1 The method consists of filling a 300 ml BOD bottle to almost overflowing, with the appropriate dilution of the sample and dilution water. The sample is incubated in the dark for 5 days at 20 ° C. The reduction in dissolved oxygen concentration during the incubation period from the dilution water yields a measure of the biological oxygen demand.

3.0 APPARATUS AND REAGENTS:

3.1 Incubator. Thermostatically controlled at 20 °C±1, closed to exclude all light.

3.2 3000 ml container for dilution water preparation.

3.3 300 ml BOD bottles.

3.4 plastic sealing caps

3.5 graduated pipettes

3.6 graduated cylinders

3.7 vacuum bubbler

3.8 HACH BOD buffer pillows cat no. 14861-98 and cat no. 1416066

3.9 Polyseed (InterLAb) BOD Seed Inoculum cat no. P-110

3.10 HACH BOD standard solution ampule 300 mg/L glucose cat. No. 14865-10

3.11 Iodate-Iodide Standard Solution 0.00125N cat no. 401-49

3.12 Hach HQ 40d DO meter and probe

3.13 Distilled water

3.14 Sodium thiosulfate 0.0125 N for dechlorination

3.15 Dissolved Oxygen Meter HACH HQ 40d

3.16 Sodium Sulfite, Cobalt chloride for Zero Std.

3.17 Hach Nitrification Inhibitor Formula 2533 Cat 253334

4.0 Washing

4.1 The BOD test is a semi-sterile procedure.

4.2 All glassware used for test are washed thoroughly with biodegradable detergent, and rinsed three time with distilled water.

1. BOD bottles are sterilized in autoclave.

5.0 SAMPLE SIZE

A. Practically all wastewater plants operate within a predictable BOD range for various sampling points based on previous in-house and State analyses of record. These must be consulted prior to using this method.

B. Select earlier HIGHEST and LOWEST recorded BOD values for types of samples desired, e.g., Raw Influent$_1$ Primary and Final Effluents.

C. Determine a reasonable day-to-day BOD value from the above and consult Table 1 - A Guide to Sample Size Selection.

D. For example, a plant has had a high Raw BOD value of 350 and a low of 110. The usual value has been about 180. If desired, proceed to Step H.

E. Inspection of the Table shows that for a BOD range of 60-420 a sample size of five (5) ml is recommended. This is a 1:60 dilution.

F. Influent (Raw) BOD dilution for this plant prepared with a 5 ml sample should show a 5-day D.O. depletion of about 2-6 mg/l corresponding to a BOD of 120-360.

G. The D.W., if prepared and stored as described below, (Section 6), will have a D.O. well in excess of 8 mg/L so that the above depletions can be easily measured.

H. A simple formula for determining BOD sample sizes may be used if we take the value from Step D and also assume that the ideal 5-day D.O. depletion for any

BOD sample is about 4 mg/L or approximately *50%* of the available D.O.

$$BOD = Depletion \times Factor$$

$$BOD = \frac{4 \times 300}{Sample\ Size}$$

$$Thus,\ Sample\ Size = \frac{1200}{BOD}$$

In the example of Step D,

$$Sample\ Size = \frac{1200}{180} = 6.7\ ml$$

2b Using Table 1, the nearest practical volume would be 6 or 7.5 ml.

I. Sample sizes may be varied periodically as required by plant operating conditions and as judged by Suspended Solids and Turbidity values.

J. Table 2 gives additional general information on BOD's and sample sizes for various water types.

6. <u>Dilution Water</u> (D.W.)

A. Allow about seven gallons of dilution water per BOD run.

B. Add 1 HACH BOD buffer pillows cat no. 14861-98 to each of three gallons of distilled water. Measure DO if below 7.5 aerate.

C. Aerate the D.W. vigorously for five (5) minutes (or more) by drawing clean air through or by shaking and place in the incubator at 20+/1 C.

D. If the dilution water is shaken to maximize the D.O. it should be allowed to sit for a minimum of 15 minutes to remove pin-point air bubbles.

Preferably, one to 24 hours will reduce the chance for oxygen super-saturation, high D.O. values initially and otherwise unexplainable D.O. losses after incubation.

E. The aeration step should be repeated before each new BOD run.

F. The D.W. should be kept in the incubator, particularly during the summer months. Exposure to bright light must be avoided to forestall algae growth.

G. D.W. should not be used for the test if the D.O. is not at least 7.5 mg/L or temp not 20°±3 C.

7. Procedure

A. Dechlorination of Effluent

1. Test the effluent sample for residual chlorine using test strip (Micro check HF Scientific). Record results on BOD worksheet.
2. If the test shows that no chlorine, proceed directly to Part B. In this case, seeding (Section 8) is not required because it may be assumed that sufficient wastewater organisms remain to exert the full BOD.

If chlorine is present, add one ml of 0.025N Sodium Thiosulfate to one liter of effluent, stir well and allow to stand for a short time until a second residual chlorine test is negative. It may then be used for the BOD test. This step will dechlorinate up to about 7 mg/l total residual chlorine.

Alternatively proceed as follows:

a) Measure out 100 ml of the effluent sample, add 0.5 g potassium iodide and one ml. of conc. sulfuric acid. Swirl until dissolved.

b) Add 5-10 drops of starch indicator solution and titrate dropwise with 0.025N (N/40) sodium thiosulfate to the disappearance of the blue color. Record the volume of "thio" used (per 100 ml effluent).

c) Using a fresh sample of effluent, measured carefully in multiples of 100 ml (depending on sample volume requirements for the BOO test), add from a buret, the calculated amount of "thio". Mix well. This mixture is used for the BOD determination.

NOTE: PAO (phenyl arsine oxide) MUST NOT BE USED FOR DECHLORINATION OF

BOD EFFLUENT SAMPLES SINCE PAO IS A POWERFUL AND PERMANENT BACTERIOSTAT. BOD VALUES OF ZERO WILL RESULT!

B. Check pH of each sample .Record on worksheet. pH should be in range of 6.0-8.0. If not in range, adjust pH with NaOH/H_2SO_4 to pH of 7.0-7.2.

C. Dissolved Oxygen meter calibration.

a. Press Calibrate. Place probe in DI water that has been saturated with air.

b. Press Calibrate. Allow to stabilize. Record slope. Save calibration.

c. Measure D.O. of Zero Standard (1 g Na_2SO_3 and few crystals of Co_2Cl crystals.

d. Check Standard (0.00125N Iodate-Iodide Standard 10.0 mg/L, purchased from HACH)

e. Record on worksheet

D. <u>Set-Up of Run</u>

1. Fill 2 BOD bottles with D.W. by pouring without bubbling or splashing. Measure D.O. and Record as initial D.O. Stopper all of them and fill the flared bottle collars with distilled water, then attach plastic caps to two of the bottles. These are the D.W. blanks. Place in BOD incubator.

2. Seeding

Quality control samples: fill 10 BOD Bottles up to half way with DW as in Step 1. a) to four(4) BOD bottles add 15,20,25, and 30 mL respectively with the polyseed solution that was previously prepared. Polyseed solution: 1 capsule of Inter Lab Polyseed (Cat # LS2001047) in 500 mL of DW. Mix for 1 hour and let stand for 15 minutes. To the remaining 5 bottles add 3 mL of Polyseed solution.

3. BOD standard: to the 6 BOD bottles with the 3 mL of Polyseed solution add 4, 4, 4, 3, 2, and 1 mL of the BOD standard (300 mg/L of glucose and glutamic acid ampule purchased from Hach Cat #14865-10.

4. Fill the BOD bottles to volume and measure the Initial DO and record. For each wastewater sample, fill BOD bottles up to half way with DW. as in Step 1, then add appropriate volume of the sample using a wide-tip pipet or graduated cylinder the same way. Add 3 mL of the Polyseed solution. For c-BOD samples add 0.16 g or 2 shots of Nitrification Inhibitor to each bottle. Use 2-4 bottles with different volumes. Measure the D.O. of each bottle and record as initial D.O. Fill the bottles with D.W., stopper and fill the bottle collars with

distilled water, then attach plastic caps to two of each set. Put these in the BOD incubator (20°C±1).

NOTES: a) For a 200 ml or greater sample the BOD bottles filled with the sample and DW to volume if necessary as in Step 1 and a concentrated buffer pillow is added to the sample (Hach Nutrient Buffer Pillow Catalogue #1416066)

b) BOD runs which use samples of 50 ml or more should be made with effluent which has been freshly aerated in a similar manner to the dilution water. These aerations provide for the maximum oxygen reserve for use during the incubation by the micro-organisms.

c) All samples and the D.W. should be brought to a temperature of 20°C before setting up the BOD run.

d) After 5-day incubation, analyze D.O. in all dilutions of each sample and record value as final D.O.

3. Determine the D.O. of one seeded blank and set aside two (2) BOD seed blank bottles as usual for five (5) days in the incubator.

4. Set-up three (3) BOD bottles containing a sample volume of dechlorinated effluent in addition to one ml of seed. Proceed as in Step 3.

5. After 5-day incubation determine the D.O. of the seed blanks and seeded effluent samples.

NOTES: a) At least two bottles of each kind are incubated for five days to provide insurance against their loss by careless D.O. measurement or accident. If a BOD sample is lost in Step 3 (initial D.O.) another may be promptly prepared instead.

b) Dilution Water D.O. depletions of over 0.2 mg/l not only invalidate the entire run for reporting purposes but require a careful check of the BOD dilution water or of BOD technique. The most common causes are dirty (bacteria, mold and algae) equipment and

reagents used for the test and blowing air into the dilution water from the pressure outlet of a mechanical vacuum pump.

c) B.O.D. bottles which deplete less than 2.0 mg/l D.O. over five days will probably not give reliable B.O.D. results. Larger samples should be used for future runs.

d) All B.O.D bottles having a 2.0 mg/L minimum depletion and at least a 1.0 mg/L residual DO must be used in calculations.. In case of no alternative, it may be reported by taking the initial D.O. value of the sample, multiplying by the dilution factor and reporting the value obtained with a + sign following.

For example: Raw Sample of 5 ml
Dilution Factor = 60
Initial D.O. = 8.2 mg/l
5-day D.O. = 0.6 mg/L (IGNORE)
Reportable BOD = 8.2 x 60 = 492+

e) BOD bottles must have at least 1.0 mg/L D.O. residual to ensure insufficient D.O. does not affect the rate of oxidation of the sample.

8. Quality Control

To check the lab technician's overall BOD methodology, 4 seeding blanks are used and the average is used to calculate a factor for the BOD concentrations. A seeded 300 mg/L BOD standard solution ampule (Purchased from HACH cat # 1486166) is used with a dilution factor of 75(add 4.0 ml std), 100(add 3.0 ml std). A 300 dilution (add 1.0 ml std) is used for MDL. The BOD value should be between 167.5 and 228.5 mg/L. Dilutions are performed for each run. Sample duplicates are performed every 10 samples. (±30%).

9. Calculations - REPORT ALL VALUES TO NEAREST WHOLE NUMBER.

BOD precision may vary up to ±25%. Changes in D.O. of the D.W. blank quality controls are NOT to be used for correcting any wastewater sample D.O. results. The D.W. blanks serve ONLY as a rough check on D.W. quality.

A. Regular (Unseeded)

$$\text{BOD (mg/l)} = (\text{Initial D.O.} - \text{5 day D.O.}) \times \text{Factor Depletion}$$

$$\text{Factor} = \frac{300}{\text{sample size}}$$

B. Seeded: **Calculations done on Excel spreadsheet**

1. Using Polyseed

$$\text{Seed Correction (S.C.)} = \frac{\text{depletion of Influent}}{\text{ml per influent sample used for regular BOD run}}$$

$$\text{BOD (Seeded) (mg/L)} = (\text{Depletion} - \text{S.C.}) \times \text{Factor}$$

2. Using SEED BLANK

$$\text{Seed Correction (S.C.) per ml} = \frac{\text{Depletion of Seed Blank}}{3}$$

$$\text{BOD (Seeded) (mg/L)} = (\text{Depletion} - \text{S.C.}) \times \text{Factor}$$

C. KHP Quality Control

S.C. is same as in Part B-I above.

$$\text{BOD of KHP (mg/L)} = (\text{Depletion} - \text{S.C.}) \times 60 \text{ D.}$$

D. %Removal

Percent Removal of BOD by WPC =

$$\frac{\text{(mg/L Influent BOD-m g/L Effluent BOD) X 100}}{\text{mg/L Influent BOD}}$$

Table 1. Guide to Sample Size Selection for BOD's

The dilution water is assumed to have D.0. in excess of 8 mg/L (store in incubator during warm weather).

BOD bottle volume is taken as 300 ml Factor = $\frac{300}{\text{Sample size}}$

BOD = (5 day D.O depletion) X Factor

BOD. Range Measurable	Sample Size ml. % Dilution	Factor	
300,000-2,100,000	0.001	300000	0.00033
30,000-210,000	0.01	30000	0.0033
10000-70000	0.03	10000	0.01
3000-18000	0.1	3000	0.033
1000-7000	0.3	1000	0.1
600-4200	0.5	600	0.17
300-2100	1	300	0.33
150-1050	2	150	0.67
100-700	3	100	1
75-525	4	75	1.33
60-420	5	60	1.67
50-300	6	50	2
40-280	7.5	40	2.5
30-210	10	30	3.33
25-175	12	25	4
20-140	15	20	5
15-105	20	15	6.67
12-84	25	12	8.33
10-70	30	10	10
7.5-52.5	40	7.5	13.33
6-42	50	6	16.67
5-30	60	5	20
4-28	75	4	25
3-21	100	3	33.33
2-14	150	2	50
1-7	300	1	100

NOTE: Allowable D.O. depletion (5-day) is 1-7 mg/L range per bottle.

TABLE 2 Biochemical Oxygen Demands of Various Types of Water

Type	BOD Range mg/L	Required Dilution Factor (300 m. bottle)	ml sample to be taken
Potable, Surface Waters	1 – 20	1-5	60-300
Trickling fi1ter OR Activated Sludge Effluents	10- 100) 5- 50)))	1- 20	15-300
Influent Sewage Primary Effluent	50 – 500	20 – 100 OR	3-15
Digested Sludge Supernatant OR Filtrate	500 - 2500	100 - 600	0.5 – 3
Industrial Wastes	500-5000	300-1000	0.3 – 1
Process Chemicals	30K – 2M	10K - 300K	0.001 -0.03

K=1000 M=Million

ı The Standing Operating Procedure for Analytical Chemistry for Envirotech Laboratories, Inc. have been reviewed

And approved 10/2/18

Electronically signed_____
Ronald J. Saari
Laboratory Director

Electronically signed_____
Maria Cathcart
QA/QC Manager

ISSUE DATE: 10/4/18

Barnstable County Department of Health and the Environment Laboratory

EPA Method 300.0

STANDARD OPERATING PROCEDURE

For

Determination of Inorganic Anions in Aqueous Samples Using Ion Chromatography

Revision 015

May 11, 2018

		Signature	Date
Analysts:	**Lacey Prior**	Lacey Adams Prior	5/11/18
	Diane Brown	Diane Brown	5/11/18
Laboratory Director:	**Gongmin Lei**	Gongmin Lei	5/11/2018

Standard Operation Procedure for the Determination of Inorganic Anions in Aqueous Samples Using Ion Chromatography

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the following inorganic anions in reagent waters, mixed domestic and industrial wastewaters, surface water, ground water, solids, leachates (when no acetic acid is used) and finished drinking water using ion chromatography.

Bromide, Chloride, Fluoride, Nitrate-N, Nitrite-N, ortho-Phosphate-P, Sulfate

1.2 This laboratory's Method Detection Limit (Section 8.1.4.) for the above analytes is listed in Table 1.

1.3 Whenever this method is used to analyze unfamiliar samples for any of the above listed anions, anion identification is supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 8.2.3.2.

2.0 SUMMARY OF METHOD

2.1 A 1.0 or 5.0 mL volume of sample is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a system comprised of an ion chromatographic pump, sample injection valve, guard column, suppressor device, and a conductivity detector.

2.2 This method may be modified for limited performance-based attributes provided that they documented and meet the requirements expressed in the Quality Control Section (Section 8.0)

3.0 INTERFERENCES

3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anions of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

3.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline ion chromatograms.

3.3 Any anion that is not retained by the column or slightly retained will elute in the area of fluoride and interfere. Known co-elution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant, however it is

the responsibility of the user to generate precision and accuracy information in each sample matrix. (Section 8.0, Quality Control.)

3.4 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. This method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

3.5 The quantitation of un-retained peaks should be avoided, such as low molecular weight organic acids (formate, acctate, propionate etc.) which are conductive and co-elute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

3.6 Any residual chlorine dioxide present in the sample will result in the formation of additional chlorite prior to analysis. If any concentration of chlorine dioxide is suspected in the sample purge the sample with an inert gas (argon or nitrogen) for a minimum of 5 minutes until no chlorine dioxide remains.

4.0 SAFETY

4.1 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are specifically listed below in Section 4.3 for hazardous materials.

4.2 The laboratory is maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the analysis.

4.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

4.3.1 Sulfuric Acid (H_2SO_4), if and when used in preparation of the suppressant and when used a preservative. Protective eyeware, clothing and gloves should be worn when handling.

EQUIPMENT AND SUPPLIES

5.1 Ion chromatograph (IC): Dionex (Model ICS-2000; S/N 04020527)

This analytical system is complete with an EluGen II Potassium Hydroxide (KOH) Cartridge, an deionized (DI) water bottle with high purity of DI water (Resistivity >18.0 megohm-cm), an AS40 Automated Sampler, an ion chromatographic pump, injection valves, both guard and analytical separator columns, column heater, chemical suppressor, conductivity detector, and computer based data acquisition and process called CHROMELEON system (Dionex). Ion chromatograph

5.1.1 Anion guard columns – Dionex IonPac AG19, 2×50 mm (P/N 062888). These guard columns function as a protector of the separator columns and packed with a substrate identical as that used in the corresponding separator column.

5.1.2 Anion separator columns. - Dionex IonPac AS19 Analytical column, 2×250 mm (P/N 062886). using the conditions outlined in Table 2.

5.1.3 Anion suppressor device – Dionex AERS 500 self-regenerating chemical suppressor (P/N 0082541). This built-in control for electrolytic Auto Suppressor eliminates the need to hand-prepare the acidic regenerant. Adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 0.5 nS per minute over the background conductivity.

5.1.4 Detector – Dionex Conductivity DS3 Detector.

5.1.5 ASDV Automated Sampler

5.1.6 AutoSampler Sample Vials – 0.5 ml vials equipped with filter caps (Dionex P/N 038010) or 5.0 ml vials equipped with filters (Dionex P/N 038141)

5.2. Data Acquisition System – The Dionex PEAKNET Data Chromatography Software was use to collect and generate all the data.

5.3 Analytical balance (±0.1 mg) – Fisher Scientific (Model ACCU-124D).

5.4 Top loading balance (±10 mg) – OHAUS (Model Scout II).

5.5 Syringes – Glass graduated syringes: 25 µL, 50 µL, 100 µL, 500 µL, 1000 µL.

5.6 Volumetric Pipets; Class A, 2, 4, 5 mL, 10 mL, 25 mL and 50 mL.

5.7 Eppendorf pipettor and tips

5.8 Volumetric Flasks – Class A, various volumes for preparing standards.

5.9 Sampling Containers – Glass or polyethylene, either purchased pre-cleaned or prepared in the laboratory. The containers should be of sufficient volumes to allow replicate analyses of anions of interest.

5.10 Water purification system (E-pure System) – Barnstead International (Model D4641 120 VAC).

5.11 Compressed Nitrogen Gas

5.12 Concentrated HCL – for glassware preparation in the use for oPhosphate-P analysis

6.0 REAGENTS AND STANDARDS

6.1 Reagent water – Deionized water from Direct QUV (Millipore Cat # ZRQSVP030): 18.0 Mohm or better. Or an equivalent Water Purification system.

6.2 Eluant – Dionex Eluent Generator Cartridge – Potassium Hydroxide (EGC III KOH) - Cat# 074532

6.3 Stock Standard Solutions
Stock standard solutions for the preparation of calibration standards, matrix spike solutions, LFB, QCS, are either purchased as certified solutions or prepared from ACS reagent grade materials as listed below.

6.3.1 Primary Source Stock standard solutions
The Primary source stock standard solutions are used for the preparation of calibration standards and are purchased from Inorganic Ventures as listed below:

	ppm	Catalogue #
Bromide (Br^-)	1000	ICBR1-1
Chloride (Cl-)	10,000	ICCL10
Fluoride (F^-)	1000	ICFL-1
Nitrate as Nitrogen (NO_3-N)	1000	ICNNO31-1
Nitrite as Nitrogen (NO_2-N)	1000	ICNNO21-1
Phosphate as Phosphorus ($PO^{=}_4$-P)	1000	ICPPO41-1
Sulfate ($SO_4^{=}$)	10,000	ICSO410

6.3.2 Secondary Source Stock standard solutions
These secondary source stock standard solutions are used for preparing the quality control check solutions (QCS). Any secondary stock solution chosen to be used for the QCS must be from a different manufacturing source or lot number that is being used as a primary source.
These secondary source standards are either purchased as certified solutions or prepared from ACS reagent grade materials as listed below:

6.3.2.1 Purchased Secondary stock standard solutions are used for the preparation of calibration standards and are purchased from UltraScientific as listed below:

	ppm	Catalogue #
Bromide (Br^-)	1000	ICC-001 (100ml)
Chloride (Cl-)	1000	ICC-002 (100ml)
Fluoride (F^-)	1000	ICC-003 (100ml)
Nitrate as Nitrogen (NO_3-N)	1000	ICC-004A (100 ml)
Nitrite as Nitrogen (NO_2-N)	1000	ICC-007A (100ml)
Phosphate as Phosphorus ($PO^{=}_4$-P)	1000	ICC-005A (100ml)
Sulfate ($SO_4^{=}$)	1000	ICC-006 (100ml)

6.4 Once standards are purchased and received, or prepared, they are logged in the Primary Standard Logbook with date of receipt, name of vendor, catalog number, expiration date and a primary standard ID assigned. Purchased chemicals with Certificate of Analyses provided by the vendor will have the laboratory assigned primary standard ID, date and the receiving analyst initials. The bottle will also be identified with primary standard ID and the date received and the analyst initials.

An example of the Logbook is attached (Figure 1).

Primary standard ID is labeled as IP mmddyy X:

Where: IP = Inorganic Primary

mmddyy = the date the standard is received

X = the order that the standard is logged into the logbook on that date in increasing alphabetical order.

6.5 Preparation of Calibration Standards – For each analyte of interest, intermediate calibration standards are prepared by first adding measured volumes of one or more stock standards (Section 6.3.1.) to volumetric flasks and diluting to volume with reagent water. These intermediate calibration standards are then further used to prepare the daily working calibration standards. This laboratory separates the calibrations into the following analytes to be determined within a sample run.

Note : Dilute working standards should be prepared weekly, except those that contain nitrite and phosphate should be prepared daily.

6.5.1 Fluoride, Chloride, NitriteN, NitrateN & Sulfate - Combined

6.5.1.1 Intermediate Calibration Standard –
Fluoride, Chloride, NitriteN, NitrateN & Sulfate - Combined

Combine the following aliquots of stock solutions are combined together in a 500 ml volumetric flask. Stable for 1 month.

a 6.25 mL of 1000 ppm Fluoride stock standard (Section 6.3.1.) to yield 12.5 mg/L Fluoride.

b. 12.5 mL of 10,000 ppm Chloride stock standard (Section 6.3.1.) to yield 250 mg/L Chloride.

c. 6.25 mL of 1000 ppm Nitrite-N stock standard (Section 6.3.1.) to yield 12.5 mg/L Nitrite as Nitrogen.

d. 12.5 mL of 1000 ppm Nitrate-N stock standard (Section 6.3.1.) to yield 25 mg/L Nitrate as Nitrogen.

e. 12.5 mL of 10,000 ppm Sulfate stock standard (Section 6.3.1.) to yield 250 mg/L Chloride.

6.5.1.2 Working Calibration Standards – a minimum of 6 levels are needed for construction a curve. Prepared Daily.

There are six concentration levels for the calibration curve for F, Cl, NO2-N, NO3-N, SO4 as follows :

ppm	Fluoride	Chloride	Nitrite-N	Nitrate-N	Sulfate
Level 1	0.050	1.00	0.05	0.10	1.00
Level 2	0.10	2.00	0.10	0.20	2.00
Level 3	0.50	10.0	0.50	1.00	10.0
Level 4	1.25	25.0	1.25	2.50	25.0
Level 5	2.50	50.0	2.50	5.00	50.0
Level 6	5.00	100	5.00	10.0	100

The working standards are prepared via dilutions starting with the combined Cl, NO2-N, NO3-N, SO4 Combined Intermediate Standard (Section 6.5.1.1.)

Level 6 – 20.0 ml of Intermediate Calibration Combined Standard to 50 mL

Level 5 – 10 ml of Intermediate Calibration Combined Standard to 50 mL

Level 4 – 5.0 ml of Intermediate Calibration Combined Standard to 50 mL

Level 3 – 2.0 ml of Intermediate Calibration Combined Standard to 50 mL

Level 2 – 10 ml of Level 3 to 50 ml

Level 1 – 5.0 ml of Level 3 to 50 ml

6.5.2 o-PO4-P

6.5.2.1 Intermediate Calibration Standard.
5 mL of 1000 ppm oPO_4-P stock standard (Section 6.3.1) in a 500 ml volumetric flask to yield 10.0 mg/L oPO_4-P.

6.5.2.2 Working Calibration Standards – Prepared Daily
There are six concentration levels for the calibration curve for oPO_4-P as follows :

	oPO_4-P (ppm)
Level 1	0.05
Level 2	0.10
Level 3	0.25
Level 4	0.50
Level 5	1.00
Level 6	2.50

The working standards are prepared via serial dilutions starting with the oPO_4-P Intermediate Standard.

Level 6 - 25 ml of Intermediate Standard to 100 ml
Level 5 – 5.0 ml of Intermediate Standard to 50 ml
Level 4 – 5.0 ml of Intermediate Standard to 100 ml
Level 3 - 5.0 ml of Level 6 to 50 ml
Level 2 – 5.0 ml of Level 5 to 50 ml
Level 1 – 5.0 ml of Level 4 to 50 ml

6.5.3 Bromide

6.5.3.1 Intermediate Calibration Standard -
10 mL of 1000 ppm Bromide stock standard (Section 6.3.1.) in a 100 ml volumetric flask to yield 100 mg/L. Bromide

6.5.3.2 Working Calibration Standards

There are seven concentration levels for the calibration curve for Bromide as follows (with Level 1 as the reporting limit concentration). Prepared Daily.

	Bromide (ppm)
Level 1	1.0
Level 2	2.5
Level 3	5.0
Level 4	10.0
Level 5	25.0
Level 6	50.0
Level 7	100.0 (Intermediate if High Concentrations is expected)

The working standards are prepared via serial dilutions starting Intermediate Standard.

Level 7 – Intermediate Calibration Standard (if high concentration is expected)
Level 6 – 25.0 ml of Intermediate Standard to 50 ml
Level 5 – 25.0 ml of Intermediate Standard to 100 ml
Level 4 – 10.0 ml of Intermediate Standard to 100 ml
Level 3 – 5.0 ml of Intermediate Standard to 100 ml
Level 2 – 2.5 ml of Intermediate Standard to 100 ml
Level 1 – 1.0 ml of Intermediate Standard to 100 ml

6.6 After the working calibration standards are made they are logged in the Working Standard Logbook with date of preparation, initial concentration, amount taken, final volume, final concentration, solvent used, expiration date , analysts initials and assigned an Working Standard ID (see Figure 2).

Working standard ID is labeled as IW mmddyy X:

Where: IW = Inorganic Working

Mmddyy = the date the standard is made.

X = the order that the standard is made on that date in increasing alphabetical order.

6.7 Preparation of Quality Control Check (QCS) solutions
These quality control check solutions are prepared using the secondary source stock standard solutions (Section 6.3.2) to verify new calibration curves and continual verification on a quarterly basis.

6.7.1 For Fluoride, Chloride, NitriteN, NitrateN & Sulfate QCS

6.7.1.1 Fluoride, Chloride, NitriteN, NitrateN & Sulfate - Combined

Using the Secondary Stock Standards, Refer to Section 6.5.1.1. for the preparation of the Combined Intermediate Standard for F, Cl, NO2N, NO3N & SO4

6.7.1.2. Using this Intermediate Combined Standatd, follow the same preparation procedure as outlined in Section 6.5.1.2. for the preparation of the following Level 5 Concentrations :

ppm	Fluoride	Chloride	Nitrite-N	Nitrate-N	Sulfate
Level 5	2.50	50.0	2.50	5.00	50.0

6.7.2. For o-PO4-P QCS

6.7.2.1. Intermediate oPO_4-P Standard
Using the Secondary Stock Standard, refer to Section 6.5.2.1. for the preparation of the Intermediate Standard for oPO_4-P

6.7.2.2. From this intermediate standard the QCS is prepared by pipetting a 5 mL aliquot into a 50 mL volumetric flask and diluted to the mark with deionized water to yield a 1.0 ppm solution

6.7.3. For Bromide QCS

6.7.3.1. Intermediate Br Standard
Using the Secondary Stock Standard, refer to Section 6.5.3.1. for the preparation of the Intermediate Standard for Bromide

6.7.3.2. From this intermediate standard the QCS is prepared by pipetting 25 mL into in a 100 ml volumetric flask to yield 25 mg/L.

6.8. Preparation of Instrument Performance Check Solution (IPC)
These quality control check solutions are prepared using standards solutions (Section 6.5) at the mid-range concentrations of the calibration curve and is used to verify the curve on an on-going basis during the sample sequence run.

6.8.1. For Fluoride, Chloride, NitriteN, NitrateN & Sulfate IPC

6.8.1.1. Using the Fluoride, Chloride, NitriteN, NitrateN & Sulfate - Combined Intermediate Standard prepared in Section 6.5.1.1. ; Prepare the Level 5 concentration as outlined in Section 6.5.1.2. to yield the following concentrations:

ppm	Fluoride	Chloride	Nitrite-N	Nitrate-N	Sulfate
Level 5	2.50	50.0	2.50	5.00	50.0

6.8.2. For oPO4-P IPC

6.8.2.1. Using the oPO4-P Intermediate Standard prepared in Section 6.5.2.1. ; Prepare the Level 5 concentration as outlined in Section 6.5.2.2. to yield a 1.0 ppm concentration.

6.8.3. For Bromide IPC

6.8.3.1. Using the Bromide Intermediate Standard prepared in Section 6.5.3.1. ; Prepare the Level 4 concentration as outlined in Section 6.5.3.2. to yield a 25 ppm concentration.

6.9 Preparation of Laboratory Fortified Blanks (LFB)

6.9.1. For Fluoride, Chloride, NitriteN, NitrateN & Sulfate LFB

Using the Fluoride, Chloride, NitriteN, NitrateN & Sulfate - Combined Intermediate Standard prepared in Section 6.5.1.1. ; Prepare the Level 4 concentration as outlined in Section 6.5.1.2. to yield the following concentrations:

ppm	Fluoride	Chloride	Nitrite-N	Nitrate-N	Sulfate
Level 4	1.25	25.0	1.25	2.50	25.0

6.9.2 For o-PO4-P LFB

Using the Intermediate Calibration Standard (Section 6.5.2.1.) pipette 5 ml into a 200 mL volumetric flask and dilute to the mark with deionized water. This yields a 0.5 ppm LFB solution.

6.9.3 For Bromide LFB
Using the Intermediate Calibration Standard (Section 6.5.3.1.) pipette 25 ml into a 100 mL volumetric flask and dilute to the mark with deionized water. This yields a 25 ppm LFB solution

6.10 Preparation of Matrix Spike (MS) solution used for fortifying samples

6.10.1. Fluoride, Chloride, Nitrite-N, Nitrate-N, Sulfate - Combined MS

6.10.1.1. Using the Fluoride, Chloride, NitriteN, NitrateN & Sulfate - Combined Intermediate Standard prepared in Section 6.5.1.1. ; Prepare the Level 5 concentration as outlined in Section 6.5.1.2. to yield the following concentrations:

ppm	Fluoride	Chloride	Nitrite-N	Nitrate-N	Sulfate
Level 5	2.50	50	2.5	5.0	50

6.10.2. o-PO4-P
Using the Intermediate Calibration Standard (Section 6.5.2.1.) pipette 1 ml into a 100 mL volumetric flask and dilute to the mark with deionized water. This yields a 1.0 ppm matrix spike solution

6.10.3. Bromide
2.5 mL of 1000 ppm Bromide stock standard (Section 6.3.2.) pipetted into in a 100 ml volumetric flask to yield 25 mg/L.

6.10.4. Sample Fortification is taking equal amounts of sample and MS as prepared in Sections 6,10.1, 6.10.2. & 6.10.3. and running this solution on the IC.

6.11. Preparation of Low Level Check Standard (LLC)

6.11.1 Fluoride, Chloride, Nitrite-N, Nitrate-N, Sulfate - LLC
Use the Level 1 as prepared in the Combined Calibration Standard (Section 6.5.1)

Fluoride	Chloride	Nitrite-N	Nitrate-N	Sulfate
0.05	1.00	0.05	0.10	1.00 ppm

6.11.2 o-PO4-P - LLC
Use the Level 1 as prepared in Section 6.5.2.2. (0.05 ppm)

6.11.3 Bromide - LLC
Use the Level 1 as prepared in Section 6.5.3.2. (1.0 ppm)

7.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1 Samples are collected in plastic or glass bottles that have been either purchased pre-cleaned or prepared in the laboratory by thoroughly cleaning and rinsing bottles (Section 5.9.) sufficiently with reagent water (Section 6.1.). The volume collected is sufficient to insure a representative sample and allow for replicate analysis and fortification if necessary.

7.2 Samples are shipped iced or stored cold in a cooler at ≤ 4.0 °C. The laboratory will not accept samples whenever the sample bottle has been violated (i.e. loose or broken cap, leaking bottle, improperly labeled), causing concern for contamination.

7.3 Following are the sample preservation and holding times :

Analyte	Preservation	Holding Time
Bromide	None Required	28 days
Chloride	None Required	28 days
Fluoride	None Required	28 days
Nitrate-N	Cool to 4.0 °C	48 hours
Nitrite-N	Cool to 4.0 °C	48 hours
Combined Nitrate/Nitrite*	conc. H_2SO_4 to pH <2	28 days
o-Phosphate-P	Cool to 4.0 °C	48 hours
Sulfate	Cool to 4.0 °C	28 days

*Note: If the determined value for the combined nitrate/nitrite exceeds 0.5 mg/L as N, a resample must be analyzed for the individual concentrations of nitrate & nitrite.

7.4 Allow any cooled sample to come to room temperature before analysis. In the case of ortho-phosphate it has been observed that degradation occurs in samples that have been held at room temperature for over 16 hrs.

8.0 QUALITY CONTROL

Consists of an initial demonstration of laboratory capability and the on-going assessment of the quality of the data being generated by analysis of laboratory reagent blanks, fortified blanks, quality control samples, and the determination of analyte recoveries. The generated performance records are kept on file and available for review for ten years in accordance this laboratory's QA/QC plan.

8.1 INITIAL DEMONSTRATION OF PERFORMANCE - Refer to Table 4

8.1.1 The instrument's performance and the laboratory's performance is assessed prior to conducting any analyses. The Instrumentation Performance is characterized via the determination of Linear Calibration Range (LCR) and analysis of Quality Control Samples (QCS) The laboratory performance is characterized via the determination of MDL's. (see Table 1).

8.1.2 Linear Calibration Range (LCR) – The LCR is determined initially The verification of linearity uses a blank and a minimum of three standards prepared in the following concentrations listed as below. If the verification data exceeds the initial values by ± 10%, linearity is re-established. Any non-linear portion of the defined range is nonlinear, then additional standards are used to define the nonlinear portion. Refer to Section 6.5. for the preparation of the Calibration Standards.

8.1.3 Quality Control Sample (QCS) – When first beginning this method, the calibration standards and instrumentation performance is verified by analyzing a QCS from a second source. If the determined concentration are not within ±10% of the expected values, performance of the determinative step of the method is unacceptable. The source of the

problem is identified and corrected before proceeding with the initial determination of MDL's

8.1.4. Method Detection Limit (MDL) – MDL's are established for all analytes using reagent water (blank) fortified at concentrations of two-to-three times the estimated instrumentation detection limit. To determine the MDL values, seven replicate aliquots of the fortified reagent water are analyzed and concentrations determined over a period a minimum of 3 days. The fortified concentrations and preparation procedures used for the analytes are listed as follows:

Analyte	mg/L	Procedure Section
Bromide	1.00	Section 6.5.3. - Level 1
Chloride	1.00	Section 6.5.1. - Level 1
Fluoride	0.05	Section 6.5.1. - Level 1
Nitrate-N	0.10	Section 6.5.1. - Level 1
Nitrite-N	0.05	Section 6.5.1. - Level 1
o-Phosphate-P	0.05	Section 6.5.2. - Level 1
Sulfate	1.00	Section 6.5.1. - Level 1

For each analyte , calculate the MDL as follows:

$MDL = (t) \times (S)$

Where:

t = Student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.
For 7 replicates **t** =3.14
If more replicates are used, use the corresponding **t**- value

S = standard deviation of the replicate analyses

MDL's are determined every at least every six months or whenever a significant change in the background or instrument response is detected or expected and kept on file for 10 years. (See example in Table 1).

8.2 ASSESSING LABORATORY PERFORMANCE

8.2.1 Accuracy & Precision Studies (A&P)

Annually, the accuracy & precision of each element is determined.

To establish this accuracy & precision for each element, a minimum of seven replicate analyses of a mid-range Calibration Standard is analyzed.

Use the listed Calibration Standards concentrations for the A&P's studies

Anion	ppm	Level Standard	See Section
Bromide	50	Level 5	Section 6.5.3
Chloride	50	Level 5	Section 6.5.1.
Fluoride	2.5	Level 5	Section 6.5.1.
Nitrate-N	5.0	Level 5	Section 6.5.1.
Nitrite-N	2.5	Level 5	Section 6.5.1.
o-Phosphate-P	1.0	Level 5	Section 6.5.2.
Sulfate	50	Level 5	Section 6.5.1.

The accuracy for each analyte is measured by determining the % Recovery of the seven results using the following calculation :

$$\%REC = \frac{(C_s - C)}{S} \times 100$$

Where:

% REC = percent recovery,
C_s = average of the seven determinations
C = concentration of prepared analyte

The Precision for each analyte is expressed as the standard deviation estimate with n-1 degrees of freedom of the seven replicate results and kept on file for 10 years (See example in Table 2)

8.2.2. Method Detection Limits (MDL)
Annually, and every six months or whenever a significant change in the background or instrument response is detected or expected the MDL's are established for all analytes. To determine the MDL values see Section 8.1.4.

8.2.3. Analyte Recovery and Data Quality – Refer to Table 5
On an on-going basis, the laboratory's performance is continually assessed.

8.2.3.1 Laboratory Fortified Blank (LFB) - At least one LFB is analyzed with each batch of 20 samples. The accuracy is calculated as percent recovery (Section 8.2.3.1.1.). If the recovery of any analyte falls outside the required control limits of 90-110%, then that analyte is considered to be out of control and the source of the problem is determined and resolved before continuing analyses. Following are the concentrations of the LFB (preparation procedure : see Section listed below).

Analyte	mg/L	Procedure Section- conc. Level
Bromide	25.0	Section 6.5.3. - Level 5
Chloride	25.0	Section 6.5.1. - Level 5
Fluoride	1.25	Section 6.5.1. - Level 5
Nitrate-N	2.50	Section 6.5.1. - Level 5
Nitrite-N	1.25	Section 6.5.1. - Level 5

o-Phosphate-P	1.00	Section 6.5.2. - Level 5
Sulfate	25.0	Section 6.5.1. - Level 5

8.2.3.1.1. Calculation of Percent Recoveries - calculate the percent recovery for each analyte, corrected for concentration measured in the unfortified sample. These values are compared to the determined LFM recovery range of 90-110-%.

The percent recovery is calculated as follows:

$$\%REC = \frac{Cm}{C} \times 100$$

where,
% REC = percent recovery,
C_m = measured fortified sample concentration,
C = prepared fortified sample concentration,

8.2.3.1.2 The LFB analyses data is used to assess the laboratory's performance against the required control limits of 90-110%. When enough internal performance data is available (minimum of 25 analyses) control limits are established for each analyte. These upper and lower control limits are determined from the percent mean recovery (x) and the standard deviation (S) and are established as follows :

UPPER CONTROL LIMIT = x + 3S

LOWER CONTROL LIMIT = x - 3S

These control limits must be equal to or better than the required control limits of 90-110%. After each 5-10 new recovery measurements, new control limits are calculated on the most recent 25 data points.

In addition, the standard deviation (S) data is used to establish an on-going performance statement for the level of concentrations included in the LFB. These data are kept on file and are available for review.

8.2.3.1.3 These results are incorporated into the on-going Control Charts to document data quality as outlined in Section 8.2.4. and are available for review for 10 years.

8.2.3.2. Laboratory Fortified Sample Matrix (LFM) – The laboratory adds a known amount of the analyte to a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. This is accomplished by adding equal volumes of the sample to be fortified with an equal amount of the following concentrations then followed by pouring and the necessary portion of such to be analyzed. The concentration of each analyte added is as follows with the preparation procedure section as listed:

Anion	MDL x4*	mg/L MS	Final Conc	Procedure Section
Bromide	0.4	25	12.5	Section 6.9.3
Chloride	4.0	50	25	Section 6.9.1
Fluoride	0.4	5.0	1.25	Section 6.9.1
Nitrate-N	0.4	5.0	2.5	Section 6.9.1
Nitrite-N	0.2	2.5	1.25	Section 6.9.1
o-Phosphate-P	0.2	1.0	0.5	Section 6.9.2
Sulfate	4.0	50	25	Section 6.9.1

In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The added analyte concentration is the same that is used in the laboratory fortified blank (The analyte concentration must be high enough to be detected above the original sample and not less than four times the MDL*). If the concentration of the fortification is less than 25% of the background concentration of the matrix the matrix recovery is not calculated.

8.2.3.2.2. Calculation of Percent Recoveries - calculate the percent recovery for each analyte, corrected for concentration measured in the unfortified sample. These values are compared to the determined LFM recovery range of 90-110- %.

The percent recovery is calculated as follows:

$$\%REC = \frac{(C_s - C)}{S} \times 100$$

Where :

% REC = percent recovery,
C_s = measured in the fortified sample,
C = measured sample concentration,
S = concentration equivalent of analyte added to sample.

Until sufficient becomes available (minimum of 20 analysis) assess the laboratory performance against recovery limits of 80-120%.
When sufficient data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery.

8.2.3.2.3. If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that all other QC performance criteria is acceptable, the accuracy problem encountered with the fortified sample is judged to be matrix related, not system related.
Repeated failure to meet suggested recovery criteria indicates potential problems with the procedure and will be investigated.

8.2.3.2.4. These results are incorporated into the on-going Control Charts to document data quality as outlined in Section 8.2.4 and are available for review for 10 years.

8.2.3.3 Laboratory Sample Duplicates – The laboratory analyzes sample duplicate for a minimum of 10% of the collected samples or at least one with every analysis batch, whichever is greater. These results are incorporated to the on-going control charts to document data quality.

Calculate the relative percent difference (RPD) of the initial quantitated concentration (I_c) and duplicate quantitated concentration (D_c) using the following formula

$$RPD = \frac{|(I_C - D_c)|}{([I_C + D_c]/2)} \times 100$$

Where:

RPD = Relative Percent Difference

I_c = initial quantitated concentration

Dc = duplicated quantitated concentration

Duplicate analysis may exhibit matrix dependence. If the RPD for the duplicate measurements falls outside ± 20% and if all other QC performance criteria are met, laboratory precision is out of control for the sample and perhaps the analytical batch. The result for the sample and duplicate will be labeled as suspect/matrix to inform the data user that the result is suspect due to a potential matrix effect, which led to poor precision. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses), it indicates a problem with the instrument or individual technique that must be corrected.

8.2.3.4. Laboratory Fortified Blank Duplicates - Quarterly, replicates of the LFB's are analyzed to determine the precision of the laboratory measurements. The RPD is determined as outlined above in Section 8.2.3.3. These results are incorporated to the on-going duplicate (precision range) control charts to document data quality.

8.2.4 QC CONTROL CHARTS

Two types of control charts are used for the continued assessment of the lab's performance :

(1) Accuracy , or Means, Control Chart
(2) Precision , or Range, Control Chart

8.2.4.1 The Accuracy Chart is constructed using the most recent 25 LFB and sample MS %Recovery results. See Section 8.2.3.1.1 for Calculation of %Recoveries for the LFB and Section 8.2.3.2.2 for the MS (see Section 6.10 for MS preparation). The upper and lower warning limits (WL) use ± 2SD and the upper and lower control limits (CL) use ± 3 SD .

8.2.4.2 The Precision Chart is constructed using the most recent 25 Sample & Sample Duplicate RPD results. See Section 8.2.3.3 for the calculation of RPD. The warning limits (WL) use ± 2SD and the control limits (CL) use ± 3 SD.

8.2.4.3. Application of Control Charts.

8.2.4.3.1. Trending – If seven successive samples are on the same side of the central line of the Accuracy Chart, discontinue analyses, investigate and correct the problem

8.2.4.3.2. Control Limit – If one measurement exceeds a CL, repeat the analysis immediately. If the repeat measurement is within the CL, continue analyses, if it exceeds the CL, discontinue analyses, investigate and correct the problem.

8.2.4.3.3. Warning Limit – If two out three successive points exceed a WL, analyze another sample. If the next point is within WL, continue analyses. If the next point exceeds the WL, evaluate potential bias and correct the problem.

8.2.5 The following items must be included in every sample batch or periodically to continually assess the laboratory's performance. See Table 5. A batch of samples is established as 20 samples:

Calibration Curve – Curve run a minimum of weekly with fresh standards for Chloride, Fluoride, Nitrate-N & Sulfate, fresh standards daily for Nitrite-N and oPO4. New curve is verified with QCS. See Section 6.5. for the preparation of the calibration standards. See Section 6.7 for preparation of QCS.

Instrument Blank (IB) – to verify system clear of residual artifacts & contaminants

Instrument Performance Check Solution (IPC)- a mid-range check standard after calibration, every 10 samples and at end of sample sequence run

Laboratory Reagent Blank (LRB) – after IPC in beginning, every 10 samples after CCS, and at end of sample sequence run.

Laboratory Fortified Blank (LFB) – one per batch of samples (every 20 samples)

Laboratory Fortified Sample Matrix (LFM) – a minimum of 10 % of sample sequence run.

Sample duplicates – a minimum of 10% of sample run

LFB Duplicates - Quarterly

Low Level Check Standard (LLC) - Quarterly

MDL's – every 6 months

8.2.5.1 Instrument Performance Check Solution (IPC) – The laboratory analyzes the IPC of the following concentrations after the Instrument & Calibration blanks are run at the beginning of the day's sample sequence, after every tenth sample and at the end of the sample run. The procedures for preparing the IPC is listed under the listed sections.

Analyte	mg/L	Procedure Section
Bromide	50.0	Section 6.5.3 - Level 5
Chloride	50.0	Section 6.5.1 - Level 5
Fluoride	2.5	Section 6.5.1 - Level 5
Nitrate-N	5.0	Section 6.5.1 - Level 5
Nitrite-N	2.5	Section 6.5.1 - Level 5
o-Phosphate-P	1.0	Section 6.5.2 - Level 5
Sulfate	50	Section 6.5.1 - Level 5

Subsequent analyses of the IPC must verify that the calibration is still within $\pm$10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC. If the second analysis of the IPC confirms the calibration to be outside the limits, sample analysis must be stopped, the cause determined. All samples following the last acceptable IPC must be reanalyzed.

8.2.5.2 Laboratory Reagent Blank (LRB) – An LRB is prepared and treated exactly as a typical field sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with field samples. Data produced are used to assess contamination from the laboratory environment.
Values that exceed the MDL indicate a laboratory or reagent contamination is present. The source of the contamination must be determined prior to conducting any sample analysis.
Any sample included in an automated analysis batch which has an invalid LRB, indicated by a quantitated result that exceeds the MDL, must be reanalyzed in a subsequent analysis batch after the contamination problem is resolved.

8.2.5.3 Laboratory Fortified Blank (LFB) – Refer to Section 6.9. for preparation procedure and Section 8.2.3.1 for use in on-going laboratories' QC/QA performance.

8.2.5.4 Laboratory Fortified Sample Matrix (LFM) – Refer to Sections 6.10.1,6.10.2,& 6.10.3, for the preparation of the MS used for fortifying the samples and section 6.10.4. for the Sample Fortification procedure. Refer to Section 8.2.3.2 for use in on-going laboratories' QC/QA performance.

8.2.5.5 Sample Duplicates – Refer to Section 8.2.3.3. and 8.2.3.4.

8.2.5.6 Low Level Check (LLC) – Quarterly, the lowest level standard (MDL) is analyzed to demonstrate the ability to analyze low level samples. Refer to Section 6.11 for preparation procedure.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Establish ion chromatographic operating parameters indicated in Table 2.

9.2 Run the initial calibration using the standards made in Section 6.5. Using injections of 25 microliters (determined by the injection loop volume) of each prepared calibration standard.

9.2.1 The initial calibration is deemed acceptable if the following criteria are met (Table 4):
$R \geq 0.9950$

9.2.2 The calibration curve is verified by analyzing a QCS (Section 6.7) immediately after the initial calibration. The acceptable limit of the QC sample is 90% - 110%.

9.2.3 Once the initial calibration and QCS are done, one blank, one LFB and ten samples could be analyzed. Following the ten samples, a IPC is analyzed as a closing instrument verification (Section 9.3).

9.3 At the beginning of any sequence except for the samples right after initial calibration (Section 9.2.3), IPCs are always analyzed at the beginning of the sequence and the end of every ten samples to confirm the instrument is acceptable.

9.3.1 The concentration of the IPC used for the separate analytes are as follows. The procedure for making these standards are the same as those from making the indicated concentration levels of the initial calibration standards (Sections 6.5) but from a separate (secondary) source as those stock solutions as used in the making of the calibration standards

Analyte	mg/L	Procedure Section
Bromide	50	Section 6.5.3.2
Chloride	50	Section 6.5.1.2
Fluoride	2.5	Section 6.5.1.2
Nitrate-N	5.0	Section 6.5.1.2
Nitrite-N	2.5	Section 6.5.1.2
o-Phosphate-P	1.0	Section 6.5.2.2
Sulfate	50	Section 6.5.1.2

9.3.1.2 The IPC concentration must fall within ± 10% of the stated value. If the response or retention time for any analyte varies from the expected values by more than ±10%, the test is repeated, using fresh IPC standards. If the results are still more than ± 10 %, a new calibration curve must be prepared for that analyte.

9.4 End of Run IPC- at the end of the sample run sequence

9.5 End of Run Blank – at the very end run of the day – an instrumentation blank is run using reagent deionized water

10.0 PROCEDURE

10.1 Samples Preparation

10.1.1 See Section 7.3 for sample storage & handling conditions. Those samples that require refrigeration, ensure the samples have come to room temperature prior to conducting sample analysis.

10.1.2 Samples Pretreatment - The pretreatments prescribed are effective at reducing the chloride and sulfate content of a sample matrix but will not reduce matrix concentrations of other anions such as nitrate or phosphate

10.1.2.1 If the Chloride concentration interferes with the determination of NO2 or NO3 then pre-treat the sample using a Ag pretreatment cartridges to remove the Chloride (Dionex P/N 057089).

10.1.2.2 If the Sulfate concentration interferes with the determination of oPO4 then pre-treat the sample using Ba pretreatment cartridges to remove the sulfate (Dionex P/N 057093).

10.1.2.3 Samples Pretreatment Procedure
Individually and thoroughly rinse each pretreatment cartridge with reagent water in order to insure all residual background contaminants are removed from the cartridge. Filter 3 mL of sample through the series of rinsed cartridges as an initial sample rinse (Ba, Ag) at a flow rate of 1.0 mL/min or less (approximately one drop every 3 to 4 seconds). This flow rate is critical to the pretreatment and must be carefully followed. Discard this fraction and begin collecting the pretreated sample aliquot of collected sample.

10.1.2.4 Pour approximately 0.75 ml sample into 0.5ml autosampler vail (or 6 ml into 5 ml autosampler vail) and place a filter cap into the vial and push down the cap with a special made tool from Dionex to certain position according to instructions provided by the Manufacturer. There is no need to filter the sample since the cap has a filter in it.

10.1.3 Prior to pretreating any field samples, prepare and pretreat both an LRB and an LFB. These pretreated quality control samples are required when an analysis batch contains a matrix that must be pretreated. The pretreated LRB and LFB are used to verify that no background interference or bias is contributed by the pretreatment. If a response is observed in the pretreated LRB, triple or quadruple the volume of reagent water rinse used and repeat until a blank measures no more than ½ the MRL. If this additional rinsing procedure is required, it must be consistently applied to all the cartridges prior to conducting any matrix pretreatment.

10.1.4 Solid Samples - The following extraction should be used for solid materials. Add an amount of reagent water equal to 10 times the weight of dry solid material taken as a sample. This slurry is mixed for 10 minutes using a magnetic stirring device. Filter the resulting slurry using a 0.45u membrane type filter. Ensure that good recovery and peak identification is obtained through the use of fortified samples.

10.2. Sample Analysis

10.2.1. Table 2 summarizes the operating conditions for the ion chromatograph. Included in this table are a representative retention time and MDL results for the analytes that has been achieved by this method

10.2.2 Verify the initial calibration by conducting a QCS. See Section 6.7 for the preparation of the QCS using stock solutions obtained from a secondary source - either purchased or prepared from reagent grade chemicals (Section 6.3.2.).

10.2.3 The injection volume is 25 microliters that is controlled by using 25 microliters sample loop (Dionex P/N: 052682). Use the same size loop for standards and samples. An AS40 Automated Sampler (Dionex P/N: 056830) is used. Data acquisition and processing are done using CHROMELEON CHM-1-IC/Win 2000 Desktop Workstation (Dionex P/N: 060929).

10.2.4 The retention time window used to make identifications in the laboratory is ± 0.2 minutes (determined by ±3 Std Dev of the RT of individual analytes over the course of a day)

10.2.5 If the response of a sample analyte exceeds the calibration range, the sample is diluted with an appropriate amount of reagent water and reanalyzed.

10.2.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

10.2.7 An analytical sequence including initial calibration and other quality control analysis for sample analysis is listed in Table 5.

11.0 DATA ANALYSIS, CALCULATIONS AND REPORTS

11.1 Identify the analytes in the sample chromatogram by comparing the retention time of a suspect peak within the retention time window to the actual retention time of a known analyte peak in a calibration standard. The retention time in the daily calibration check standards (QCS) is used for the identification.

11.2 Compute sample concentration using the initial calibration curve generated in Section 8.1.1.

11.3 Report those values that fall between the MRL and the highest calibration standards without any flagging. Sample analytes with responses that exceeds the highest calibration standard concentration are diluted and reanalyzed.

11.4 A printout of the sample sequence is printed out, dated & initialed, and kept in a notebook (Example of sequence run copy is attached – Table 5). Hard copies of the integrated analyses are printed and kept in filing folder indentified by the sequence number.

11.5 Report results in mg/L. The MRL reported is the lowest Calibration Standard Level used

11.6 Report : NO_2^- as Nitrogen

NO_3^- as Nitrogen
HPO_4 as P

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

The laboratory waste management practices are conducted consistent with all applicable rules and regulations as stated in the laboratory's "Sample and Waste Disposal Standard Operating Procedure" , Revision 003 - July 6, 2006). Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner in this SOP.

13.0 REFERENCES

1. U.S. Environmental Protection Agency, "Determination of Inorganic Anions by Ion Chromatography", Method 300.0, Revision 2.1, August 1993

2. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 22th Edition of Standard Methods (2012)

Table 1. Method Detection Limits (MDLs)

MDL Study	**Year**	**2017**				
Analyte	**Fluoride**	**Chloride**	**Nitrite-N**	**Nitrate-N**	**Sulfate**	**Bromide**
Spiking Level (ppm)	**0.050**	**1.00**	**0.050**	**0.100**	**1.00**	**1.00**
Run #1	0.058	1.07	0.049	0.108	1.00	0.962
Run #2	0.081	1.15	0.043	0.116	1.02	0.964
Run #3	0.059	1.27	0.046	0.106	1.54	0.962
Run #4	0.059	1.08	0.052	0.107	1.24	0.958
Run #5	0.054	1.13	0.051	0.101	1.10	0.952
Run #6	0.060	0.98	0.025	0.111	1.00	0.966
Run #7	0.061	1.03	0.061	0.105	1.20	0.974
Average	0.062	1.10	0.047	0.108	1.16	0.962
1 Std Dev	**0.009**	**0.096**	**0.011**	**0.005**	**0.195**	**0.007**
MDL	**0.030**	**0.300**	**0.035**	**0.015**	**0.612**	**0.021**
Reporting MDL	**0.050**	**1.0**	**0.050**	**0.10**	**1.0**	**1.0**
Dates Run	07/07/17	07/07/17	07/07/17	07/07/17	07/70/17	09/17/13
	07/08/17	07/08/17	070/8/17	07/08/17	07/08/17	09/19/13
	07/11/17	07/11/17	07/11/17	07/11/17	07/11/17	09/19/13
Analyst	L.Prior	L.Prior	L.Prior	L.Prior	L.Prior	L.Prior

Table 2. Accuracy and Precision (A&P)

A &P Study	**Year**	**2017**				
Analyte	**Fluoride**	**Chloride**	**Nitrite-N**	**Nitrate-N**	**Sulfate**	**Bromide**
Spiking Level (ppm)	**2.5**	**50.0**	**2.50**	**5.00**	**50.00**	**50.0**
Run #1	2.49	49.8	2.51	4.95	49.6	49.69
Run #2	2.51	49.7	2.51	4.95	49.8	49.55
Run #3	2.50	49.9	2.51	4.97	49.0	49.32
Run #4	2.51	49.9	2.52	4.96	49.0	49.42
Run #5	2.54	50.5	2.53	5.02	49.3	49.59
Run #6	2.55	50.3	2.52	5.01	49.4	49.41
Run #7	2.51	50.5	2.55	5.04	49.3	49.57
Average	2.52	50.1	2.52	4.99	49.4	49.51
% RSD	**0.023**	**0.32**	**0.015**	**0.038**	**0.28**	**0.128**
% Mean	**100.6**	**100.2**	**100.8**	**99.7**	**98.7**	**99.0**
Analysis Dates	01/04/17	01/04/17	01/04/17	01/04/17	01/04/17	9/17/13
Analyst	L.Prior	L.Prior	L.Prior	L.Prior	L.Prior	L.Prior

Table 3. Chromatographic Conditions and Equipment of the Ion Chromatographic Instrument

Ion Chromatograph:	Dionex ICS-2000
Sample Loop:	25 µL
Eluent:	EGC III KOH @ 22.0 mM
Eluent Flow:	0.23 mL/min
Columns:	Dionex IonPac AG19 Guard Column 2×50 mm Dionex IonPac AS19 Analytical column, 2×250 mm
Typical System Backpressure:	1900 psi
Suppressor:	Dionex AERS 500 self-regenerating chemical suppressor @ 16 mA current
Detector:	Dionex DS6 - Detection Stabilizer Conductivity at 16 mA held at a temperature of 30°C . Background Conductivity: 0.2 – 1.0 µs
Total Running Time:	15 minutes

Table 4. Initial Demonstration of Capability and Acceptance Requirements

Reference	Requirement	Specification and Frequency	Acceptance Criteria
Section 8.1.2	Linear Calibration Range (LCR)	Generate calibration curve. At least 5 calibration standards are recommended.	MRL must be no lower than the lowest calibration standard. $R \geq 0.9950$
Section 8.1.3	Quality Control Sample	An external/second source of analyte standard must be run following the initial calibration.	The QCS must be ± 10% of the true value.
Section 8.1.4.	Method Detection Limit (MDL) Determination	Analyze 7 replicate LFBs of the lowest Calibration Standard Level over a period of three days minimum. MDL is determined based on these results.	
Section 8.2.1.	Initial Demonstration of Accuracy and Precision	Analyze 7 replicate LFBs fortified with analyte. Calculate the mean recovered concentration ($C_{\overline{X}}$) and the relative standard deviation (%RSD).	The $C_{\overline{X}}$ must be ± 10% of the true value, and the %RSD must be ≤ 10%.
Section 11.0	Minimum Reporting Level (MRL)	MRL = Chloride, Sulfate = 1.0 mg/L, Nitrate-N,=0.10 mg/L, Nitrite-N= 0.05 mg/L, Fluoride =0.50mg/L, Bromide=1.0 mg/L	The low CAL standard can be lower than the MRL, but the MRL must be no lower than the low CAL standard.

Table 5. Quality Control Requirements

Reference	Requirement	Specification and Frequency	Acceptance Criteria
Section 8.1.2.	Calibration Curve	At least 5 calibration standards are recommended. New Curves should be established each day for Nitrite & oPO4P and at a maximum weekly for F, Cl, NO3N & SO4.	MRL must be no lower than the lowest calibration standard. $R \geq 0.9950$
Section 8.2.5.1.	Initial IPC	Analyze after Instrument and Method blanks	Recoveries must be between 90-110% of fortified level.
Section 8.2.5.1	Continuing IPC and Ending IPC	Analyze after 10 samples and after the last sample in an analysis batch.	Recoveries must fall between 90-110%.
Section 8.2.5.2	Laboratory Reagent Blank (LRB)	Analyze at the beginning, after 20 samples and after the last sample in an analysis batch	The LRB concentration must be $\leq$ the proposed MDL.
Section 8.2.2.1.	Laboratory Fortified Blank (LFB)	Analyzed with each batch of samples (20 or less).	Recoveries must be between 90-110% of fortified level
Section 8.2.5.6	Low Level Check (LLC)	Analyzed Quarterly	Recoveries must be between 70-130% of fortified level
Section 8.2.3.2.	Laboratory Fortified Sample Matrix (LFM)	Must add known amount of analyte to a minimum of 10% of field samples or at least one within each analysis batch.	Recovery must be 80-120%. If fortified sample fails the recovery criteria, label both as suspect/matrix.
Section 8.2.3.3.	Field or Laboratory Duplicates	Analyze either a field or laboratory duplicate for a minimum of 10% of field samples or at least one within each analysis batch. Calculate the relative percent difference (RPD).	RPD must be ±15%.
Section 8.2.3.4.	Laboratory Fortified Blank (LFB) Duplicates	Quarterly replicates of LFB's are run & included on the on-going charts.	Duplicate Recovery must be 80-120%.
Section 8.1.3.	Quality Control Sample	Analyzed Quarterly – from 2nd source	The QCS must be ± 10% of the true value
Section 8.2.2.	MDL Determination	Every six months or whenever a significant change has occurred	

Table 6. Typical Analytical Sequence with Quality Control Requirements

Injection #	Description of Quality Control Standards and Samples	Chapter 2 Acceptance Criteria
	Calibration Blank	
	Level 1 of Initial Calibration	R ≥ 0.9950 Calibration curve to be done each day for NO2N & oPO4P and maximum of weekly for F, Cl, NO3N & SO4
	Level 2 of Initial Calibration	
	Level 3 of Initial Calibration	
	Level 4 of Initial Calibration	
	Level 5 of Initial Calibration	
1	Instrumentation Blank	≤ ½ MDL
2	QCS (after new calibration curve and quarterly)	90 -110%
3	Initial IPC	90 -110 %
4	LRB	≤ ½ MDL
5	LFB (Duplicates Quarterly)	90 -110 %
6	LLC (Quarterly)	70 -130 %
7	MS (Check Periodically)	80 -120 %
8	Sample 1	
9	Sample 1 – Laboratory Duplicate	
10	Sample 1 – LFM	80-120 %
11- 19	Sample 2 to Sample 10	
20	Continuing IPC	90 -110%
21	Blank	≤ ½ MDL
22	Sample 11	
23	Sample 11 – Laboratory Duplicate	
24	Sample 11 – LFM	
25-34	Sample 12 to Sample 20	
35	Continuing IPC	90 -110%
36	Blank	≤ ½ MDL
37	LFB	90 -110%
38	Sample 21	
39	Sample 21 – Laboratory Duplicate	
40	Sample 21 – LFM	
41	Sample 22…. And so forth	
Last Injections	Ending IPC Calibration Blank LRB	Criteria As Above

Figure 1. Inorganics Primary Standards Logbook

Barnstable County Laboratory

Inorganics Primary Standard Logbook

Date	Analyte	Vendor	Catalog No	Lot No	Concentration	Primary Standard ID	Exp Date	Initial

Figure 2. Inorganics Working Standards Logbook

Barnstable County Laboratory

Inorganics Working Standards Logbook

Date	Analyte	Primary Standard ID	Initial Conc	Amt taken	Final Vol	Final Conc	Solvent	Working Standard ID	Exp Date	Initial

Barnstable County Department of Health and the
Environment Laboratory

EPA Method 351.2

STANDARD OPERATING PROCEDURE

For

Determination of Total Kjeldahl Nitrogen in Aqueous Samples by Semi-Automated Colorimetry

(Revision 007)

29 December 2020

	Signature	Date
Analyst: Andrew Barker	Andrew Barker	**December 29, 2020**
Laboratory Director: Dan White		29DEC20

STANDARD OPERATING PROCEDURE (SOP)

For

Determination of Total Kjeldahl Nitrogen in Aqueous Samples
by Semi-Automated Colorimetry

1.0 SCOPE AND APPLICATION

1.1 This SOP provides procedure for determination of total Kjeldahl nitrogen in drinking, ground, and surface waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

1.2 The applicable range is 0.25 – 20 mg/L TKN. The range may be extended with sample dilution.

2.0 SUMMARY OF METHOD

2.1 The sample is heated in the presence of sulfuric acid, H_2SO_4 for three hours. The residue is cooled, diluted to 25 mL and analyzed for ammonia. The digested sample may also be used for phosphorus determination.

2.2 Total Kjeldahl nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$, under the conditions of digestion described.

2.3 Organic Kjeldahl nitrogen is the difference obtained by subtracting the free-ammonia value from the total Kjeldahl nitrogen value.

3.0 DEFINITIONS

3.1 Calibration Blank (CB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analyte .

3.2 Calibration Standard (CAL) – A solution prepared from the primary dilution standard or stock standard solutions.

3.3 Instrument Performance Check Solution (IPC) – A Solution of one or more method analytes or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 Laboratory Fortified Blank (LFB) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 Laboratory Fortified Sample Matrix (LFM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7 Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

3.8 Safety Data Sheets (SDS) [Used to be called as Material Safety Data Sheet (MSDS)] – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 Quality Control Sample (QCS) – A solution of method analytes of known concentrations that is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.11 Stock Standard Solution (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4.0 INTERFERENCES

4.1 High nitrate concentrations (10x or more than the TKN level) result in low TKN values. If interference is suspected, samples should be diluted and reanalyzed.

4.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Barnstable County Health Laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Reference files of Safety Data Sheets (SDS) are available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult SDS.

5.3.1 Sulfuric acid.

5.3.2 Sodium nitroprusside.

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance – Analytical, capable of accurately weighing to the nearest 0.0001g. Fisher Scientific, Model ACCU-124D Dual Range.

6.2 Glassware – Class A volumetric flasks and pipets as required.

6.3 Block Digestor with Tubes – TKN 50 well AIM600 Block/Controller with rack and 100 mL glass digestion tubes (Environmental Express, Item#: SC900).

6.4 Automated Continuous Flow Analysis Equipment – QuickChem 8500 Series 2 Flow Injection Analysis System (LACHAT Instruments, A Hach Company Brand)

6.4.1 LACHAT XYZ Autosampler.

6.5 BD Kjeldahl Digestion Granules from Environmental Express, Item#. 8032178

6.6 Seal Analytical Teardrop Stoppers, Item No. SC9703

7.0 REAGENTS, CHEMICALS AND STANDARDS

7.1 Potassium Sulfate (K_2SO_4). Fisher, Cat No. P305-500

7.2 Copper (II) Sulfate ($CuSO_4$). Fisher, Cat No. AC422871000

7.3 Sulfuric Acid (H_2SO_4). Fisher, Cat No. A300-212

7.4 Sodium Hypochlorite (NaClO). Cat No. 19-546-929

7.5 Sodium Salicylate $C_6H_4(OH)(COO)Na$. Fisher, Cat No. 50-700-6201

7.6 Sodium Nitroprusside [sodium nitroferricyanide dehydrate, $Na_2Fe(CN)_5NO{\cdot}2H_2O$]. Fisher, Cat No. AC21164-1000

7.7 Sodium Phosphate dibasic heptahydrate ($Na_2HPO_4{\cdot}7H_2O$). Fisher, Cat No. AC20651-5000

7.8 disodium EDTA (ethylenediaminetetracetic acid salt). Fisher, Cat No. BP120500

7.9 Sodium Hydroxide (NaOH). Fisher, Cat No. S613-3

7.10 Ammonium Chloride (NH_4Cl). Fisher, Cat No.^{s} AC199975000 and A661-3

7.11 **Reagent Water**: Ammonia free deionized water produced from Millipore Milli-Q Water Purification System.

7.12 **Degassing with Helium**:

7.2.1 To prevent bubble formation, degas the carrier and buffer with helium. Use He at 140 kPa (20 lb/in2) through a helium degassing tube. Bubble helium through one liter of solution for one minute.

7.2.2 All reagents used in heated chemistry must be degassed.

7.13 **Reagent 1: Digestion Solution**

In a 1.0-liter volumetric flask, add 134 g potassium sulfate (K_2SO_4) and 7.3 g copper sulfate ($CuSO_4$) in 800 mL water. Then add 134 mL conc.

Sulfuric acid (H_2SO_4) and dilute to the mark with reagent water. Stir to mix.

7.14 Reagent 2: Hypochlorite Solution

In a 250 mL volumetric flask, dilute 15 mL 5.25% sodium hypochlorite (NaOCl) to the mark with reagent water. Invert to mix.

7.15 Reagent 3: Salicylate Nitroprusside

In a 1.0-liter volumetric flask, dissolve 150 g sodium salicylate [salicylic acid sodium salt, $C_6H_4(OH)(COO)Na$] and 1.0 g sodium nitroprusside [sodium nitroferricyanide dehydrate, $Na_2Fe(CN)_5NO{\cdot}2H_2O$] in about 800 mL reagent water. Dilute to the mark with reagent water and invert to mix. Store in a dark bottle and prepare fresh monthly.

7.16 Reagent 4: Buffer

In a 1.0-liter volumetric flask containing 900 mL reagent water, completely dissolve 35 g sodium phosphate dibasic heptahydrate ($Na_2HPO_4{\cdot}7H_2O$). Next, add 20 g disodium EDTA (ethylenediaminetetracetic acid salt). The EDTA will not dissolve but will form a turbid solution. Finally, add 50 g sodium hydroxide (NaOH), dilute to the mark with reagent water and invert to mix. Degas weekly and prepare fresh monthly.

7.17 Reagent 5: Sodium Hydroxide (0.8M)

In a 1.0-liter volumetric flask, dissolve 32 g sodium hydroxide (NaOH) in about 800 mL reagent water. Dilute to the mark with reagent water and stir to mix.

7.18 Reagent 6: Digestion Diluent (for Carrier and Simulated Standards)

In a 1.0-liter volumetric flask, dissolve 400 mL digestion solution (**Reagent 1**) in about 600 mL reagent water. Dilute to the mark with reagent water and shake to mix.

7.19 Calibration Standards

7.19.1 Stock Standard: 1000 mg/L

In a 1.0 liter volumetric flask, dissolve 3.819 ammonium chloride (NH_4Cl) that has been dried for two hours at 110°C in about 800 mL reagent water. Dilute to the mark with reagent water and invert to mix.

7.19.2 Calibration Standards:

There are six levels calibration standards and their respective concentrations and preparation procedures are listed as follows:

Level	Volume (mL) Taken from Stock Standard (1000 mg/L, Section 7.19.1)	Final Volume (mL) Diluted with Reagent Water	Concentration (mg/L)
6	10	500	20
5	5	500	10
4	2.5	500	5
3	1.25	500	2.5
2	0.125	500	0.25
1	Reagent Water	Reagent Water	0.0

The calibration standards are digested using the same procedures as actual samples.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples are collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected must be sufficient to insure a representative sample, allow for replicate analysis, and minimize waste disposal.

8.2 Samples must be preserved with H_2SO_4 to a pH<2 and cooled to 4°C at the time of collection.

8.3 Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held for up to 28 days.

9.0 QUALITY CONTROL

9.1 Barnstable County Health Laboratory operates a formal quality control (QC) program. The QC program for this method consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks and other laboratory solutions as a continuing check on performance. The laboratory maintains performance records that define the quality of the data that are generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibration ranges and analysis of QCS) and laboratory performance (determination of MDL) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) – The LCR is determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity uses a blank and five calibration standards. If any verification data exceeds the initial values by ±10%, linearity will be reestablished.

9.2.3 Quality Control Sample (QCS) – The QCS is analyzed right after initial calibration (Section 9.2.2) to verify the calibration standards and acceptable instrument performance with preparation and analysis of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL) – MDL must be established using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, seven replicate aliquots of the fortified reagent water are taken, processed and analyzed over a period of a minimum of three days. The spiking level is 0.25 mg/L which is the same as L2 in Section 7.9.2. The following equation is used to calculate the MDL:

$$MDL = (t) \times (S) \qquad (1)$$

Where

t = Student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates, and if more replicates are used, use the corresponding t-value].

S = Standard deviation of the replicate analyses.

9.2.4.1 The Standard deviation (S) can be calculated using the following equation:

$$S = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}} \qquad (2)$$

Where, n = number of samples;
x = concentration in each sample.

9.2.4.2 MDLs must be determined every six months, when a new operator begins work, or whenever there is a significant change in the background or instrument response.

9.2.4.3 One set of MDLs is listed as follows:

Spiking Level =0.25 mg/L							Unit: mg/L			
	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MEAN	STDEV	MDL
DATE	10/12/2017	10/12/2017	10/12/2017	10/19/2017	10/19/2017	10/19/2017	10/25/2017			
Conc	0.313	0.291	0.281	0.302	0.246	0.315	0.352	0.30	0.033	0.103

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) – The laboratory analyzes at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination must be suspected and corrective actions must be taken before continuing the analysis.

9.3.2 Laboratory Fortified Blank (LFB) – The laboratory analyzes at least one LFB with each batch of samples. Calculate accuracy as percent recovery as follows:

$$R = \frac{C_s - C}{S} \times 100 \qquad (3)$$

Where, R = percent recovery;
Cs = recovered fortified blank concentration;
C = blank background concentration;
S = concentration equivalent of analyte added to blank.

9.3.2.1 If the recovery of any analyte falls outside the required control limits of 90-110%, the result is judged out of control, and the source of the problem must be identified and resolved before continuing analysis.

9.3.3 The laboratory also uses LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 25 analyses), optional control limits and control charts can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = x + 3S$$
$$\text{LOWER CONTROL LIMIT} = x - 3S$$

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also the standard deviation (S) data must be used to establish an on-going precision statement for the level of concentration included in the LFB. These data are kept on file and be available for review.

9.3.4 Instrument Performance Check Solution (IPC) – For all determinations the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every 10th sample (or more frequently, if required), and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, the IPC solution is reanalyzed. If the second analysis of the IPC solution confirms calibration to be outside the limits, the sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution are kept on file with the sample analysis data.

9.4 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

9.4.1 Laboratory Fortified Sample Matrix (LFM):

9.4.1.1 The laboratory adds a known amount of analyte to a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. The added analyte

concentration should be the same as that used in the laboratory fortified blank.

9.4.1.2 The percent recovery for TKN is calculated and corrected for concentration measured in the unfortified sample using the following equation:

$$R = \frac{C_s - C}{S} \times 100 \qquad (4)$$

Where, R = percent recovery;
Cs = fortified sample concentration;
C = sample background concentration;
S = concentration equivalent of analyte added to sample.

Acceptable range of R is 90-110%.

9.4.1.3 If the recovery falls outside the designated LFM recovery range (90-110%) and the laboratory performance is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be matrix related, not system related.

9.4.2 Laboratory Duplicate Sample

9.4.2.1 Duplicate samples are analyzed to demonstrate the precision of an analytical system. The duplicate analyses are performed on each batch of samples analyzed at a frequency of 10% of all samples in the batch or at least one sample if less than 10 samples are analyzed.

9.4.2.2 Relative Percent Difference (RPD): The relative percent difference is used to evaluate precision for the duplicate analyses, and RPD is calculated as follows:

$$RPD\ (\%) = \frac{|C_1 - C_2|}{C_{AVG}} \times 100 \qquad (5)$$

Where: C_1 = original sample concentration;
C_2 = duplicate sample concentration;
C_{AVG} = average of the two samples.

9.4.2.3 RPD Acceptable Limits: Acceptable limits of RPD for TKN are ≤20%. If the recovery falls outside the designated duplicate recovery range and the laboratory performance is

shown to be in control (Section 9.3), the recovery problem encountered with the duplicate analysis is judged to be matrix related, not system related.

10 CALIBRATION AND STANDARDIZATION

10.1 Prepare reagents and standards as described in Section 7.

10.2 Set up TKN manifold as is shown in Section 17.3 of the Lachat Instruments Methods Manual (reference 14.5.)

10.3 Input data system parameters as shown in Section 17 of the Lachat Instruments Methods Manual (reference 14.5.)

10.4 Pump reagent water through all reagent lines and check for leaks and smooth flow. In order to avoid precipitate forming in the manifold tubing: Add the **Buffer Line First** and allow to pump through manifold for at least 5 minutes. Then add reagent lines one by one, ending with the salicylate nitroprusside added last. For removal after analysis, reverse this order with the salicylate nitroprusside line disconnected first, and the buffer line last. When finished, place all respective reagent lines into water and allow to pump through manifold for ten minutes.

10.5 Place standards in the sampler and sequence the required information in the data system.

10.6 Calibrate the instrument by injecting the standards. The system will then associate the concentrations with the peak area for each standard to determine the calibration curve.

10.7 The initial calibration is deemed acceptable if the following criteria are met:

10.7.1 $R \geq 0.995$

10.7.2 Quality Control Sample (QCS) standard is run right after the initial calibration. The concentration of the QCS is 10 mg/L. This standard (Ammonium chloride) is ordered from Fisher Scientific, Acros Organics, ACS reagent grade. The procedure for making the QCS is the same as the one for Level 5 of the calibration standards described in (Section 7.9.1, 7.9.2, and 7.9.3). The QCS concentration must fall within ± 10% of the stated value.

10.7.3 Instrument Performance Check (IPC) refer to (Section 9.3.4).

10.8 **Figure 1.** Lists a set of initial calibration peaks and a linear calibration curve

11 PROCEDURE

11.1 All samples, any quality control samples and the initial calibration standards are digested using the following procedures. At a minimum, two blanks and one standard (LFB) should be prepared in reagent water and carried through the digestion procedure.

11.2 DIGESTION PROCEDURE

11.2.1 To a **25.0 mL** sample add **10 mL digestion solution** (Reagent 1) and mix.

11.2.2 Add 2 to 4 BD Kjeldahl Digestion Granules to each tube.

11.2.3 Place tubes in the preheated block digester for one hour at 200°C. Water from the sample must be boiled off before increasing the temperature.

11.2.4 Place the cold finger, teardrop stopper on the top of the sample tube.

11.2.5 Continue to digest for 2 hours at 380°C. This includes the ramp time (approximately 30 minutes) for the block temperature to come up to 380°C.

11.2.6 Remove the sample tubes from the block and allow about 3 minutes to cool.

11.2.7 Dilute to **25.0 mL** with **reagent water (add 23.5 mL)** to each tube and vortex to mix.

11.2.8 If the samples are not run immediately they should be covered tightly and refrigerated at 4°C.

11.3 SYSTEM START-UP PROCEDURE

11.3.1 Prepare reagent and standards as described in section 7.

11.3.2 Set up manifold as shown in Section 17.3 of the Lachat Instruments Methods Manual.

11.3.3 Input peak timing and integration window parameters as specified in section 17.2 of the Lachat Instruments Methods Manual.

11.3.4 Pump reagent water through all the reagent lines and check for leaks and smooth flow. Switch to reagent lines - add buffer first and pump through the system for 5 minutes, followed by the other reagents, adding salicylate nitroprusside last – and allow the system to equilibrate until a stable baseline is achieved.

11.3.5 Place the standards in the autosampler, and fill the sample tray. Input the information required by the data system, such as concentration, replicates and QC scheme.

11.3.6 Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with responses for each standard.

11.3.7 After a stable baseline has been obtained, start the sampler and perform the analysis.

11.4 TROUBLESHOOTING AND SYSTEM NOTES

11.4.1 Allow at least 15 minutes for the heating unit to warm up to 60°C.

11.4.2 If sample concentrations are greater than the high standard the digested sample should be diluted with **Reagent 6 (diluent.)** Do not dilute digested samples or standards with reagent water, as this will cause a problem with matix-matching, pH differences.

11.4.3 If the salicylate reagent is merged with a sample containing sulfuric acid in the absence of the buffer solution, the salicylate reagent will precipitate. If this occurs NaOH can be run through the system to attempt to clear clogs, flush system with NaOH for 20 minutes. If clogged tubing cannot be cleared, the tubing should be replaced. To prevent this, prime the system by first placing the buffer transmission line in the buffer solution.

11.4.4 In normal operation nitroprusside gives a yellow background color which combines with the blue indosalicylate to give an emerald green color. This is the normal color of the solution in the waste container.

11.4.5 If the block digestor tubes are not completely dry and have water droplets on them, there exists the possibility of ammonia contamination in the water droplets.

11.4.6 If phosphorus is also determined with the Lachat System, a second helium degassing tube should be used and segregated for the individual chemistries.

11.4.7 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

11.4.7.1 Place transmission lines in water and pump to clear reagents first.

11.4.7.2 Place reagent lines in 1M HCl and pump for several minutes

11.4.7.3 Place all lines back into water and pump out HCl.

11.4.8 If digested samples contain turbidity allow to settle prior to analysis, decant sample slowly into test tube.

11.4.9 Alternatively, if turbid conditions persist, filter the digested sample with 0.45uM filter.

12 POLLUTION PREVENTION

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in the laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

12.2 Quantity of the chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

13 WASTE MANAGEMENT

13.1 The laboratory waste management practices are conducted consistent with all applicable rules and regulations as stated in the laboratory's Sample and Waste Disposal (Revision 001) on February 25, 2004. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner in this SOP.

14 REFERENCES

14.1 U.S. Environmental Protection Agency, **Methods for Chemical Analysis of Water and Wastes**, EPA-600/4-79-020, Revised March 1993, Method 351.2

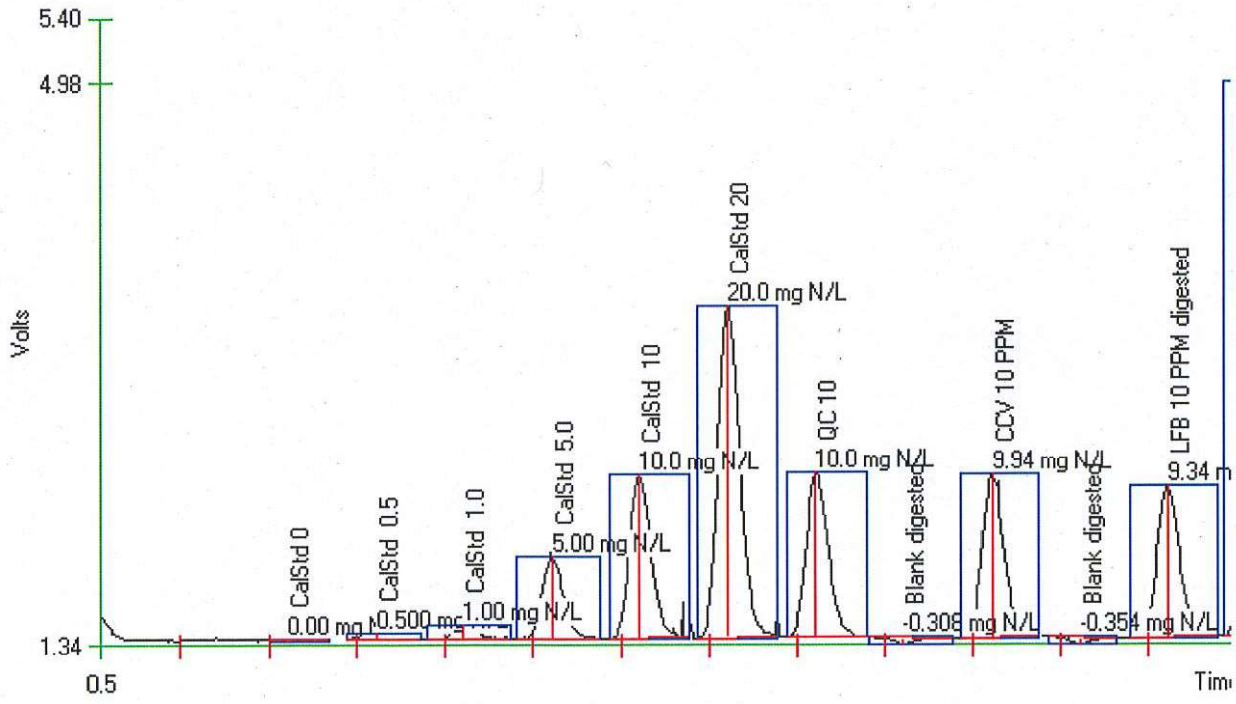
14.2 ASTM, Water(I), Volume 11.01, Method D3590-89, Test Methods for Kjeldahl Nitrogen in Water, p. 447

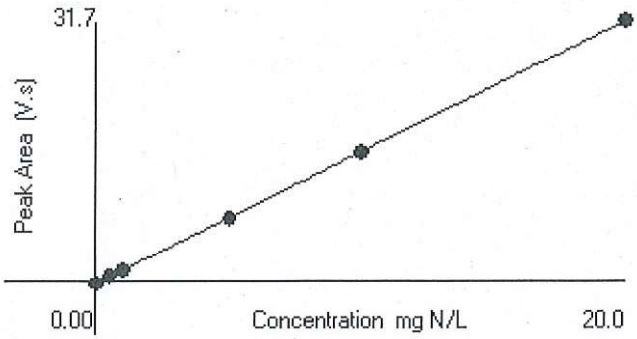
14.3 Code of Federal Regulation 40, Chapter 1, Part 136, Appendix B

14.4 Guidelines and Format for EMSL-Cincinnati Methods. EPA-600/8-83-020, August 1983.

14.5 Lachat Instruments Methods Manual, QuikChem Method 10-107-06-2-L, Revision Date, 14 May 2008

Figure 1.





Conc = 0.631 * Area + 0.0687

Area = 1.59 * Conc - 0.109

Correlation Coefficient (r) = 0.99998

Weighting : None

STANDARD OPERATING PROCEDURE FOR

Phosphorus, All Forms

EPA 365.1

Revision Author: Nicole Paradise

This SOP is effective upon signed approval by the following:

Nicole Paradise
Quality Assurance Officer

Ronald Warila
General Manager / Technical Director

Reference

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 365.1, Revision 2.0, 1993

Standard Methods for the Examination of Water and Wastewater, 2011, Method 4500P-F

Technical Report EPA/ CE-81-1, Procedures for Handling and Chemical Analysis of Sediment and Water Samples, 1995

QuikChem® Method 10-115-01-1-A, Lachat Instruments, Milwaukee, WI, 2000.

SmartChem 170/200 Method PHO-001-A

TNI Standard, Volume 1: Management and Technical Requirements for Laboratories Performing Environmental Analysis, EL-V1-2016-Rev. 2.1

I. Applicability

1.1 Analyte: Total Phosphorus, Orthophosphate

1.2 Matrix: Drinking water, Aqueous, Solid

1.3 Regulation: NPDES, CWA, SDWA

1.4 Reporting Limit:

1.4.1 Drinking Water & Aqueous: 0.01mg/L

1.4.2 Solid: 0.02mg/L

II. Summary

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

2.2 Only orthophosphate forms a blue color in this test. Phosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by manual sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by manual persulfate digestion. The developed color is measured automatically.

III. Definitions

3.1 **Calibration Blank (CB) –** A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

3.2 **Calibration Standard (CAL) –** A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 **Instrument Performance Check Solution (IPC) –** A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 **Blank Spike (BS) –** An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 **Matrix Spike (MS) –** An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

3.6 **Laboratory Reagent Blank (BLK) –** An aliquot of reagent water or other blank matrices that is digested exactly as a sample in including exposure to all glassware, equipment, and reagents that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.

3.7 **Linear Calibration Range (LCR) –** The concentration range over which the instrument response is linear.

3.8 **Method Detection Limit (MDL) –** The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.9 **Quality Control Sample (ICV) –** A solution of method analytes of known concentrations that is used to spike an aliquot of BLK or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.10 **Stock Standard Solution (SSS) –** A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

3.11 For additional definitions, see the Quality Glossary and Acronyms document, SOP Q-024.

IV. Interferences

4.1 Concentrations of ferric iron (Fe^{3}-) greater than 50 mg/L will cause a negative error due to precipitation of, and subsequent loss, of orthophosphate. Samples high in iron can be pretreated with sodium bisulfite to eliminate this interference. Treatment with bisulfite will also remove the interference due to arsenates.

4.2 Sample turbidity must be removed by filtration prior to analysis for orthophosphate. Samples for total or total hydrolyzable phosphorus should be filtered only after digestion. Sample color that absorbs in the photometric range used for analysis will also interfere.

4.3 Glassware contamination is a problem in low level phosphorus determinations. Glassware should be washed with 1:1 HC1 and rinsed with deionized water. Commercial detergents should rarely be needed but, if they are used, use special phosphate-free preparations for lab glassware.

V. Sample Collection, Preservation and Handling

5.1 Samples should be collected in plastic or glass containers. Volume collected should be sufficient to ensure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

5.2 Samples must be preserved with H_2SO_4 to a pH <2 and cooled to >0 - ≤6°C at the time of collection.

5.2.1 If samples are received at the laboratory non-preserved, they must be preserved within 48 hours of collection.

5.3 Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at >0 - ≤6°C and may be held for up to 28 days.

5.4 Orthophosphate is **never preserved** and may be held for up to 48 hours.

5.4.1 Samples for dissolved orthophosphate must be filtered within 15 minutes of collection.

VI. Procedure

6.1 Orthophosphate

6.1.1 Analyze unfiltered, with no digestion or hydrolysis.

6.1.2 Samples for dissolved ortho-phosphorus must be filtered through a 0.45µm filter within 15 minutes of collection.

6.1.3 Batch QC Preparation

6.1.3.1 Prepare the following QC samples for orthophosphate batches.

6.1.3.2 Blank (BLK)

6.1.3.2.1 The BLK is reagent water.

6.1.3.3 Blank Spike (BS)

6.1.3.3.1 An orthophosphate BS is prepared by adding 5mL of the 50mg/L PO_4 stock solution to a 250mL volumetric flask. Dilute to volume and mix. The orthophosphate BS is not digested.

6.1.3.4 Duplicate (DUP)

6.1.3.4.1 The DUP is a separate aliquot of one of the samples in the batch.

6.1.3.5 Matrix Spike (MS)

6.1.3.5.1 One MS must be prepared for every 10 samples in the batch. Therefore, if a full batch of 20 samples is being analyzed, 2 MS samples must be prepared with it.

6.1.3.5.2 An orthophosphate MS is prepared by adding 0.1 mL of 250mg/L stock to 25 mL of sample. This will result in a 1.0mg/L spike. The orthophosphate MS is not digested.

6.2 Total Phosphorus – Autoclave Digestion Procedure (SmartChem170)

6.2.1 Samples are digested in an autoclave for 30 minutes at 121ºC and 15-20 psi with ammonium persulfate and sulfuric acid to convert all phosphorus to orthophosphate.

6.2.2 Load metal rack with 16 x 100 mm glass test tubes. Add 8 mL of sample or standard to each tube.

6.2.3 Batch QC Preparation

6.2.3.1 Prepare the following batch QC samples and carry each through the entire digestion procedure.

6.2.3.2 Blank (BLK)

6.2.3.2.1 The BLK is 8mL of reagent water.

6.2.3.3 Blank Spike (BS)

6.2.3.3.1 The total phosphate BS is prepared by adding 200mL of the 5mg/L intermediate stock solution to 1L to create a 1.0mg/L BS. Digest 8mL of the BS with the sample batch for total phosphate.

6.2.3.4 Duplicate (DUP)

6.2.3.4.1 The DUP is a separate aliquot of one of the samples in the batch.

6.2.3.5 Matrix Spike (MS)

6.2.3.5.1 One MS must be prepared for every 10 samples in the batch. Therefore, if a full batch of 20 samples is being digested, 2 MS samples must be prepared with it.

6.2.3.5.2 Each total phosphate MS is prepared by adding 0.04 mL of 250mg/L stock to 8 mL of sample. This will result in a 1.25mg/L spike.

6.2.4 Add 0.5 mL of working acid/persulfate digestion solution to each tube. Vortex and cap loosely.

6.2.5 Fill autoclave with water to fill line as needed. Place rack on tray and place inside autoclave. Close and securely latch door. Be careful of thermometer gauge when closing the door.

6.2.6 Autoclave the digestion tubes for 30 minutes at 121 °C and 15-20 psi.

6.2.7 When finished, the Temp and Time indicator lights will turn off. Allow to cool to less than 90ºC before opening.

6.3 Total Phosphorus – Hot Plate Digestion of Aqueous Samples (Lachat Backup Procedure)

6.3.1 Add 1 mL of sulfuric acid solution to a 50 mL sample, blank, and/or standard in a 125 mL Erlenmeyer flask.

6.3.2 Add 0.4g mL of the ammonium persulfate.

6.3.3 Boil gently on a pre-heated hot plate for approximately 30-40 minutes or until a final volume of about 10 mL is reached. Do not allow sample to go to dryness. Alternately, heat for 30 minutes in an autoclave at 121°C (15-20 psi). See Section 6.2 for Total Phosphorus procedure using autoclave digestion for details.

6.3.4 Cool and dilute the sample to 50mL. If sample is not clear at this point, filter.

6.4 Persulfate Digestion for Sediment Samples

6.4.1 Weigh 0.5-1.0g dry weight equivalent of the sample and transfer to a 150-mL beaker. Add approximately 5 boiling stones.

6.4.2 Batch QC Preparation

6.4.2.1 Prepare the following batch QC samples and carry each through the entire digestion procedure.

6.4.2.2 Blank (BLK)

6.4.2.2.1 The BLK is 100mL of reagent water.

6.4.2.3 Blank Spike (BS)

6.4.2.3.1 The total phosphate BS is prepared by adding 200mL of the intermediate stock solution to 1L to create a 1.0mg/L BS. Digest 100mL of the BS with the sample batch.

6.4.2.4 Duplicate (DUP)

6.4.2.4.1 The DUP is a separate aliquot of one of the samples in the batch.

6.4.2.5 Matrix Spike (MS)

6.4.2.5.1 One MS must be prepared for every 10 samples in the batch. Therefore, if a full batch of 20 samples is being digested, 2 MS samples must be prepared with it.

6.4.2.5.2 Each total phosphate MS is prepared by adding 1mL of 250mg/L stock to the sample. This will result in a 2.5mg/L spike.

6.4.3 Add 10 mL of 11N H_2SO_4 and 2 g potassium persulfate to each beaker.

6.4.4 Mix the suspension and boil gently on a hot plate for 1 hr.

6.4.5 Let cool and add 2ml of 10N NaOH. Dilute to volume.

6.4.6 Filter with a pre-rinsed paper filter (Advantec 5B or equivalent) into a l00-mL volumetric flask.

6.5 Hydrolyzable Phosphorus

6.5.1 Add 1 mL of sulfuric acid solution to a 50 mL sample, blank, and/or standard in a 125 mL Erlenmeyer flask.

6.5.2 Boil gently on a pre-heated hot plate for approximately 30-40 minutes or until a final volume of about 10 mL is reached. Do not allow sample to go to dryness. Alternately, heat for 30 minutes in an autoclave at 121°C (15-20 psi). See Section 6.2 for Total Phosphorus procedure using autoclave digestion for details.

6.5.3 Cool and dilute the sample to 50mL. If sample is not clear at this point, filter.

VII. Analysis

7.1 Instrument Quality Control Preparation

7.1.1 Prepare the following quality control samples and carry each through the procedure detailed in Section 7.2.

7.1.1.1 The Low Calibration Verification (LCV) is prepared by adding 2.5mL of the 50mg/L stock solution to a 250mL volumetric flask and diluting to volume with reagent water. This will create a 0.5 mg/L standard.

7.1.1.2 The High Calibration Verification (HCV) is prepared by adding 7.5mL of the 50mg/L stock solution to a 250mL volumetric flask and diluting to volume with reagent water. This will create a 1.5 mg/L standard.

7.1.1.3 The Minimum Reporting Level (CRL) is prepared by adding 1.25mL of the 2mg/L PO_4 stock solution to a 250mL volumetric flask and diluting to volume with reagent water. This will create a 0.01mg/L CRL standard.

7.1.1.4 The ICV is prepared by spiking with Simple Nutrients standard to a 200mL to achieve ~1.25mg/L. Dilute to volume with DI and invert to mix.

7.2 SmartChem 170 Analysis

7.2.1 The calibration standards (0.01 mg/L, 0.02 mg/L, 0.04 mg/L, 0.125 mg/L, 0.5 mg/L, 1.0 mg/L, 2.0mg/L) are prepared automatically by dilution of the 2.0 mg/L standard (Section 10.25).

7.2.1.1 For more details see the SmartChem Operation Manual Chapter 3 for instructions how to configure the Methods Parameter File.

7.2.1.2 The correlation coefficient must be greater than 0.995.

7.2.1.3 Back calculate the concentration of at least the low and mid-level calibration points. The back-calculated and true concentrations should agree within ±10% of expected value for the mid-level calibration point, and within ±50% for the low-level.

7.2.1.4 If the correlation coefficient is less than 0.995 or the back calculated results are not within the established acceptance limits, the calibration must be reanalyzed.

7.2.2 The instrument has a system wash feature that will automatically clean each cuvette.

7.2.3 Fill clean reagent bottles with the appropriate reagents for this method. When filling the bottles pour the solutions slowly to minimize foaming.

7.2.4 Select the existing method or create a new method in the software with the following parameters.

7.2.5 Analyze the samples and QC in the established sequence.

7.2.6 Sample results that exceed the calibration range are automatically diluted.

7.2.7 SmartChem Start-Up Routine

7.2.7.1 Go to Sample Entry.

7.2.7.2 Double click on the desired Method to be run.

7.2.7.3 Enter # of samples.

7.2.7.4 Click on the check mark "√".

7.2.7.5 Enter Sample ID's.

7.2.7.6 Review the Run Plan Sample ID's and click Save.

7.2.7.7 Enter the Run Plan description and click Save.

7.2.7.8 Go to System Monitor.

7.2.7.9 Select and Click on the Run Plan that was created.

7.2.7.10 Verify that the selected Run Plan is correct.

7.2.7.11 Load Samples, Standards, Controls, Diluent and Empty Cups as displayed in the System Monitor.

7.2.7.12 Check the Probe Rinse, DI Water and Cleaning Solution bottles.

Note: When filling the Probe Rinse and Cuvette Wash Reservoirs, pour the solutions slowly to minimize foaming. If the probe, DI Water, and/or Cuvette Wash reservoirs require filling while a run is in progress, do not remove the cap and lift the siphon tube above the liquid surface. Insert a long stem funnel through the open port in the reservoir cap and fill the container slowly with the respective solution to minimize foaming.

7.2.7.13 Click Start in System Monitor to display user selectable options before beginning the analysis.

7.2.7.14 If a Wash Cuvettes and/or WBL is required before the start of the analysis, then click on the appropriate box. It is recommended that a Wash Cuvette and WBL be run at the start of each day.

Note: It is recommended that a new WBL be run after a Wash Cuvette operation is performed and/or after the removal and/or replacement of the same or a new cuvette into the reaction tray.

7.2.7.15 Click the Start Button to begin the analysis.

7.3 Lachat Analysis (Backup Analysis)

7.3.1 pH Adjustment of Samples

7.3.1.1 Test the pH of all samples submitted for orthophosphate analysis using the pH test strip method.

7.3.1.2 If samples have a pH >8, add 1 drop of phenolphthalein indicator to a 50 mL aliquot of sample. If a red color develops, add 11 N sulfuric acid (310 mL concentrated H_2SO_4/L) drop-wise to just discharge the color. Acidic samples (pH<4) must be neutralized with 1 N NaOH (40 g NaOH/L).

7.3.2 Prepare reagent and standards as described in Section 10.9.

7.3.3 Set up manifold as shown in Diagram 1.

7.3.4 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

7.3.5 Input the sample data into the sample tray application.

7.3.6 Calibrate using the prepared standards to create a curve with a correlation coefficient of 0.995 or better.

7.3.7 Back calculate the concentration of at least the low and mid-level calibration points. The back-calculated and true concentrations should agree within ±10% of expected value for the mid-level calibration point, and within ±50% for the low-level.

7.3.8 Analyze the samples and QC in the established sequence.

7.3.9 Dilute any sample that results exceed the calibration range.

VIII. Calculations

8.1 Report only the values that are less than 90% of the highest standard in the calibration. Dilute appropriately and re-analyze samples that do not meet these criteria.

8.2 Aqueous Samples

8.2.1 Direct reading in mg/L. Report results as either Phosphate-Total as PO_4 or Otho-phosphate as PO_4.

8.3 Solid Samples

8.3.1 Calculate the phosphate concentration on a dry weight basis as follows:

$$\textbf{Total phosphate mg/kg (dry weight)} = \frac{(x)(y)(1000)}{(g)(\%S)}$$

where: x = phosphate concentration in sediment digest, **mg/L**
y = final volume of sediment digest, **L**
g = wet weight of sample digest, **g**
%S = percent of solids in sediment sample, **as a decimal fraction**

IX. Quality Control

9.1 All quality control data should be maintained and available for easy reference or inspection.

9.2 Method Detection Limit (MDL)

9.2.1 Method detection limits must be determined before analysis for any method can begin. Method detection limits are performed as required by a method, when a

new analysis method is performed or when the laboratory management determines that a change in the instrument hardware, operating conditions or analytical procedure would cause changes in the analytical performance.

9.2.2 Refer to Corporate Document Q-029 for requirements and guidelines.

9.3 Limit of Quantitation (LOQ) Verification

9.3.1 All sample-processing and analysis steps of the analytical method shall be included in the determination of the LOQ.

9.3.2 The validity of the LOQ shall be verified through analysis of initial verification samples. An initial verification sample consists of a spiked matrix blank at or below the selected LOQ.

9.3.2.1 **This requirement is satisfied if the MDL study was spiked at or below the LOQ.**

9.3.3 The LOQ is verified if the following criteria are met:

9.3.3.1 All results are quantitative (above zero and meet the qualitative identification criteria of the method; e.g., recognizable spectra, signal to noise requirements, and presence of qualifier ions). If a result from an LOQ verification sample is not above zero and/or does not meet the qualitative identification criteria in the method, the problem shall be corrected and the verification repeated, or the LOQ verification shall be repeated at a higher concentration.

9.3.3.2 Recovery of each analyte is within 50-150% of the assigned value of the QC sample.

9.3.3.3 If the recovery for any analyte is not within 50-150%, the initial LOQ/MDL (if satisfying both requirements with one study) must be re-established.

9.3.3.4 The LOQ is greater than the established DL and at or above the spiking concentration. If the LOQ is less than or equal to the DL, the LOQ shall be raised to greater than the DL.

9.3.4 The LOQ shall be verified quarterly for each quality system matrix, technology and analyte.

9.3.4.1 **This requirement is satisfied if the MDL study was spiked at or below the LOQ, and the quarterly MDL verification samples are at the same concentration as the initial study.**

9.3.4.2 If the recovery for any analyte is not within 50-150%, the LOQ standard may be rerun only once. If the LOQ recovery is still outside of the acceptance range, the initial LOQ/MDL (if satisfying both requirements with one study) must be re-established.

9.4 Linear Calibration Range (LCR)

9.4.1 The LCR must be determined initially and verified daily.

9.4.2 The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear.

9.4.3 The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by 10%, linearity must be reestablished. If any of the standards do not recover within 10%, the standard may be re-prepared and reanalyzed only once. If the recovery is now within 10%, the linear range has been verified. If the recovery is still not within 10%, the linear range must be re-established at lower concentrations until the recovery is within 10%. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.4.3.1 Daily verification accomplished by analyzing the LCV, HCV, and IPC daily before sample analysis begins. The recovery limits are 90-110%.

9.4.3.2 If the recovery is not within 10%, the standard may be rerun only once. If the recovery is still out of range, the instrument must be recalibrated.

9.5 Initial Calibration Verification (ICV)

9.5.1 Immediately following the calibration, verify the calibration standards and acceptable instrument performance with the preparation and analyses of an ICV.

9.5.2 The ICV recovery must be within ± 10% of the true value The ICV may be run one additional time if the specified recoveries are not met, however if the second analysis fails, corrective action must be taken and the instrument recalibrated.

9.6 Minimum Reporting Limit (MRL) Check (CRL)

9.6.1 Verify quantitation at the MRL daily before sample analysis begins.

9.6.2 The validity of the MRL shall be verified by successful analysis of a 0.01mg/L CRL standard. A passing analysis is one where the recovery is within 50-150% of the assigned value of the QC sample.

9.6.3 If the calibration cannot be verified within the specified limits, reanalyze the CRL solution. If the second analysis of the CRL solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated.

9.7 Instrument Performance Check Solution (CCV and CCB)

9.7.1 For all determinations the CCV (a mid-range check standard) and a calibration blank (CCB) must be analyzed immediately following daily calibration, and after every tenth sample (or more frequently, if required) and at the end of the sample run.

9.7.2 Analysis of the CCV immediately following calibration must verify that the instrument is within 10% of calibration. Subsequent analyses of the CCV must verify the calibration is still within 10%.

9.7.2.1 If the calibration cannot be verified within the specified limits, reanalyze the CCV.

9.7.2.2 If the second analysis of the CCV confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated.

9.7.2.3 All samples following the last acceptable CCV must be reanalyzed.

9.7.3 The results of the calibration blank are to be less than the MDL. If not, rerun only once. If recoveries are greater than the MDL, and sample concentrations are either less than the reporting limit or greater than ten times the contamination level in the CCB, the sample results may be accepted. Otherwise, all contaminated samples must be reanalyzed.

9.8 Laboratory Reagent Blank (BLK)

9.8.1 The laboratory must prepare and analyze at least one BLK with each batch of 20 samples or less.

9.8.2 Data produced are used to assess contamination from the laboratory environment.

9.8.3 The results of the BLK are to be less than the MDL. If not, rerun only once. If recoveries are greater than the MDL, and sample concentrations are either less than the reporting limit or greater than ten times the contamination level in the BLK, the sample results may be accepted. Otherwise, all contaminated samples must be reprepared and reanalyzed.

9.9 Blank Spike (BS)

9.9.1 At least one BS must be prepared and analyzed with each batch of 20 samples or less.

9.9.2 Calculate accuracy as percent recovery using the following equation:

$$R = \frac{BS}{S} * 100$$

where: R = percent recovery
BS = laboratory fortified blank
S = concentration of the analyte added to fortify the BS solution

9.9.3 If the recovery of any analyte falls outside the required control limits of 90-110%, BS may be rerun only once. If recoveries are still outside of the acceptance range, all associated samples must be reprepared and reanalyzed.

9.10 Matrix Spike (MS)

9.10.1 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples.

9.10.2 In each case the MS aliquot must be a duplicate of the aliquot used for sample analysis.

9.10.3 For total phosphate, the MS undergoes the digestion process.

9.10.4 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated MS recovery range 90-110%. Percent recovery may be calculated using the following equation:

$$\mathbf{R = \frac{C_s - C}{s} \times 100}$$

where: R = percent recovery
C_s = fortified sample concentration
C = sample background concentration
s = concentration equivalent of analyte added to sample

9.10.5 If the recovery of any analyte falls outside the designated MS recovery range and the BS performance for that analyte is shown to be in control, the recovery problem encountered with the MS is judged to be either matrix or solution related, not system related. The result in the unfortified sample must be qualified to inform the data user that the results are suspect due to matrix effects.

9.11 Sample Duplicate (DUP)

9.11.1 Analyze one duplicate sample for every 20 samples or less.

9.11.2 A duplicate sample is a sample brought through the entire sample preparation and analytical process.

9.11.3 Calculate the RPD using the following equation:

$$\mathbf{RPD = \frac{|Sample\ Result - Duplicate\ Result|}{\left(\frac{Sample\ Result + Duplicate\ Result}{2}\right)} \times 100}$$

9.11.4 A control limit of ± 20% for RPD shall be used for sample values greater than 5 times the reporting limit.

9.11.5 A difference of ±2 times the reporting limit is to be used to evaluate samples below 5 times the reporting.

9.11.6 If the duplicate RPD is outside of these ranges the sample may be qualified and reported to the data user as such

X. Equipment, Reagents and Standards

10.1 SmartChem170 Discrete Analyzer

10.2 Glass sample vials used in autoclave – C&G Scientific part # LT23-A020-A01B-ZS1 or equivalent

10.3 Autoclave

10.4 Sample Cups for Discrete Analyzer – 4mL

10.5 Various Class A Volumetric Flasks – ranging in volumes

10.6 Various Mechanical Pipettes – ranging in volumes

10.7 Various Glass Pipettes – ranging in volumes

10.8 Porous Boiling Chips – VWR Scientific part # 26397-409 or equivalent

10.9 Potassium Persulfate – Alfa Aesar part # 13145 or equivalent

10.10 Ammonium Persulfate – Fisher part # A682-500 or equivalent

10.11 10N Sodium Hydroxide (NaOH) – LabChem part # LC145004 or equivalent

10.12 Anhydrous Potassium Phosphate Monobasic (KH_2PO_4) – Fisher part # P285-500 or equivalent

10.13 Sulfuric Acid – Fisher part # A30051-212 or equivalent

10.14 Potassium Antimonyl Tartrate, Trihydrate ($K(SbO)C_4H_4O_6 \cdot ½H_2O$) – Fisher part # A867-500 or equivalent

10.15 Antimony Molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) – Acros Organics part # 42331-5000 or equivalent

10.16 Sodium Dodecyl Sulfate ($CH_3(CH_2)_{11}OSO_3Na$) – Fisher part # BP166-100 or equivalent

10.17 Ascorbic Acid ($C_6H_8O_6$) – Fisher part # A62-500 or equivalent

10.18 Probe Rinse – Unity Scientific part # 3AS-RN00-21 or equivalent

10.19 Cuvette Cleaning Solution – Unity Scientific part # 365-0366-900 or equivalent

10.20 EDTA (disodium) – Fisher part # S311-500 or equivalent

10.21 1000mg/L Organic Nutrients (TKN Total Phosphate Solution) - Absolute Standards part # 54152 or equivalent

10.22 Phenolphthalein Indicator Solution – J.T. Baker part # 287.04 or equivalent

10.23 250.0 mg/L of Phosphate as P

10.23.1 In a 1000 mL volumetric flask dissolve 1.099 g primary standard grade anhydrous potassium phosphate monobasic (KH_2PO_4) that has been dried for one hour at 105°C in approximately 400 mL water.

10.23.2 Dilute to the mark with DI water and invert to mix.

10.24 50.0 mg/L of Phosphate as P

10.24.1 In a 200 mL volumetric flask, dilute 40.0 mL Stock Standard Solution #1 to the mark with DI water.

10.24.2 Invert to mix.

10.25 2.0mg/L Working Standard Solution

10.25.1 Prepare a 2mg/L intermediate stock solution by adding 10mL of 50.0mg/L stock standard to a 250mL volumetric flask. Dilute to volume and invert to mix.

10.25.2 For the Total Phosphate analysis, add 200ul of sulfuric acid into 100ml of 2 mg/L standard.

10.26 5mg/L Intermediate Solution

10.26.1 Prepare an 5mg/L intermediate solution by adding 5mL of 1000mg/L Organic Nutrients and 2mL sulfuric acid to a 1L volumetric flask. Dilute to volume and invert to mix.

10.27 Reagent water: Distilled or de-ionized water, free of the analyte of interest, ASTM type II or equivalent.

10.28 Sulfuric acid solution, 5N

10.28.1 Slowly add 70 mL of conc. H_2SO_4 to approximately 400 mL of reagent water.

10.28.2 Cool to room temperature and dilute to 500 mL with reagent water.

10.29 Sulfuric Acid Solutions

10.29.1 11N H_2SO_4 Solution

10.29.1.1 Carefully add 310 mL concentrated H_2SO_4 to approximately 690mL of distilled water and dilute to 1L with distilled water.

10.29.2 5.6M H_2SO_4 Solution

10.29.2.1 Carefully add 314 mL concentrated H_2SO_4 to approximately 690mL of distilled water and dilute to 1L with distilled water.

10.30 Acid-Persulfate Digestion Solution

10.30.1 Dissolve 6.4g of ammonium persulfate and 16 mL of 5.6M stock sulfuric acid solution in a 50 mL volumetric flask. Dilute to the mark with distilled water and spin until dissolved.

10.30.2 Prepare fresh daily.

10.31 **Reagents and Standards – Smart Chem 170**

10.31.1 Potassium antimonyl tartrate solution (0.3%)

10.31.1.1 Weigh 0.6 g potassium antimonyl tartrate trihydrate $K(SbO)C_4H_4O_6 \cdot ½H_2O$; dissolve in 50 mL distilled water in a 100 mL volumetric flask, dilute to volume.

10.31.1.2 Store at >0 - ≤6°C in a dark, glass-stoppered bottle.

10.31.2 Ammonium molybdate solution (4 %)

10.31.2.1 Dissolve 8 g ammonium molybdate tetrahydrate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 200 mL reagent water.

10.31.2.2 Store in an acid washed plastic bottle at >0 - ≤6°C.

10.31.3 Ascorbic acid (0.1M):

10.31.3.1 Dissolve 1.8 g ascorbic acid $C_6H_8O_6$ in 100 mL reagent water.

10.31.3.2 Add 1.0 mL 15 % SDS (Section 10.31.5 and swirl gently to mix minimizing foaming.

10.31.3.3 Prepare this solution fresh daily.

10.31.3.4 ***Do not refrigerate the ascorbic acid solution containing SDS***.

10.31.4 Color Reagent

10.31.4.1 Mix the above reagents in the following proportions for 100 mL of the mixed reagent:

a. 37 mL 5N H2SO4 (Section 10.28)

b. 5 mL antimony potassium tartrate solution (Section10.31.1)

c. 15 mL ammonium molybdate solution (Section 10.31.2)

d. 5 mL 15% w/w Sodium dodecyl sulfate (Section 1031.5)

e. 38 mL reagent water

10.31.4.2 Mix after addition of each reagent.

10.31.4.3 Store in an acid washed plastic bottle at room temperature.

10.31.4.4 ***Do not refrigerate the mixed color reagent solution containing SDS***.

10.31.4.5 It is recommended that this solution be prepared fresh weekly.

10.31.5 Sodium dodecyl sulfate (SDS) 15% w/w: Use only purest grade. Phosphate concentration in the SDS reagent should be ≤0.0001%.

10.31.5.1 Dissolve 15 g SDS $CH_3(CH_2)_{11}OSO_3Na$ in 85 mL reagent water.

10.31.5.2 May require gentle stirring and heat to fully dissolve.

10.31.6 Cuvette cleaning solution

10.31.6.1 Add 30 mL of Cuvette Cleaning Solution to approximately 700 mL of reagent water and dilute to 2 L with reagent water. Invert five times to mix.

10.31.6.2 Store the solution at room temperature.

10.31.7 Probe rinse solution

10.31.7.1 Add 1.0 mL of Probe Rinse Solution 2L of reagent water. Invert five times to mix.

10.31.7.2 Store the solution at room temperature.

10.31.8 The Reagent #1 Diluent is reagent water or the appropriate sample matrix blank. To 98 mL of reagent water or the appropriate sample matrix blank in a clean plastic bottle, add 2.0 mL of 15 % w/w SDS. Cap the bottle and invert five times to mix. Pour the solution slowly when filling a clean reagent bottle to minimize foaming.

10.32 **Preparation of Lachat Reagents and Standards**

10.32.1 Use deionized water for all solutions.

10.32.2 Degassing with helium

10.32.2.1 To prevent bubble formation, degas the carrier solution with helium. Use He at 5-20 psi through a disposable narrow tip pipette. Bubble He vigorously through the solution for one minute. Dispose of the pipette after each use.

10.32.3 Stock Ammonium Molybdate Solution

10.32.3.1 In a 1 L volumetric flask dissolve 40.0 g ammonium molybdate tetrahydrate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] in approximately 800 mL DI water.

10.32.3.2 Dilute to the mark and stir for four hours.

10.32.3.3 Store in plastic and refrigerate.

10.32.3.4 May be stored up to two months when kept refrigerated.

10.32.4 Stock Antimony Potassium Tartrate Solution

10.32.4.1 In a 1L volumetric flask, dissolve 3.22 g antimony potassium tartrate (potassium antimony tartrate trihydrate $K_2(C_4H_2O_6Sb)_2 \cdot 3H_2O$) in approximately 80 mL of DI water.

10.32.4.2 Dilute to the mark and invert three times.

10.32.4.3 Store in a dark bottle and refrigerate.

10.32.4.4 May be stored up to two months when kept refrigerated.

10.32.5 Molybdate Color Reagent

10.32.5.1 To a 1 L volumetric flask add about 500 mL DI water.

10.32.5.2 Add 35.0 mL concentrated sulfuric acid and swirl to mix.

(**CAUTION**: The reaction is exothermic; it will get warm!)

10.32.5.3 When it can be comfortably handled, add 72.0 mL Stock Antimony Potassium Tartrate Solution (Reagent 2) and 213 mL Stock Ammonium Molybdate Solution (Reagent 1).

10.32.5.4 Dilute to the mark and invert three times.

10.32.5.5 Degas with helium.

10.32.5.6 Prepare fresh weekly.

10.32.6 Ascorbic Acid Reducing Solution, 0.33 M

10.32.6.1 In a 1 L volumetric flask dissolve 60.0 g granular ascorbic acid in about 700 mL of DI water.

10.32.6.2 Dilute to the mark and invert to mix.

10.32.6.3 Degas the solution before proceeding or it will get extremely soapy.

10.32.6.4 Add 1.0 g dodecyl sulfate ($CH_3(CH_2)_{11}OSO_3Na$).

10.32.6.5 Prepare fresh weekly.

10.32.6.6 Discard if the solution becomes yellow.

10.32.7 Sodium Hydroxide - EDTA Rinse

10.32.7.1 Dissolve 65 g sodium hydroxide (NaOH) and 6 g tetrasodium ethylenediamine tetraacetic acid (Na_4EDTA) in 1.0 L DI water.

10.32.8 **Lachat Calibration**

10.32.8.1 Prepare fresh daily using deionized H_2O as shown below:

Standard	A	B	C	D	E	F	G	Blank
Conc., mg/L	2	1	0.5	0.2	0.05	0.02	0.01	--
Volume 50mg/L Phosphate Standard, mL	10	5	2.5	1.0	0.25	0.1	--	--
Volume 2mg/L Phosphate Standard, mL	--	--	--	--	--	--	1.25	--
Final Vol., mL	250							

XI. Safety

11.1 Every sample should be considered hazardous when performing the analysis. Standard laboratory safety guidelines must be adhered to. Gloves, eye protection, and lab coats must be worn during sample retrieval, analysis and disposal.

11.2 Refer to SDS on file at http://microbac.online-msds.com/index.php .

XII. Pollution Prevention

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in the laboratory. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

12.2 Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation by the following means:

12.2.1 Ensure that the quantity of the chemicals purchased is based on expected usage during its shelf life and the disposal cost of unused material.

12.2.2 Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

12.2.3 Control the usage by closely monitoring the instrument operation to avoid pumping reagents through after sample run has completed.

XIII. Waste Management

13.1 All waste is handled in accordance with Microbac's Chemical Hygiene Plan, which is mandatory reading for all employees and is readily available for any interested parties.

XIV. Method Performance

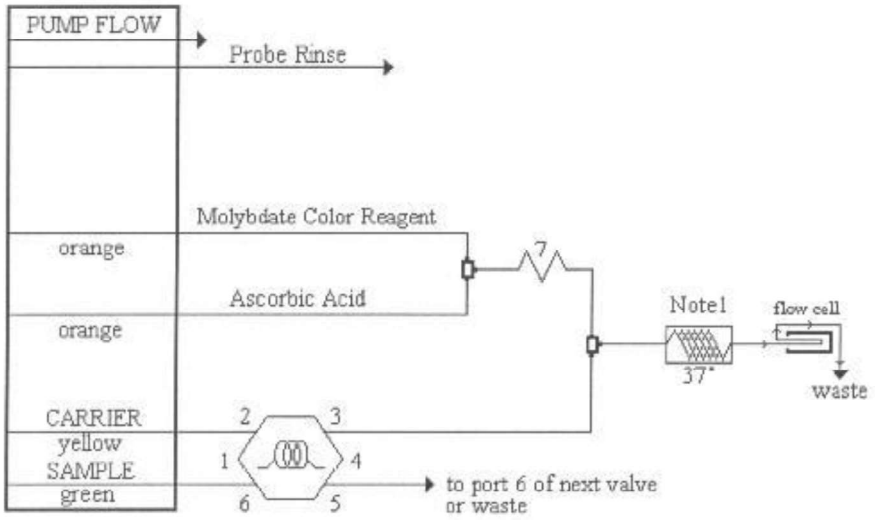
14.1 Initial Demonstration of Capability

14.1.1 Before new analysts run any samples, verify their capability with the method. Run a laboratory fortified blank four times and compare to the limits listed in Section 9.9.3.

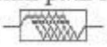
14.2 Continuing Demonstration of Capability

14.2.1 On an annual basis, each analyst must verify their continuing capability. This may be done by passing a proficiency test or by running a laboratory fortified blank four times and comparing to the limits listed in Section 9.9.3.

Diagram 1: Phosphate Manifold Setup



Carrier: DI water
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
AE Sample Loop: 70 cm x 0.8 mm i.d.
QC8000 Sample Loop: 75.5 cm x 0.8 mm i.d.
Interference Filter: 880 nm

Apparatus: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required. The  shows 175 cm of tubing wrapped around the heater block at 37°C.

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

Note 1: 175 cm of tubing on the heater.

Appendix I

Smart Chem method setup.

Note: The method as described here is equivalent to the EPA protocol 365.1. Modifications to this test method may void equivalency. If modifications are made, then the user must perform the required testing to document the method performance in accordance with EPA/NELAC requirements.

Method Code: WP1W **Range: 0.01 – 1.00 mg P/L**	**Vol., µL**	**Delay Time, sec.**	**Read Time, sec.**	**Rinse, µL**	**Code**
Sample Volume	290				
Reagent 1: Reagent Dilution (See Section XX Note 1)	9	36	0	0	DIL1
Reagent 2: Color Reagent (See Section XX and XX	65	0	0	0	MOL1
Reagent 3: Ascorbic Acid	28	0	342	0	ASC1

Note (1): If the method is modified, then the combined Sample + Reagent 1 + Rinse volume must be ≤ 390µL. If the method is modified, and sample blanking is used, then the combined volume of sample + Reagent 1 volume must be ≥ 290 µL and ≤ 390 µL.

Note (2): The maximum total reaction volume is 670 µL. The recommended maximum total reaction volume is 500 µL.

Note (3): The minimum sample and reagent volume is 3 µL. The recommended minimum sample and reagent volume is 5 µL.

Note (4): If the method is modified and the combined Sample + Reagent 1 volume is ≤ 290 µL , then the Sample Blanking option should not be used after Reagent 1. If Sample blanking is desired, create a new method file using the following parameters (Section xxx) and select the Sample Blanking Option after the addition of Reagent 1.

- Extending the Range of the Method: The range of the method can be extended by dilution using Reagent 1 as the diluent as shown below.
  - In the extended range method **the total volume of Sample plus Reagent 1 must equal 299 µL and** the diluent matrix (Reagent 1) in the extended range must match (be the same) as that of the sample matrix.
  - For example the Reagent 1 diluent, (290-X) shown in the parameter file below, for orthophosphate determinations of unpreserved samples would be reagent water, and for acid preserved samples the diluent

would be reagent water containing the same strength H_2SO_4 (2 mL conc. H_2SO_4 per liter) used to preserve the samples.

- To prepare the Reagent 1 diluent, combine 9 mL of Reagent1 with (290 - X) mL of the appropriate diluent as stated above.
- The dilution equation $C_1V_1 = C_2V_2$ is used to calculate the new sample and Reagent 1 (diluent) volumes.
- The value of C_1 is 1.00 mg/L P and the V_1 volume is 299 µL; C_2 is the target high calibrant standard for the extended range method; and V_2 is the new sample volume "**X**" shown below in the "Extended Range Parameter File With or Without Sample Blanking" after the addition of Reagent 1.

Extended Range with or without Sample Blanking after Reagent 1					
Method Code: User Assigned **Range: 0.01 – X.XX mg P/L**	**Vol., µL**	**Delay Time, sec.**	**Read Time, sec.**	**Rinse, µL**	**Code**
Sample Volume	X				
Reagent 1: Reagent Dilution (See Section XX Note 1)	9 + (290-X)	36	0	0	DILX
Reagent 2: Color Reagent (See Section XX and XX	65	0	0	0	MOL1
Reagent 3: Ascorbic Acid	28	0	342	0	ASC1

SOP Revision History

Revision No.	Description of Changes	Effective Date	Initiated by
2.3	Added revision history table	6/8/09	LM
2.4	Clarified addition of H2SO4 in Section 5.1.1 Clarified filtration requirement in Section 5.1.5 Added requirement to prepare LFB, LRB, and LFM/LFMD for sediments in Section 5.2.1.5 Changed standards preparation instructions for smaller amounts in Section 7.2	2/25/11	BS, LM
3	Added Section 5.3 for autoclave digestion procedure Added Section 7.3 for SmartChem 170 analysis Added Section 8.2 for SmartChem 170 analysis	1/14/14	GP LM
4	Created cover page Updated entire document to reflect Element nomenclature Updated references Reformatted Section I Added Section II, 3.11, 4.2, 5.2.1, 5.4.1, 6.1.2, 6.4.5, 7.2.6, 7.2.7, 7.3.7, 7.3.9, 10.1-10.22, 10.31.8, 10.32.6.3, XI, XIV Updated Sections 5.1, 6.2.6, 6.2.7, 6.4.3, 9.1, 9.9.3.9, 10.5, 10.32.4.1 Added Batch QC Preparation – Sections 6.1.3, 6.2.3, 6.4.2 Combined previous Sections 5.1 and 8.2.4 into current Section 6.3 Added instrument QC preparation – Section 7.1 Added calibration preparation instructions – Section 7.2.1 Added calibration acceptance criteria – Section 7.2.1.2 Added requirement to back calculate calibration points – Section 7.2.1.3 Revised MDL Section 9.2	12/21/2020	NJP/KL

Revision No.	Description of Changes	Effective Date	Initiated by
	Added LOQ requirement – Section 9.3 Changed LCR frequency to daily in Section 9.4.1 and added acceptance criteria in Section 9.4.3.1 Added Section 9.6 – MRL Check Added Section 9.11 – Duplicate Added all purchasing information to Section X Updated the 250mg/L standard preparation in Section 10.23.1 Added Section 10.25 – 2mg/L Standard Preparation Added Section 10.26 – 5mg/L Standard Preparation Added requirement to spin acid-persulfate digestion solution until dissolved to Section 10.30.1 Updated standard storage temperature in Sections 10.31.1.2 and 10.31.2.2 Updated color reagent preparation in Section 10.31.4 Updated cuvette cleaning solution preparation in Section 10.31.6 Updated probe rinse solution preparation in Section 10.31.7 Revised Lachat calibration table in Section 10.32.8 Deleted notes from Previous Sections 8.2.9 and 7.3.6.5 Deleted previous Sections 7.3.6 – 7.3.10, 7.3.14, 8.1.5, 8.2.1 – 8.2.3, 8.2.6 – 8.2.8, 8.2.11.3, 8.2.11.5, 9.2.1, 9.4.2.1, 9.4.5, 9.6.3, 9.6.7, 9.6.9,		
5	Updated reference methods	7/13/2021	NJP
6	Added Sections 7.2.1.4, 9.3.3.3, 9.3.4.2, 9.4.3.2, 9.7.3, 9.11.3, 9.11.6, 10.29.2 Updated Sections 8.2.1, 9.3.3.2, 9.4.3, 9.4.3.1, 9.5.2, 9.8.3, 9.9.3, 9.10.5, 9.11.4, 9.11.5 Deleted Previous Section 9.3.2	8.27/2021	NJP

Barnstable County Department of Health and the Environment Laboratory

SM 2540 D

STANDARD OPERATING PROCEDURE

For

Determination of Total Suspended Solids in Aqueous Samples

(Revision 010)

September 6, 2019

		Signature	Date
Analyst:	Lacey Prior / TBD	LaceyAnnPrior	9/6/19
Laboratory Director:	Dan White		06SEP19

Barnstable County Laboratory

STANDARD OPERATING PROCEDURE (SOP)
For

Determination of Total Suspended Solids in Aqueous Samples

1. SCOPE AND APPLICATION

1.1 This method covers the determination of total suspended solids (TSS) in drinking water, surface water, domestic water, industrial wastes, and other aqueous samples.

2. SUMMARY OF METHOD

2.1 A well-mixed sample is filtered through a Preweighed glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105 °C.

3. INTERFERNCES

3.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specific because these variables have been shown to affect results.

3.2 Samples high in dissolved solids, such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken to thoroughly wash the filter to ensure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

4. SAFETY

4.1 Follow general laboratory safety guidelines, such as wearing safety glasses, gloves and a lab coat.

5. EQUIPMENT AND SUPPLIES

5.1 ProWeigh Filters, 47mm (Environmental Express, catalog # F93447MM)

5.2 Filtration apparatus with reservoir and a coarse (40-60 microns) fritted disc as a Filter support (VWR, catalog # 28143-550)

5.3 Suction flask, 5 gallons

5.4 Drying Oven (Fisher Scientific Isotemp Oven, Model # 625G, catalog # 13-247-625G) for operation a 103-105 °C.

5.5 Desiccator, provided with a desiccant containing a color indicator of moisture concentration.

5.6 Aluminum weighing dishes (VWR, Catalog # 25433-008)

5.7 Analytical balance (± 0.1mg)- Fisher Scientific (Model ACCU-124D)

5.8 Graduated cylinder, 100mL (Fisher Scientific, catalog # 08572D)

6. REAGENTS AND STANDARDS

6.1 Reagent water – Deionized water from Milli-Q Direct 8/16 System, Millipore Direct-Q 3 System.

6.2 30 mg/L TSS standard from NSI Solutions Catalog # QCI-057 for low level standard check.

7. SAMPLE COLLECTION, PRESERVATION AND STORAGE

7.1 Samples are collected in 1 liter plastic bottles. All bottles must be thoroughly cleaned and rinsed with reagent water.

7.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Samples are shipped iced or stored cold in a refrigerator at 4°C.

7.3 The holding time is 7 days and no preservation is required.

7.4 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.

8. QUALITY CONTROL

8.1 A method blank using 100 mL of deionized is processed with the same

procedures as the samples in each batch.

8.2 A duplicate sample is to be included in any set of 10 samples. The agreement between the sample and the duplicate must be within 5% of their average.

8.3 Method Detection Limit (MDL)- An MDL is established using reagent water fortified at a concentration of three to five times the estimated detection limit. seven replicate aliquots of the fortified reagent water are run and processed through the entire analytical method. Then the MDL is calculated as follows:

$$MDL = (t) \times (S_{n-1}) \qquad (1)$$

Where,
t = student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.
S_{n-1} = sample standard deviation (n-1) of the seven replicate analyses.

The laboratory conducts MDL study annually. One set of MDL study is shown in Table 1.

8.4 Accuracy and Precision- 4 replicates of a known value are prepared and analyzed. The mean measured concentration ($C_{\bar{x}}$) of the replicate values is calculated as follows:

$$C_{\bar{x}} = \frac{(C_1 + C_2 + C_3 + C_4)}{n}$$

Where,

$C_{\bar{x}}$ = Mean recovered concentration of the replicate analysis.
C_1, C_2, ...C_n= Recovered concentrations of the replicate 1,2, …n.
The value derived for $C_{\bar{x}}$ must be within ± 10% of the true value.
The percent relative standard deviation (%RSD) of the replicate analysis as stated above is calculated using the following equation.

$$\%\ RSD = \frac{(S_{n-1})x100}{(C_{\bar{x}})}$$

Where,
S_{n-1} = sample standard deviation (n-1) of the replicate analysis.
$C_{\bar{x}}$ = mean recovered concentration of the replicate analysis.

8.5 A low level standard is to be run on an ongoing basis. Use NSI Solutions Catalog Number QCI-057.

9. CALIBRATION OF BALANCE

9.1 Check the analytical balance with three Class 's' weights: 0.05(g), 10(g), and 100(g) and record them in the Analytical Balance Calibration Logbook.

10. PROCEDURE

10.1 Preparation of glass-fiber filter disk:

10.1. 1 Take a Preweighed 47mm glass fiber filter from Environmental Express and record the weight in the Initial Weight column in the Solids Runlog Book. Follow this procedure for all samples.

10.2 Sample Analysis:

10.2. 1 Assemble filtering apparatus and filter and begin suction. The filter is seated on fritted support by wetting it with deionized water. After shake thoroughly, the sample volume is measured in a graduated cylinder and slowly poured on the filter. The filter is then rinsed with three successive 10 mL volumes of deionized water, allowing complete drainage between washings and suction is continued for three minutes after filtration is complete.

10.2 2 Filters are carefully removed from filtration apparatus and transferred to an aluminum weighing dish for support. Filters placed in their aluminum weighing dish are put in the oven at 103-105 ° Celsius for at one hour. Cool in desiccator to balance temperature and weigh. Repeat the cycle of drying , cooling, desiccating and weighing until a constant weight is obtained or until weight change is less than 4% of the previous weighing or 0.5mg, whichever is less. Store in desiccator until needed.

11. DATA ANALYSIS, CALCULATIONS AND REPORTS

11.1 Calculation:

$$\text{TSS (mg/L)} = \frac{(A-B) \times 1000}{V}$$

Where:
A= weight of filter + dried residue (mg);
B= weight of filter (mg);
V= sample volume (mL).

11.2 Reports:

All data, including the date, Lab ID, Client ID, sample volume (V), weight of filter (B), weight of filter + dried residue (A), the calculation, and analyst initials are

recorded in the Inorganics Solid Runlog Book (Figure 1)

12. POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The laboratory waste management practices are conducted consistent with all Applicable rules and regulations as stated in the laboratory's Sample and Waste Disposal (Revision 003) on July 6,2006. Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner in this SOP.

13. REFERENCES

1. U.S. Environmental Protection Agency, "Method or Guidance for Analysis of Water, Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)", Method 160.2, Issued 1971.

2. American Public Health Association (APHA)
American Water Works Association (AWWA)
Water Environment Federation (WGI)
Standard Methods (SM) for the Examination of Water and Wastewater, 22nd edition SM2540D, 2012.

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: YSI ProDSS Field Meter

Effective Date: 2021-10-18	**Number:** MASSTC-SOP-016	**Revision:** 004

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site.

History	*Effective Date*
Revision 004 – Updated Added section about turbidity calibration (step 9.10) as well as occasionally references throughout the rest of the document when pertinent. Added definition of Ultrapure Water. Removed not in Scope in Application about nitrate/ammonia probes (no longer relevant). Edited listing of SDS's to one-sentence phrase about where to find all SDS's. Updated by EMO.	2021-10-18
Revision 003 – Updated Section 9 based on new reference point (MASSTC-FRM-033 – ProDSS Calibration Checklist). Updated Section 10 based on new reference point (MASSTC-FRM-034 – ProDSS End of Day Checklist). Added instruction to Section 10 concerning project following NSF protocol. Updated references to Calibration Worksheet to instead reference new form. Added instruction for performing Zero ODO calibration. Added instruction concerning pH calibration mV range (9.7.6.1). Updates done by EMO.	2021-06-09
Revision 002- Removed references to needing to write down CCV for pH and ODO on calibration worksheet. Added instruction of needing to write calibration information and change in standards on white board in lab. Added clarification about reviewing downloaded file to ensure correct site readings were taken at end of day.	2019-12-09
Revision 001- Reformatted several sections for consistency. Removed section on data logging. Made small edits due to changes that have been made by our staff including updates in cleaning procedure. Removed directives to record values in yellow field book which has been since updated to electronic records only. Removed reference to nutrient probes from title as this is stated clearly in the first section of the document.	2019-10-28
Revision 000 - Initial Release	2019-06-07

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1. Scope and Application

1.1. This Standard Operating Procedure (SOP) describes the calibration and use of the YSI ProDSS Handheld Field Meter.

1.2. This SOP DOES NOT apply to the calibration and use of the YSI 556 Handheld Field Meter.

1.3. This SOP is to be adhered to by all MASSTC staff and all others utilizing this field meter.

2. Definitions

2.1 **Meter Body** – the handheld portion of the meter.

2.2 **Sonde** – the component housing individual sensor probes.

2.3 **Sensor Guard** – the black plastic caging that attaches to the outside of the sonde to protect the sensors from being bumped or harmed.

2.4 **Sealing Ring** – the black moldable plastic ring that seals in any air and moisture in the optical dissolved oxygen calibration cup.

2.5 **Calibration cup** – the clear plastic tubing in which liquid for calibrations is placed. If a standard is not specified, this specifically refers to the YSI-manufactured clear plastic tubing with threading in which the sonde sits used for Optical Dissolved Oxygen calibrations.

2.6 **SDS (Safety Data Sheet)** – a document provided by the chemical manufacturer which details the safety precautions and hazards as well as other information on a specific chemical.

2.7 **Small Cleaning Brush** – a hooked wire with black bristles found hanging in the lab.

2.8 **Sonde Weight** – a weight attached to the bottom of the sensor guard.

2.9 **PPE (Personal Protective Equipment) –** equipment worn to minimize exposure to hazards that cause serious workplace injuries and illnesses.

2.10 **Ultrapure water (ASTM Type I Reagent Grade Water) –**water that has been purified to strict chemical and biological specifications, containing, by definition, only H_20, and H+ and OH- ions in equilibrium. Conductivity for ultrapure water is about 0.055 µS/cm at 25°C, also expressed as resistivity of 18.2 MΩ.

3. Health and Safety Warnings

3.1 **Physical Hazards** – use care and good judgement when utilizing field meters. If a sample location is in a place where it cannot be safely analyzed (e.g. confined space), notify the MASSTC director immediately and do not attempt to retrieve it. Environmental conditions (e.g. rain, snow, etc.) can lead to uneven and/or slippery surfaces so care should be taken to prevent slips and fall. **PPE Required: Closed-toe shoes/boots. Care should be taken to dress appropriately.**

3.2 **Infectious Materials** – even the cleanest wastewater can contain pathogens or toxic materials. Proper precautions should be taken to isolate yourself. **PPE Required: gloves**.

3.3 **Fire/Explosive Hazards** - Charge the battery pack in an open area away from flammable materials, liquids, and surfaces. Do not charge or handle a battery pack that is hot to the touch. Failure to follow the safety warnings and precautions can result in personal injury and/or instrument damage. Read **Rechargeable Lithium-Ion battery pack safety warnings and precautions (Section 7.1 of MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F).**

3.4 **Skin Corrosion/Serious Eye Damage** - Some of the chemicals required for these solutions could be hazardous under some conditions; therefore, the standards should only be prepared by qualified chemists in laboratories where proper safety precautions are possible. The user should obtain and read the Safety Data Sheet (SDS) for each chemical and to follow the required instructions with regard to handling and disposal of these chemicals. **PPE Required: Gloves and safety goggles**.

3.4.1 Consult Sharepoint's MASSTC-Safety Data Sheets for hazardous material information.

4. CAUTIONS

4.1 The sensor probes should never be allowed to dry out. Store the sonde in the calibration cup with a small amount of tap water, and ensure the seal is tightened. Store unused probes according to manufacturers' instructions. (Section 4 of MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F)

4.2 When transferring the meter between locations, be sure to keep the sonde and cable off the ground to reduce wear and prevent damage. Be careful not to step on the cable.

4.3 Be careful not to put undue strain on the cable.

4.4 Do not allow the meter body to become submerged in liquid. This can cause irreparable damage to the unit.

4.5 When connecting the meter to the charging cable or computer download cable, be careful not to bend or flex the connector, as this can damage the charging/download port.

5. INTERFERENCES

5.1. Change pH buffer solutions twice per week (Monday and Wednesday unless change in the weeks' work days or significant change in sample loading days) to ensure standards are accurate. When appropriate, standards can be changed more frequently if needed to ensure accuracy of projects following NSF protocol.

5.2. Change conductivity solution once per week to ensure standards are accurate.

5.3. Change turbidity standards once per week to ensure standards are accurate.

5.4. Ensure that probes are kept clean and stored properly to minimize bio-fouling interference.

5.5. Change water used for dissolved oxygen calibration daily to minimize bio-fouling interference.

5.6. Store stock pH buffer solutions in closed area, capped and away from sunlight.

5.7. Always put caps on poured buffer solutions to reduce evaporation loss.

6. Personnel Qualifications

6.1. Personnel are required to be knowledgeable of the procedures in this SOP and all referenced SOP's.

6.2. Personnel are required to receive training on the proper use of the instrument from a qualified member of staff.

6.3. Personnel performing calibrations are required to review relevant Safety Data Sheets specified in Section 3.

7. Special Apparatus and Materials

7.1. US pH buffer solutions (4.00, 7.00, and 10.00)

7.2. Conductivity calibration solution (447 µS/cm)

7.3. Turbidity standards (Ultrapure Water (0 FNU), 124 FNU standard, and 1010 FNU standard)

7.4. YSI ProDSS meter and accompanying probes, cables, and equipment.

8. Sample Handling and Storage

8.1. Measurements of effluent must be taken as close to laboratory analysis sample time as possible unless otherwise specified by the client or director.

8.2. Measurements of effluent should be taken directly from the location as a free-flowing source whenever possible.

8.3. Samples should not be stored for long periods of time before taking measurements, either refrigerated or otherwise, to maximize representativeness of measurements to direct conditions.

8.4. Any measurements of samples not taken by following the above directives should be noted.

9. Opening and Calibration Procedure

9.1 Turn on the ProDSS meter by pressing the power button. Fill out all information on MASSTC-FRM-033 – ProDSS Calibration Checklist.

9.2 Remove the cap to the sensor guard and sealing ring. With a lint-free wipe, gently remove any moisture from the sensors.

9.3 Record the specific conductance reading on MASSTC-FRM-033 – ProDSS Calibration Checklist. If the reading is above 1 µs/cm:

9.3.1 Clean the sensor using the small cleaning brush; dip the brush in clean water and insert it into each hole of the conductivity probe 10-12 times; rinse thoroughly with clean water. Dry the probe using a lint-free wipe and recheck the air reading and record it on MASSTC-FRM-033 – ProDSS Calibration Checklist. If reading is still above 1 µs/cm consult User Manual for cleaning the sensor port.

9.3.2 Use compressed air to blow debris from holes.

9.4 If reading is still above 1 µs/cm see MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F for instruction on cleaning the sensor port.

9.5 (If applicable) complete a conductivity calibration once per week by doing the following:

9.5.1 Use fresh, traceable conductivity calibration solution (solution can be used for one month after being opened). Write the change in standard and the calibration date on the white board in the lab. Pour into the calibration cup to the indicated line.

9.5.2 Place the probe into the calibration cup. The solution must be above the second holes on the conductivity probe.

9.5.3 Gently rotate and/or move the sensor up and down to dislodge any bubbles. Allow for at least 40 seconds of temperature equilibration.

9.5.4 Press the Calibration key and then choose Conductivity. Choose Specific Conductance.

9.5.5 Select Calibration Value and key in the standard for the given temperature; note that temperature compensation values can be found on the side of the bottle.

9.5.6 Observe the actual measurements (the white line on the graph should be flat for 40 seconds).

9.5.7 Select Accept Calibration.

9.5.8 Write the date of the Calibration and meter color as well as the date of the new standards on the white board in the lab.

9.6 Calibrate Optical Dissolved Oxygen (ODO) **daily**:

9.6.1 Make sure the sensor guard is installed on the meters. Make sure there is no water on the sensors; use a moistened lint-free wipe to gently pat them dry. Make sure the threaded black cap and ring are removed – there needs to be ample air exchange.

9.6.2 Put a small amount of tap water into the bottom of the calibration cup; water should be changed every day before doing calibration to reduce bio-fouling. There should be no other debris or fouling of the cup; clean cup with brush as needed.

9.6.3 Insert the probe into the calibration cup, making sure that the top is not sealed for atmospheric venting.

9.6.4 Wait 5-15 minutes so that the air in the cup can be saturated.

9.6.5 Press the Calibration key. Choose ODO, then choose DO%.

9.6.6 Wait for the readings to be stable – the white line on the graph should be flat for about 40 seconds. Record information on MASSTC-FRM-033 – ProDSS Calibration Checklist.

9.6.7 Write the day's ODO post-calibration value on the white board in the lab.

9.7 Complete a 3-point calibration **every day** on pH by doing the following:

9.7.1 Make sure the sensor guard is off. If standards need to be poured:

-Make sure the cups are clean.

-Pour old buffer into the allocated bottles (can be reused for rinse).

-Pour enough buffer of each 4, 7, and 10 so that the liquid level just comes to the bottom white fitting of the cup.

-Write the date of the change in standards on the white board in the lab.

9.7.2 Rinse the sensors with used 7 buffer. Always start the calibration with pH 7 buffer.

9.7.3 Carefully lower the probe into the calibration cup with pH 7 buffer solution. Make sure both the pH sensor and temperature sensor are submerged.

9.7.4 Push the Calibration key then select pH. The Calibration value will automatically be adjusted based on the selected buffer and temperature.

9.7.5 Wait for the pH mV and temperature readings to stabilize; the white line on the graph should be flat for about 40 seconds.

9.7.6 Press the Enter button to accept the calibration. **You must accept the calibration before moving onto the next standard.** The bottom of the screen should say "Ready for cal point 2".

9.7.6.1 **If the pH mV reading for the buffer solution of 7 is NOT within -50 mV to +50 mV, the meter needs further evaluation. Stop the calibration.** Consult the MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F to troubleshoot (including reconditioning probe). Consider using a different meter if the meter cannot be brought back into -50 to +50 mV.

9.7.7 Remove the probe from the 7 standard and rinse it with used buffer of the next standard you're going to calibrate.

9.7.8 Place probe in the next buffer (can be 10 or 4). Wait for the pH mV and temperature readings to stabilize; the white line on the graph should be flat for about 40 seconds. The Calibration value will automatically be adjusted based on the selected buffer and temperature.

9.7.9 Press the Enter button to accept the calibration. **You must accept the calibration before moving onto the next standard.** The bottom of the screen should say "Ready for cal point 3".

9.7.10 Remove the probe from the last standard and rinse it with used buffer of the next standard you're going to calibrate.

9.7.11 Place probe in the last buffer. Wait for the pH mV and temperature readings to stabilize; the white line on the graph should be flat for about 40 seconds. The Calibration value will automatically be adjusted based on the selected buffer and temperature.

9.7.12 **Press the Enter button to accept the calibration.** It will take you back to the calibration screen.

9.7.13 **Change pH standards 2/week (usually Monday and Wednesday).**

9.7.14 If a calibration error message occurs, do not continue calibration. Abort and restart. Pour new pH buffer standards and examine pH bulb on sensor for debris or issues. Consult MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F for further troubleshooting.

9.8 Complete a calibration verification (CCV) on the 7 pH.

9.8.1 Rinse the sensor with used 7 pH buffer and place in the 7 pH standard calibration cup.

9.8.2 Press the Probe button.

9.8.3 Choose the third option (Auto Stable) and then press Enter when highlighted.

9.8.4 Scroll to the very bottom option (Start Auto Stable) and click Enter to begin auto-stabilization. The rest of the settings should remain as is. *Current settings are for 5 samples at 10 second interval; pH stability of 0.2 units, ODO stability of 0.5 units.*

9.8.5 The meter will flash AS lettering when still stabilizing. When stable, the following will occur:

-An audible beep will sound.

-The AS lettering will be green.

-The AS lettering will no longer be flashing.

9.8.6 The choice of Log One Sample should be highlighted; press the Enter button.

9.8.7 If the incorrect site is showing, highlight the second option (Site) and press Enter. Find 1 Check 7 pH and then push Enter; on the next screen, press Enter (the screen should show Select[1Check 7 pH]. The last screen should have "Log Now!" highlighted – press Enter again.

9.8.8 The accuracy of the pH probe is ±0.2 units from the expected pH at the temperature in the solution. If the pH reading in the 7 pH buffer solution is more than ±0.2 units from the expected value, clean and recalibrate the probe. Consider consulting MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F for other troubleshooting issues as to why the reading is inaccurate.

9.9 Complete Optical Dissolved Oxygen (ODO) calibration verification.

9.9.1 Rise off probes and gently pat dry with moistened lint-free wipe.

9.9.2 Reattach sensor guard then place in calibration cup with a small amount of water in the bottom of the cup and wait 5-15 minutes to check ODO.

9.9.3 Press the Probe button.

9.9.4 Choose the third option (Auto Stable) and then press Enter when highlighted.

9.9.5 Scroll to the very bottom option (Start Auto Stable) and click Enter to begin auto-stabilization. The rest of the settings should remain as is. *Current settings are for 5 samples at 10 second interval; pH stability of 0.2 units, ODO stability of 0.5 units.*

9.9.6 The meter will flash AS lettering when still stabilizing. When stable, the following will occur:

-An audible beep will sound.

-The AS lettering will be green.

-The AS lettering will no longer be flashing.

9.9.7 The choice of Log One Sample should be highlighted; press on the Enter button.

9.9.8 If the incorrect site is showing, highlight the second option (Site) and press Enter. Find 2 Check ODO and then push Enter; on the next screen, press Enter (the

screen should show Select[2Check ODO]. The last screen should have "Log Now!" highlighted – press Enter again.

9.9.9 The accuracy of the ODO probe is ±1% from the calibration value (the post-calibration value) of that morning. If the ODO reading has been given 5-15 minutes to be fully saturated in calibration cup and is more than ± 1.0 % from that day's calibration value, clean and recalibrate the probe. Consider consulting the MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F for other troubleshooting issues as to why the reading is inaccurate.

9.10 Complete a Turbidity calibration, if applicable, for field measurements by completing the following:

9.10.1 Clean sensor guard, calibration cup, and sensor weight to ensure no biofouling. A black, unscratched sensor weight must be used to minimize reflection interference.

9.10.2 Clean off probes and volume measuring device with Ultrapure Water (obtain from virus lab). Dry probes using a lint-free cloth. Attach sensor guard and weight.

9.10.3 Pour approximately 150 mL of Ultrapure Water (obtain from virus lab) into clean volume measuring device (flask/graduated cylinder) for first calibration point to measure, then pour into marked calibration cup. **Always pour into container at an angle to minimize bubbles that enter cup.**

9.10.4 Slowly insert probes into solution.

9.10.5 Press the Calibration key, then choose Turbidity. The Calibration value will automatically be adjusted.

9.10.6 Wait for readings to stabilize; the white line on the graph should be flat for about 40 seconds.

9.10.7 Press the Enter button to accept the calibration. **You must accept the calibration before moving onto the next standard.** The bottom of the screen should say "Ready for cal point 2".

9.10.8 Clean off probes, sensor guard, weight, and measuring device with RO Water. Rinse first with RO water, followed by used 124 FNU standard in labeled rinse bottles if available. Dry using a lint-free cloth.

9.10.9 Pour approximately 150 mL of next standard (124 FNU) into clean measuring device (flask/graduated cylinder) to measure, then pour into marked calibration cup. **Always pour into container at an angle to minimize bubbles that enter cup.**

9.10.10 Wait for readings to stabilize; the white line on the graph should be flat for about 40 seconds.

9.10.11 Press the Enter button to accept the calibration. **You must accept the calibration before moving onto the next standard.** The bottom of the screen should say "Ready for cal point 3".

9.10.12 Clean off probes, sensor guard, weight, and measuring device with RO Water. Rinse first with RO water, followed by used 1010 FNU standard in labeled rinse bottles if available. Dry using a lint-free cloth.

9.10.13 Pour approximately 150 mL of next standard (1010 FNU) into clean measuring device (flask/graduated cylinder) to measure, then pour into marked calibration cup. **Always pour into container at an angle to minimize bubbles that enter cup.**

9.10.14 Wait for readings to stabilize; the white line on the graph should be flat for about 40 seconds.

9.10.15 Press Enter to complete the calibration.

9.11 Ensure that MASSTC-FRM-033 – ProDSS Calibration Checklist is completed.

9.12 If needed, perform ODO Zero Point Calibration. This calibration should be used when ODO values at known low-DO situation (such as the influent channel) are showing negative ODO readings.

9.12.1 Create a zero DO solution by mixing 8-10 grams of sodium sulfite into 500 mL of tap water. This should be done in a container that will allow sensor access. **Wait 60 minutes to ensure the solution is oxygen-free.**

9.12.2 Ensure that the ODO and Conductivity/Temperature sensors are installed on the meter, but remove any other probes and store safely.

9.12.3 Submerge the ODO and Conductivity/Temperature probes into the zero ODO solution.

9.12.4 Press the “Cal” key, then choose ODO, then Zero.

9.12.5 Allow the reading to stabilize. The white line on the graph should show no significant change for 40 seconds.

9.12.6 Press Enter to Accept Calibration. You should see “Calibration successful.”

9.12.7 **You must follow up with an ODO % calibration.**

9.12.8 Clean the sensors well to rinse off all zero-ODO solution. If any sensors were taken off (ex. pH), return to place in meter.

9.12.9 Follow step 9.6 of this document to complete the ODO % calibration.

10. TAKING MEASUREMENTS PROCEDURE

10.1 Disconnect meter from power supply and make sure cap to electronic port is closed to prevent debris from entering.

10.2 Make sure the sensor guard (the black caging) is installed and that the sonde weight is attached to the bottom of the probe. Detangle and untwist the cord as needed.

10.3 Bring meter to desired location and gently lower into place. The liquid level should come up to the bottom of the higher cylindrical holes, as indicated by a label.

10.4 Click on the Probe button.

10.5 Choose the third option (Auto Stable) and then press Enter when highlighted.

10.6 Scroll to the very bottom option (Start Auto Stable) and click Enter to begin auto-stabilization. The rest of the settings should remain as is. *Current settings are for 5 samples at 10 second interval; pH stability of 0.2 units, ODO stability of 0.5 units.*

10.7 The meter will flash AS lettering when still stabilizing. When stable, the following will occur:

-An audible beep will sound.

-The AS lettering will be green

-The AS lettering will no longer be flashing.

10.8 The choice of Log One Sample should be highlighted; press on the Enter button.

10.9 If the incorrect site is showing, highlight the second option (Site) and press Enter. Choose from the list of site names. Scroll to and highlight the desired site then push Enter; on the next screen, press Enter (the screen should show Select[sitename]. The last screen should have "Log Now!" highlighted – press Enter again.

10.10 Record any notes in the yellow field book, including initials of the staff member who took the sample and who took the field measurements.

10.11 Bring the meter to the next location and start again at step 10.3.

10.12 Every ten samples must include a continuing calibration verification (CCV) on pH 7 standard and the ODO saturation; bring the meter into the laboratory and do the following:

10.12.1 Rinse meter with tap water and remove sensor guard. Rinse sensors with used 7 pH rinse. Place in 7 pH buffer solution. Perform auto-stable and record electronically under the site name "1 Check 7 pH"

10.12.1.1 The accuracy of the pH probe is ±0.20 units from the expected pH at the temperature in the solution. If the pH reading in the 7 pH buffer solution is more than ±0.2 units from the expected value, clean and recalibrate the probe. **Do not use any pH readings taken since last acceptable calibration verification.** Consider consulting MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F for other troubleshooting issues as to why the reading is out of range.

10.12.2 Rinse off probes with deionized water and pat dry. Reattach sensor guard then place in calibration cup with a small amount of water in the bottom of the cup and wait 5-15 minutes to check ODO. Perform auto-stable and record value electronically under the site name "2 Check ODO".

10.12.2.1 The accuracy of the ODO probe is ±1.0% from the calibration value (the post-calibration value) of that morning. If the ODO reading has been given 5-15 minutes to be fully saturated in calibration cup and is more than ±1 % from that day's calibration value, clean and recalibrate the probe. **Do not use any DO readings taken since last acceptable calibration verification.** Consider consulting MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F for other troubleshooting issues as to why the reading is inaccurate.

10.13 If needed, you can add a sample location in the field. Please note that this is easier to do on the computer if possible.

10.13.1 Go through the steps of auto-stabilization.

10.13.2 Log One Sample should be highlighted; press the Enter key.

10.13.3 Scroll down to Site [] and press the Enter key.

10.13.4 Go to the top of the list and choose Add new…

10.13.5 Site Name [] should be highlighted; press the Enter key.

10.13.6 Key in the desired name and choose the Enter at the bottom of the screen when finished.

10.13.7 Scroll down to Save and push Enter.

10.13.8 Make sure this site is chosen if a sample needs to be logged here.

10.14 If sample was logged incorrectly, there must be an electronic record of this.

10.14.1 The choice of Log One Sample should be highlighted; press on the Enter button.

10.14.2 Highlight the second option (Site) and press Enter. Choose "3 PREVIOUS SITE INCORRECT" from the list of site names. Push Enter. On the next screen, press Enter (the screen should show Select[3 PREVIOUS SITE INCORRECT]. The last screen should have "Log Now!" highlighted – press Enter again.

10.14.3 Log the sample under the correct name.

10.14.4 Make a note in the yellow field book and when downloading data, make sure to be aware of this data going into the database.

10.15 To set the meter on logging mode, do the following:

10.15.1 Follow steps 10.1 to 10.3.

10.15.2 Press the System button on the meter.

10.15.3 Use the down arrows to highlight "Logging [Single]" and press Enter.

10.15.4 Scroll down to the box next to Continuous Mode and press Enter to check the box.

10.15.5 Scroll down to Site[] and make sure the correct site is chosen.

10.15.6 Scroll down to Log Interval and press Enter. Key in the correct interval in the format of HH:MM:SS.

10.15.7 Press Esc.

10.15.8 You should be at the main screen. The green bar at the top should be on "Start Logging".

10.15.9 Press Enter to Start Logging, the screen will show your log interval and site again; double-check that these are correct.

10.15.10 The green bar should be highlighting "Start Now!" Press Enter.

10.15.11 To stop logging, the green bar should be highlighting "Stop Logging" so press Enter to stop logging.

10.15.12 To take out of Logging Mode, press the System Key, go down to Logging Interval, and then uncheck the box next to Continuous Mode.

PROJECTS FOLLOWING NSF PROTOCOL

10.16 Projects following National Sanitation Foundation (NSF) protocol are especially important for documentation of field measurements as this data is reported quickly to NSF and affects the determination of the test.

10.17 Projects following NSF protocol usually have a specified pH range that the effluent sample is required to meet. Any sample that is out of the specified range could constitute a failure of the NSF test and so must be rigorously confirmed and documented.

10.17.1 Typically the acceptable range for pH readings of an NSF test is 6.0 – 9.0 SU (±0.2 SU). Consult the exact Test Plan to confirm for the project in question.

10.18 If any NSF effluent is outside of the specified range (consider only the range given by NSF, i.e. 6.0-9.0 SU, and not 5.8 to 9.2 given the ±0.2 SU), ensure to record the reading.

10.19 Stop taking measurements and complete a CCV per the steps in section 10.12. Be certain to log the values of the CCV for documentation.

10.20 Complete a new calibration of all parameters per section 9. Use new buffers/standards for all parameters to ensure the most accurate readings. Log opening CCV values again after calibration.

10.21 Retake the field measurements for the effluent of the project following NSF protocol.

10.21.1 If NSF effluent is back within range, continue with field measurements as usual.

10.21.2 If NSF effluent is still out of range for pH, repeat steps 10.19 through 10.21.

10.21.2.1 If, after a second recalibration and accompanying CCVs the effluent of the NSF project is still out of range, take final effluent reading and final CCV. Then, alert MASSTC Director so that proper notification can reach NSF personnel.

11. CLOSING PROCEDURE

11.1. Bring meter in and run under sink water to clean off outer debris. Rinse the sensor guard with clean water. Once each week, use a brush and water with dish soap to remove light bio-fouling from the sensor guard and weight.

11.2. Locate and fill out MASSTC-FRM-034 – ProDSS End of Day Checklist for the next steps.

11.3. Detangle the cord and remove any dirt.

11.4. Remove sensor guard and weight.

11.5. Rinse sensors with used 7 pH rinse. Place in 7 pH buffer solution. Record as Close value electronically under the site name “1 Check 7 pH”.

11.5.1. If 7 pH is outside of ±0.2 units from expected value, recalibrate and retake field measurements done since last acceptable check.

11.6. Clean the conductivity sensor at end of each day. Dip the sensor’s small cleaning brush in water, insert the brush at the top of the channels and sweep the channels 15 to 20 times (see MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F page 51 for diagram). Reading should be 0.0; if any higher, reclean using brush.

11.7. Rinse off sensors with deionized water and gently pat dry using a moistened lint-free delicate task wipe. Before placing into calibration cup, brush out calibration cup and replace water with new tap water. Reattach sensor guard then place in calibration cup with a small amount of water in the bottom of the cup and wait 5-15 minutes for cup to be saturated and then check ODO. Record as close value electronically under the site name “2 Check ODO.”

11.7.1. If ODO percent is outside of ±1% from that morning's ODO calibration, recalibrate and retake field measurements done since last acceptable check.

11.8. Reattach black threaded cap and sealing ring and screw onto calibration cup. This will ensure that the sensors are stored in a moist environmental for the short term (less than 4 weeks).

11.9. Make sure to download data and upload onto the MASSTC Sharepoint.

11.10. Review downloaded data to ensure that all recorded sites were recorded under the correct name and that no sites were skipped.

11.10.1. If any sites were missed, reopen meter by performing CCVs and take missing measurements. The meter must then be closed again, starting at 11.1.

11.11. Make sure the meter is connected to the power supply.

11.12. Make sure the cord is hanging off of the ground on the lab bench hook.

11.13. Make sure the cord is still attached to the meter to prevent dust entry into the meter.

11.14. Make sure the meter has been powered off (after downloading data).

11.15. Make sure you have completed MASSTC-FRM-034 – ProDSS End of Day Checklist.

12. DATA ANALYSIS/CALCULATIONS:

12.1. None

13. DATA MANAGEMENT/RECORDS MANAGEMENT

13.1. Measurement data are to be recorded on the meter and downloaded and imported into the MASSTC Data and Facility Management System. Data in CSV (Comma Separated Value) file format are to be downloaded to a backed up and secure file location each day that the meter is used. CSV files are to be imported into the MASSTC Data and Facility Management System as soon as practicable.

13.2. Observations germane to each measurement are to be recorded in indelible ink in a numbered field notebook and will be transcribed into the MASSTC Data and Facility Management System with the appropriate field measurement record as soon as practicable.

13.3. Archived data are subject to official retention schedule contained in MASSTC-SOP-003, Records and Archives.

14. QUALITY CONTROL

14.1. <u>Calibration</u>

14.1.1. pH

14.1.1.1. pH probes are to be calibrated **daily via three-point calibration with 4.00, 7.00, and 10.00 buffers**.

14.1.1.2. pH calibration standards are to be changed twice per week.

14.1.1.3. Rinses between pH calibration standards are to be done using the appropriate buffer solution to eliminate cross-contamination or dilution of buffer solutions.

14.1.2. Dissolved Oxygen

14.1.2.1. Dissolved Oxygen (DO) probes are to be calibrated **daily with fresh tap water**.

14.1.3. Specific Conductance

14.1.3.1. Specific Conductance probes are to be calibrated **weekly**.

14.1.3.2. Specific Conductance standards are to be changed once per week.

14.1.4. Turbidity

14.1.4.1. Turbidity probe is to be calibrated **weekly or on the day of use**.

14.1.4.2. Turbidity standards are to be changed once per week during weeks of use.

14.1.5. Handling of Calibration Standards

14.1.5.1. Upon receipt and subsequent opening, calibration standards are to be logged into the Chemical Receipt Log (MASSTC-FRM-014)

14.1.5.2. When standards are changed, an entry will be made into the Calibration Standards Log (MASSTC-FRM-028)

14.2. Calibration Acceptance Criteria

14.2.1. pH - ± 0.20 pH units

14.2.2. Dissolved Oxygen - ± 1.0%

14.3. Continuing Calibration Verifications (CCV)

14.3.1. pH and Dissolved Oxygen CCV's are to be done following initial calibration, every 10 non-calibration measurements, and as part of the daily meter closeout procedure.

14.3.2. pH CCV's are to use 7.0 buffer.

14.4. Measurements (General)

14.4.1. AutoStable is to be used for all readings to eliminate user bias.

14.4.2. The sonde is to be sufficiently submerged in the liquid to be measured.

14.5. Location of Measurements

14.5.1. Measurement locations are to be defined in writing by the client. Locations will be marked out on a site diagram (MASSTC-FRM-040 – Sampling Plan) and labeled with a printed ¾" label where possible.

14.5.2. Soil-Based Systems Installed at MASSTC (Non-field installations)

14.5.2.1. Final effluent is to be measured within a distribution box prior to final discharge to void.

14.5.2.2. The sonde is to be placed such that flow from the discharge pipe comes into direct contact with the probes (I.E. in very close proximity to the discharge pipe).

14.5.3. Pan Lysimeters

14.5.3.1. Liquid obtained via pan lysimeters can be measured in one of two ways:

14.5.3.1.1. Directly in the lysimeter sump, which is the preferred method.

14.5.3.1.2. By pumping a volume into a separate container.

14.5.3.2. On occasion, it may be necessary to apply a vacuum to the lysimeter port to obtain a sufficient sample. In this case, the Dissolved Oxygen values should be disregarded, and a note stating that a vacuum was used is to be entered into the log for that sample.

14.6. Timing of Measurements

14.6.1. Unless otherwise specified by the client, measurements are to be taken during times of system dosing.

14.6.2. Unless otherwise specified by the client and where practicable, measurements are to be taken within one hour of the time of laboratory sampling, if applicable.

15. Nonconformance and Corrective Action

15.1 Refer to MASSTC-SOP-003 – Control of Nonconforming Work and MASSTC-SOP-004 – Corrective Action for general nonconformance and corrective action procedures.

15.2 Calibration Nonconformance

15.2.1 If CCV is outside of acceptance criteria at any point, the meter must be re-calibrated, and all measurements taken after the last acceptable CCV must be retaken.

15.2.2 If standards are of unknown age, discard and re-pour.

15.2.3 If standards are contaminated, discard and re-pour.

15.3 Measurement Nonconformance

15.3.1 If the sonde impacts a surface with sufficient velocity (e.g. dropped from a height), a physical inspection and a CCV shall be done prior to taking any other measurements.

16. Internal and External References

16.1 MASSTC-SOP-003 – Control of Nonconforming Work

16.2 MASSTC-SOP-004 – Corrective Action SOP

16.3 MASSTC-EXT-MAN-003 – YSI ProDSS User Manual Rev. F

16.4 MASSTC-EXT-MAN-004 – YSI ProDSS Calibration Guide

16.5 MASSTC-EXT-SDS-004 - USA Bluebook pH 10.00 Buffer Solution

16.6 MASSTC-EXT-SDS-005 - USA Bluebook pH 7.00 Buffer Solution

16.7 MASSTC-EXT-SDS-006 - USA Bluebook pH 4.00 Buffer Solution

16.8 MASSTC-EXT-SDS-007 - Conductivity Standard SDS

17. Forms and Data Sheets

17.1. MASSTC-FRM-014 – Chemical Receipt Log

17.2. MASSTC-FRM-028 – Calibration Standards Log

17.3. MASSTC-FRM-030 – Calibration Worksheet

17.4. MASSTC-FRM-033 – ProDSS Calibration Checklist

17.5. MASSTC-FRM-040 – Sampling Plan

APPENDIX 2:
MASSTC TRAINING LOG

Massachusetts Alternative Septic System Test Center

Barnstable, Massachusetts

Form

Title: Training Log

Effective Date: 2020-02-11	**Number:** MASSTC-FRM-011	**Revision:** 002

Authors

Name: Brian Baumgaertel
Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... Date: 8/21/2020

Approvals

Name: Brian Baumgaertel
Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407 Date: 8/21/2020

Training Log

Document ID#: MASSTC-FORM-011
Revision#: 001
Released: 2020-02-11
Released By: Brian Baumgaertel

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site

History	Effective Date
Revision #002: Added new items and removed outdated items. Edits by BB	2020-02-11
Revision #001: Reformatted. Edits by BB.	2018-08-04
Revision #000: Original Issue	2018-02-12

Training Log

Document ID#: MASSTC-FRM-011
Revision#: 001
Released: 2020-02-11
Released By: Brian Baumgaertel

Trainee Name: _____ Service Start Date: _____ Staff Category: (P)ermanent / (T)emporar

Task	SOP Reference	Req'd For	Initial Training Date	Trainee Initials	Trainer Initials	Verification Date	Trainer Initials
Documentation and Data							
MASSTC Quality Manual - where to find, how to read	MASSTC-PLN-001	P,T					
Daily log books	MASSTC-SOP-003	P,T					
Electronic journal	MASSTC-SOP-003	P,T					
Manual entry of sample data	MASSTC-SOP-003	P					
Importing of sample data	MASSTC-SOP-003	P					
Site security (Locking Gate, etc)	MASSTC-SOP-020	P,T					
Weekday quality assurance checklist	MASSTC-SOP-021	P					
Friday quality assurance checklist	MASSTC-SOP-021	P					
Weekend quality assurance checklist	MASSTC-SOP-021	P					
System Dosing							
Morning shed checks (sign-off sheet, dosing QA sheet)	MASSTC-SOP-018	P,T					
Afternoon shed checks (sign-off sheet)	MASSTC-SOP-018	P,T					
Daily shed checks (cleaning, etc)	MASSTC-SOP-018	P,T					
Dosing pump calibration	MASSTC-SOP-018	P					
Dosing pump scheduling	MASSTC-SOP-018	P					
Sampling and Samplers							
Sample pouring technique	MASSTC-SOP-008	P,T					
Bacterial grab samples	MASSTC-SOP-007	P,T					
Programming auto samplers for uniform time	MASSTC-SOP-017	P					
Programming auto samplers for non-uniform time	MASSTC-SOP-017	P					
Programming auto samplers for flow proportion	MASSTC-SOP-017	P					
Auto sampler calibration	MASSTC-SOP-017	P					
Auto sampler maintenance	MASSTC-SOP-017	P,T					
Creating Chains of Custody	MASSTC-SOP-015	P					
Printing, applying bottle labels	MASSTC-SOP-015	P,T					

Training Log

Transportation of samples to lab	MASSTC-SOP-015	P					
Cleaning auto sampler jugs	MASSTC-SOP-017	P,T					
Field Meters and Electronic Recording Devices							
Calibration of ProDSS	MASSTC-SOP-016	P,T					
Use of ProDSS	MASSTC-SOP-016	P,T					
Closeout of ProDSS	MASSTC-SOP-016	P,T					
Ozone meter calibration	MASSTC-SOP-014	P					
Ozone meter use	MASSTC-SOP-014	P					
Power meter use	MASSTC-SOP-011	P					
Sound meter use	MASSTC-SOP-012	P					
Moisture sensor use	MASSTC-SOP-024	P					
D-Box tipper trays	MASSTC-SOP-023	P					
Facility and Equipment Maintenance							
Lift station maintenance	MASSTC-SOP-019	P					
Influent channel maintenance	MASSTC-SOP-019	P					
Effluent sumps maintenance	MASSTC-SOP-019	P					
Effluent discharge network maintenance	MASSTC-SOP-019	P					

Training Log

Document ID#: MASSTC-FRM-011
Revision#: 001
Released: 2020-02-11
Released By: Brian Baumgaertel

APPENDIX 3:
DOCUMENT CONTROL PROCEDURE

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: Document Control SOP

Effective Date: 2020-02-06	**Number:** MASSTC-SOP-001	**Revision:** 001

Authors

Name: Brian Baumgaertel
Title: MASSTC Director
Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... Date: 3/8/2021

Approvals

Name: Brian Baumgaertel
Title: MASSTC Director
Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... Date: 3/8/2021

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site.

History	*Effective Date*
002 – Added reference to MASSTC-FRM-007 for tracking deployment of new/revised controlled documents. Removed reference to watermarking archived documents.	2021-03-08
001 – Removed section 4 – Document Revision History, added new page with revision history for consistency with other Controlled Documents. Added "OSH" document type. Revised revision method. Revisions by Brian Baumgaertel.	2020-02-06
000 - Initial Release	2019-02-11

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1 General Information

1.1 Purpose

The documents that form the MASSTC Quality Management System (either internally generated or from external sources) are managed as controlled documents and information as defined in ISO/IEC 17025-2017. This procedure describes the process for development, review, authorization, control, and distribution of controlled documents.

1.2 Scope/Application

MASSTC's field quality management documents include internally generated documents which provide information regarding how to conduct business and documents which provide a format for recording information.

Quality management documents also include those of external origin used in the implementation of the quality system such as standards, regulations, equipment software, and manufacturers' manuals.

1.3 Documentation/Verification

The official copy of this procedure resides on the MASSTC SharePoint Site. The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the MASSTC SharePoint Site and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Document Control Coordinator

The Document Control Coordinator (DCC) is a quality management position appointed by management to maintain documents that form the MASSTC Quality Management System.

1.4.2 Subject Matter Expert

For the purposes of this procedure, subject matter experts (SMEs) are persons deemed competent, experienced, and knowledgeable by MASSTC management in the topic of the procedure, standard, guidance, or other subject matter for which the document is intended.

2 Internal Documents

2.1 Document Development

Internally issued MASSTC Quality Management System documents include standards, management plans, policies, manuals, operating procedures, test methods and guidance. Once the need for a new document has been identified, management assigns an author. A subject matter expert is usually assigned as either the author or a reviewer.

The following steps are used to develop, review, authorize, control, and distribute internal documents related to the MASSTC Quality Management System:

1. Author will prepare the first draft and submit it to the reviewers

2. Reviewers will provide comments to author.
3. Author will address comments and submit second draft to reviewers.
4. Reviewers will provide comments to author.
5. Steps 2-3 are repeated until all comments are addressed.
6. Once all comments have been addressed, the document will be submitted to the DCC for a format check and effective date assignment.
7. The document will then be submitted to the Director for final review and approval.

2.2 Document Format

Flexible formatting is permitted for plans, manuals and methods, depending on the applicable program. Standard Operating Procedures should follow a defined template. All internally issued documents, regardless of format, must contain the following elements:

1. Document number
2. Release Date
3. Released By Name
4. Page numbering indicating total number of pages

2.3 Document Approval

All documents generated by MASSTC, which form part of the Quality Management System, will be reviewed, and approved for use by the Director.

2.4 Review

Document review is the process through which persons with subject matter knowledge contribute to the development of internal documents. Documents are periodically reviewed (see Section 2.8) and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements. Document review includes grammatical, editorial and technical assessment.

2.5 Control

The official copy of all MASSTC Quality Management System documentation resides on the MASSTC SharePoint Site. All other electronic or printed copies are unofficial.

A master list identifying the current revision status of Quality Management System documents is available on the MASSTC SharePoint Site. The list is maintained by the DCC.

When internal documents have completed final review, they are forwarded to the DCC for a final format check, authorization and distribution, and placement in electronic form (read only) on the MASSTC SharePoint Site. The DCC will ensure that the all procedures on the MASSTC SharePoint Site are updated.

Document control numbers are assigned to MASSTC quality system documents using the following alpha-numeric scheme: MASSTC-Document type-sequential#

Example: MASSTC-SOP-001

Revisions are tracked with a 3-digit revision number, starting from “000”.

External Documents are assigned using the following alphanumeric scheme: MASSTC-EXT-Document Type-sequential#

Example: MASSTC-EXT-MAN-001

2.5.1 Document Types:

- FRM – Forms and Checklists
- INS – Insurance Form
- MAN - Manual
- MTH – Method
- OSH – Occupational Health and Safety
- PCY – Policy
- PLN - Plan
- SDS – Safety Data Sheet
- SOP – Standard Operating Procedure
- STD - Standard

2.6 Authorization

MASSTC Quality Management System documents are subject to approval by the MASSTC Director.

2.7 Distribution

The Document Control Coordinator is responsible for ensuring that all internally issued documents that form the MASSTC Field Quality System are readily available. The official copy of all Quality Management System documents resides on the MASSTC SharePoint Site. The DCC will notify all personnel within the via email of document updates and will maintain a copy of the notification. Tracking of distribution notifications will be recoded on the Controlled Document Deployment Tracking Form (MASSTC-FRM-007).

It is the responsibility of the individual to ensure that all hard and/or electronic copies of documents in their possession are the most recent version.

When documents are revised or retired, the DCC will move obsolete copies to the “Archived” folder of the MASSTC SharePoint Site.

2.8 Periodic Review and Revision

Internal documents are subject to periodic review, and where necessary, revised to ensure continuing suitability and conformance with applicable requirements. Internal documents will be reviewed at least once every four years. This schedule will apply to MASSTC internal documents issued after the effective date of this operating procedure.

In January of each year, the DCC will develop and maintain a document review schedule for the upcoming review period. The schedule will include the effective date of the most recent version of the document and the review date. The DCC will update the review schedule as additional reviews are conducted or new documents are developed. The following procedure will be followed:

1. The DCC will notify the author of the need for the review.
2. The author will review the procedure to determine if updates are needed. If so, the author will notify the DCC and proceed to step 3 below. If no updates are needed, the author will notify the DCC.
3. The DCC will consult with the Director to assign reviewers

4. The author will update the procedure and provide the first draft to the reviewers.
5. The reviewers will provide comments to the author.
6. Steps 4-5 are repeated until all comments are addressed.
7. Once all comments have been addressed, the document will be submitted to the DCC for a format check and effective date assignment.
8. The document will then be submitted to the Director for final review and approval.

Changes Quality Management System documents will be clearly indicated in the revision history of the document, except for forms which will be indicated in a separate record. MASSTC's document control system does not allow for the temporary amendment of Quality Management System documents by hand

3 External Documents

Documents of external origin referenced in the implementation of the MASSTC Quality Management System may include national and international standards, EPA manuals and directives, manufacturer's manuals, equipment software, and other associated types of information. External documents will be reviewed for context to determine their applicability in the MASSTC Quality Management System

When applicable, national and international standards, and EPA manuals and directives will be controlled by documenting, as appropriate, the title, document number (if any), and date of publication. Documents will be assigned document control numbers as described in Section 2.5 of this procedure. The DCC will maintain a list of all controlled external documents. The DCC will review the list each January and consult with personnel to determine if updates are available. If they are available, the obsolete copy will be removed from service and the updated version will be labeled with the next revision of the document control number. On a case by case basis, the DCC in consultation with affected personnel will determine if it is necessary to maintain a copy of the previous version.

APPENDIX 4:
DATA AND RECORDS MANAGEMENT

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: Data and Records Management

Effective Date: 2021-07-30	**Number:** MASSTC-SOP-003	**Revision:** 001

Authors

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Approvals

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Title: MASSTC Director

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REVISION HISTORY

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site.

History	Effective Date
Revision #001: Added direction about properly voiding blank space. Edits done by Emily Michele Olmsted.	2021-07-30
Revision #000: Original Issue	2021-02-02

Data and Records Management	Document ID#: MASSTC-SOP-003 Revision: 001 Released Date: 2021-07-30 Released By: Brian Baumgaertel

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1. PURPOSE

1.1. This document defines the policies and procedures by which MASSTC shall record and maintain data, notes, and other documentation.

2. SCOPE AND APPLICATION

2.1. This procedure applies to all work conducted by all MASSTC personnel.

3. DEFINITIONS

3.1. Data – Facts about an object [Source: ISO 9000:2015, 3.8.1]

3.2. Document – Information, and the medium on which it is contained. Examples: record, specification, procedure document, drawing, report, standard. [Source: ISO 9000:2015, 3.8.5]

3.3. Record – Document stating results achieved or providing evidence of activities performed. [Source: ISO 9000:2015, 3.8.10]

4. RECORD IDENTIFICATION

4.1. Records are identifiable to the firm, product, person, or event to which they pertain. Records are dated and identify the person who established the record.

4.2. Laboratory records contain sufficient information to maintain an audit trail.

5. DATA FORMATTING

5.1. To maintain consistency in MASSTC records, data shall be recorded in the following formats:

5.1.1. Date – ISO 8601 format: YYYY-MM-DD. Example: 2020-03-27

5.1.2. Time – ISO 8601 format: HH:MM where hours are based on a 24-hour clock. Example: 13:35

5.1.3. Temperature – ISO 80000 format: degrees Celsius or shorthand °C. Example: 5.5°C

5.1.4. Length – SI Units: meters (m), centimeters (cm), millimeters (mm), etc. Example: 15mm

5.1.5. Volume –

5.1.5.1. Gallons (gal) in the context of tank sizes and system flow. Examples: 1,500-gal tank, 7.5 gallons per dose.

5.1.5.2. Liters (L), or milliliters (mL) in every context not defined in 4.6.1.

5.1.6. Mass – SI Units: kilogram (kg), gram (g), milligram (mg), microgram (µg), nanogram (ng).

5.1.7. Concentration – milligrams/liter (mg/L), micrograms/liter (µg/L), nanograms/liter (ng/L).

5.1.8. Electrical Power Use – kilowatt-hours (kWh)

6. HANDWRITTEN DATA

6.1. All data shall be recorded in permanent, indelible ink which cannot be erased.

6.2. Accuracy should always take precedence over expediency. Ensure that a future reader will be able to read what you have written. For example, a hastily-written zero (0) can appear like a six (6) or vice-versa.

6.3. Handwritten data should be converted to electronic format whenever possible. Where an electronic recording method exists, data shall be recorded by the end of the business day.

6.4. Column Headings - column headings shall be carried over between pages.

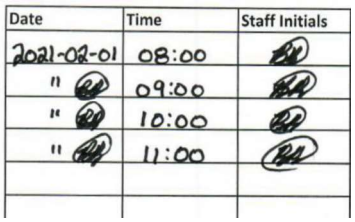
6.5. Skipping lines or pages – the space between handwritten observations or data shall be as minimal as possible to prevent nonchronological additions or observations from being added.

6.5.1. If any space is intentionally left blank, a single diagonal line striking through the space shall be included with the initials of the person recording the strike and the date of striking the blank space, as well as a brief explanation or the word "VOID."

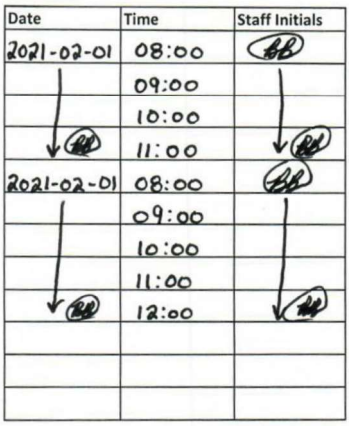
6.6. Errors - shall be struck with a single horizontal line, with the editor's initials placed nearby.

6.7. Repeating rows in tables – two methods are acceptable:

6.7.1. "Ditto" or quotation marks, with initials



6.7.2. Down arrow, with initials



7. MASSTC Electronic Data Management System

7.1. The MASSTC Electronic Data Management System is accessible internally or via VPN at http://10.14.20.130:31983/

7.2. Care should be taken to include as much detail and pertinent events and information in the electronic data management system, as known as the MASSTC Database.

7.3. Care should be taken to ensure that the recording of any event or note is stored in the best place possible with as correct an association as possible.

7.4. The electronic Journal shall function as the general place to record notes and events unless otherwise instructed.

7.4.1. Each entry automatically includes the time, date, and the user. If the event or note being recorded took place at a different time and date, the date and time must be changed by the user to when the event occurred.

7.4.2. In most cases, an entry should be logged in association with an existing project.

7.4.3. In most cases, entries can be logged for a specific asset, infrastructure, and/or equipment if not already associated with a specific project.

7.4.4. The correct category should be chosen for the Journal entry when possible.

7.4.5. Follow any protocol for Journal entry if directed elsewhere (ex. sampling plans, Weekly Quality Assurance tasks).

7.4.6. Journal entries should be used for the following examples:

7.4.6.1. Routine inspections of equipment or assets which do not otherwise have a designated physical location of records (ex. sump checks).

7.4.6.2. Unanticipated system issues with flow, mechanics, or otherwise.

7.4.6.3. Site issues, especially those relevant to the influent channel.

7.4.6.4. Any other circumstances which would be beneficial to have on record.

7.5. Shed checks must be recorded electronically and should include counter information, where applicable. Any notes regarding system issues or other relevant information should be included in the comments section of a shed check.

7.6. Influent pump schedules must be updated online as soon as possible after a pump schedule is changed to ensure that shed checks can remain accurate.

7.7. Laboratory Chains of Custody must be created in the MASSTC Database, per direction of MASSTC-SOP-015 – Sample Preparation and Transportation.

7.8. Projects should be kept as up to date as possible with information such as the start-up, flow, assigned pump, and activity status retained in the digital record in the MASSTC Database.

8. Digital Records Storage

8.1. All MASSTC staff must use Microsoft Office's Sharepoint for work-related documents. Sharepoint exists as a source for storing large amounts of files and eliminates the need for files to be backed up on an external location in the event of local equipment failure.

8.2. Documents that are necessary to one or more MASSTC staff members shall **not** be stored locally, such as through the use of OneDrive or on a computer's desktop, because they are not accessible to all staff.

8.3. Care should be taken when saving a file to Sharepoint to ensure it is stored in the best possible location so that it can be easily found by other staff, including a clear document title and a date formatted as specified in Section 5 whenever possible.

8.4. Consult MASSTC-SOP-001 – Document Control Procedure for Controlled Documents, which must also be stored on Sharepoint.

8.5. The MASSTC-Documents Sharepoint should be used to save all other MASSTC-Documents not falling under the designation of controlled documents.

8.6. The following should be guidance when saving uncontrolled documents:

8.6.1. All documents specific to one project should be saved within the correct Client and Project folder within MASSTC-Documents.

8.6.2. All records pertaining to the upkeep or inspection of MASSTC site equipment or assets should be saved in the Facility folder of MASSTC-Documents.

8.6.3. All lab reports shall be saved in the Labscans folder within the Data folder of MASSTC-Documents. Lab reports shall be retitled in the format of LIMS Client ID_Sample ID_Date of Sample_LIMS ID.

8.6.4. All records and data pertaining to the ProDSS field meter, including scanned Calibration documents, End of Day documents, and direct measurements taken by the meters shall be saved in the ProDSS Downloaded Data folder within the Data folder of MASSTC-Documents.

9. Record Retention Schedule

9.1. Records retention schedules are defined in the Massachusetts Municipal Records Retention Schedule (MASSTC-EXT-PLN-002).

9.2. Records retention schedules not defined in the above are as follows:

Record	Cutoff	Retention
Employee Training Records	End of FY after employee leaves.	5 years
Instrument Calibration Records	End of FY after final action.	3 years
Client Project Data/Notes	End of FY after project completion.	10 years
Grant Project Data/Notes	As defined in grant agreement OR end of FY after project completion.	As defined in grant agreement OR 10 years.
Shed Check Records	End of FY.	5 years
Weekly/Weekend Checklists	End of FY.	3 years
Dosing Pump Schedules	End of FY after project completion.	3 years

APPENDIX A – ELECTRONIC RECORD AND DATA FILE HIERARCHY

Type	Examples	Path
Client project records and data	Tipper data, HOBO Data, pictures	Projects/Client Projects/[*client identifier*]/[*project identifier*]/
Grant project records and data		Projects/Grant Projects/[*grant project identifier*]/
Lab Reports		BCDHE Lab Reports/{ *LIMS Client ID_Sample ID_Date of Sample_LIMS ID*}
Field Meter records and data	Calibration records, end of day records, data exports	Field Meter
General Facility Log Sheets (Including Scans)	Alkalinity logs, sampler check logs	Facility/[*Log Title*]/
Pump Schedules		Facility/Pump Schedules/[Pump Identifier]/
Weather Data		Facility/Weather Data

APPENDIX 5: SAMPLE COLLECTION

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: Sample Collection

Effective Date: 2021-11-17	**Number:** MASSTC-SOP-037	**Revision:** 002

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Sample Collection	Document ID#: MASSTC-SOP-037 Revision#: 001 Released Date: 2021-11-17 Released By: Brian Baumgaertel

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site.

History	Effective Date
Revision #002: Added field blank to section on virus sample analysis. Reformatted coversheet to include QAM approval. Edits done by EMO.	2021-11-17
Revision #001: Expanded definition of whaler pump to include details about power supply. Added section about sampling lysimeters. Added section about virus samples. Added section about collection from external plants. Edits done by EMO and GH.	2021-09-22
Revision #000: Original Issue	2021-07-20

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1. Scope and Application

1.1. This protocol describes the procedure used to collect water and/or wastewater samples for analysis.

2. Definitions

2.1. Aliquot – a sample taken for chemical analysis or other treatment that represents a portion of the whole; composite samples.

2.2. Aliquoting – the process of dividing a whole sample into aliquots.

2.3. "Bellies" – low spots in sample tubing that can hold liquid. This liquid is susceptible to freezing and can provide conditions for biological growth. See Section 5 – Interferences.

2.4. Composite sample – a mixture of grab samples taken over a period of time, usually 24 hours.

2.5. Composite sample cup – a device fitted to an effluent discharge pipe that maintains a small reservoir of liquid to enable collection by a composite sampler. Cup should have holes to allow drainage and to ensure sampler is accessing new source of non-stagnant liquid.

2.6. Dipper pole – a sampling device composed of a cup attached to a pole. The cup is constructed so that a sterile sample bottle can be slipped in. The pole is of sufficient length to allow the user to avoid entering a confined space or other hard-to-reach area.

2.7. Grab sample – an individual sample taken without the addition of other samples.

2.8. Grabber stick – a device that allows user to close hooks on an object by use of a stick, usually acting as an extension of hands in locations that are difficult to reach.

2.9. Peristaltic sample pump – a type of pump used to "draw up" a sample.

2.10. Project-specific sample tubing – sample tubing which is cut to length for a specific project sampling location to minimize low spots ("bellies") between the sample location and sample equipment.

2.11. Project-specific sample carboy – a sample carboy that is labeled and used for one specific project sampling location. See also "sample carboy".

2.12. Sample bottle – a capped container made of plastic, glass, or other material to contain, prevent contamination of, and securely transport a sample from a sample location to the laboratory where the sample is analyzed.

2.13. Sample carboy – a capped 10-liter Nalgene or equivalent container which is maintained in a clean condition which is used to collect composite samples from an automated sampling device.

2.14. Sample tubing – a section of plastic tubing that is maintained in a clean condition.

2.15. "Whaler" sample pump – a sample pump attached to tubing and a power supply (usually a 12-volt DC battery encased in weather-proof housing unit), often used to sample lysimeters or other hard-to-access locations

3. Health and Safety Warnings

3.1. **Physical Hazards** – use care and good judgement when taking samples. If a sample location is in a place where it cannot be safely collected (e.g. confined space), notify the MASSTC director immediately and do not attempt to retrieve it. Environmental conditions (e.g. rain, snow, etc.) can lead to uneven and/or slippery surfaces so care should be taken to prevent slips and fall. **PPE Required: Closed-toe shoes/boots. Care should be taken to dress appropriately.**

3.2. **Infectious Materials** – even the cleanest wastewater can contain pathogens or toxicants. Proper precautions should be taken to isolate yourself. **PPE Required: gloves, goggles.**

3.3. **Skin Corrosion/Serious Eye Damage** – Some sample bottles contain sulfuric acid (H_2SO_4) as a preservative. **Gloves and safety goggles required.**

4. Cautions

4.1. Whaler sample pumps are hand-made sample retrieval equipment and usually have some unprotected wires. Care should be taken to handle wire connections as gently as possible.

4.2. All sampling bottles must be clearly labelled prior to sampling. Sampler should be familiar with sampling port locations and exercise all safety precautions before accessing areas for wastewater sampling.

4.3. Consult *MASSTC-FRM-040 – Sampling Plan* to ensure the proper samples at the proper location as confirmed by client.

5. Interferences

5.1. Temperature of composite sample should be taken before placing aliquot in refrigeration to ensure the reading is representative of the temperature of the sample in situ.

5.2. Composite samples can become contaminated while transporting from the sampler to the field lab. Aliquot caps should be used.

5.3. Samples can become contaminated while transferring between containers. Care should be taken to sample directly from the source whenever possible.

5.4. Pathogen samples are usually sensitive to ultraviolet light. Keep sterile bottles containing sample in as dark of a condition as possible when transporting from field to longer-term holding (ex. refrigerator).

5.5. Sample retrieval equipment (pumps, tubing, etc.) should be thoroughly cleaned in between sample locations to prevent contamination of samples in between sites.

6. Personnel Qualifications

6.1. Personnel are required to be knowledgeable of the procedures in this SOP.

6.2. Personnel should be trained in the proper use of Personal Protective Equipment (PPE).

7. Special Apparatus and Materials

7.1. Influent channel composite sampler setup (Section 10)

7.1.1. Influent sample carboy and clean carboy cap.

7.2. Project-specific composite sampler setup (Section 11)

7.2.1. Portable or refrigerated sampler.

7.2.2. Project-specific tubing, which attaches to peristaltic pump of composite sampler and draws liquid.

7.2.3. Project-specific composite sample cup.

7.2.4. Power supply – battery or plug in AC adapter.

7.2.5. Ice, if using portable sampler, usually two bags unless below-freezing temperatures expected.

7.2.6. Project-specific sample carboy and clean aliquot cap.

7.2.7. Dipper pole to reach sample locations without confined space entry.

7.2.8. Grabber stick to reach cup or tubing without confined space entry.

7.3. Composite sample aliquoting (Section 12).
  7.3.1. Clean sample bottles.
7.4. Influent channel grab samples (Section 13)
  7.4.1. Clean sample bottles.
  7.4.2. Dipper pole.
7.5. Lysimeter Samples (Section 16)
  7.5.1. Clean sample bottles. Sterile if for biological analysis.
  7.5.2. "Whaler" or peristaltic sample pump and accompanying tubing (depends on location; see definition for more detail).
  7.5.3. YSI ProDSS, depending on volume amount.
7.6. Project-specific grab samples (Sections 14 and 15)
  7.6.1. Clean sample bottles. Sterile if for biological analysis.
  7.6.2. Dipper pole (depends on location).
  7.6.3. "Whaler" or peristaltic sample pump and accompanying tubing (depends on location; see definition for more detail).
7.7. Virus Samples (Section 19)
  7.7.1. Clean, sterile one-gallon HDPE handled containers. See section 19 for instruction on appropriate disinfection.
  7.7.2. PVC cap and tube assembly
  7.7.3. Tape
7.8. Procedure for Wastewater Collection from Locations at External Wastewater Treatment Plants (Section 20)
  7.8.1. Clean, sterile one-gallon HDPE handled containers. See section 20 for instruction on appropriate disinfection.
  7.8.2. "Whaler" or peristaltic sample pump and accompanying tubing (depends on location; see definition for more detail).

8. Instrument or Method Calibration

8.1. Thermometers should be calibrated per *MASSTC-SOP-013 – Thermometer Calibration SOP*.

9. Sample Preparation, Storage, and Transportation

9.1. Consult *MASSTC-SOP-015 – Sample Preparation and Transportation*.

10. Procedure for Influent Channel Composite Sample Setup and Retrieval

10.1. Set up influent channel composite sampler
  10.1.1. The influent channel composite sampler should be set up 24 hours prior to the anticipated sample retrieval time.
  10.1.2. Obtain equipment needed (see section 7.1)
  10.1.3. Bring all equipment to sample location

10.1.4. Inspect sampler tubing for damage, freezing, "bellies", and bio-growth. Tubing should be replaced prior to initiation of sampling if needed.

10.1.5. Place the project-specific sample carboy in the refrigeration compartment.

10.1.6. Pass the sample tubing through the hole in the sample carboy cap.

10.1.7. Place the refrigerator temperature probe in contact with the sample carboy.

10.1.8. Press "On" button on peristaltic sampling pump.

10.1.9. Verify that the first pump cycle runs and that a sample is properly drawn up through the sampler tubing and into the sample carboy.

10.1.10. Fill out the appropriate row on *MASSTC-FRM-020 – Daily DC West Sampler Logs*.

10.2. Retrieve the sample carboy from the sampler after the proscribed period of time (i.e. 24 hours).

10.2.1. Remove the holed sample carboy cap and affix a solid sample carboy cap to prevent contamination during transport from field to pouring station.

10.2.2. Check that sample carboy label is correct for the location being sampled.

10.3. Follow the Procedure for Composite Sample Aliquoting into Sample Bottles (Section 12)

10.4. Consult *MASSTC-SOP-015 Sample Preparation and Transportation*.

11. Procedure for Project-Specific Composite Sample Setup and Retrieval

11.1. Consult *MASSTC-FRM-040 – Sampling Plans* to confirm location of project-specific composite sample.

11.2. Set up composite sampler.

11.2.1. Composite samplers, unless directed differently by client, must be set up 24 hours in advance of the anticipated retrieval time.

11.2.2. Obtain equipment needed (see section 7.2)

11.2.3. Bring all equipment to sample location.

11.2.4. Place project-specific sampling cup/collection container under liquid discharge pipe. Ensure cup is draining to allow for fresh, non-stagnant sample.

11.2.5. Secure tubing in sampling cup/container, usually through a zip tie loop.

11.2.6. Attach tubing to composite sampler, ensuring to place tubing through hole in riser, if applicable, so that riser can be covered without obscuring the tubing.

11.2.7. Place carboy in sampler. Add ice, if not using refrigerated sampler, ensuring that no ice enters the carboy. **Check that carboy label is correct for the project.**

11.2.8. Close up sampler, ensuring that the top fits or that any other internal tubing will allow liquid to enter carboy. Close riser, if applicable.

11.2.9. Attach power supply.

11.2.10. Program sampler to normal conditions – 48 composite samples over 24 hours with tubing rinses – unless directed otherwise by client or MASSTC Director.

11.2.11. **Remain at the sampler to visually confirm the first sample is taken.** This can decrease the loss of any samples due to errors in set up (ex. tubing not submerged in collection cup).

11.2.12. Throughout the rest of the day, check on sampler to ensure that there are no issues and that the ice supply is acceptable. Add a third bag of ice in hot temperatures (usually above 80° F).

11.3. Retrieve the sample carboy from the sampler after the proscribed period of time (i.e. 24 hours).

11.3.1. Affix a solid sample carboy cap to prevent contamination during transport from field to pouring station.

11.3.2. Check that sample carboy label is correct for the location being sampled.

11.4. Follow the Procedure for Composite Sample Aliquoting into Sample Bottles (see section 12)

11.5. Consult *MASSTC-SOP-015 Sample Preparation and Transportation*.

12. Procedure for Composite Sample Aliquoting into Sample Bottles

12.1. Bring composite sample carboy to secure, clean space (usually the kitchen sink in the main office trailer).

12.2. Record temperature and volume of aliquot sample as soon as possible. Aliquot temperature and sample volume should be recorded in the aliquot logbook.

12.3. Inspect sample bottles for improper labels, physical damage, and cleanliness.

12.4. Place opened sample bottles in sink. Be sure to keep preserved and non-preserved caps separate.

12.5. With aliquot cap firmly in place, shake vigorously for 10 seconds.

12.6. Remove aliquot cap and pour non-sterile samples, being careful to maintain a constant stream of liquid from the aliquot. Fill each sample bottle as needed (preserved bottles should not be overfilled), ensuring to leave headspace.

12.7. Recap sample bottles, ensuring that preserved caps are placed on preserved bottles and unpreserved caps are placed on unpreserved bottles.

12.8. Rinse sample bottles in clean tap water, followed by a disinfecting rinse, and one final rinse with tap water.

12.9. Place completed sample bottles in refrigerator. Record time of sample as the last composited sample time on chain of custody, along with initials of sampler.

12.10. If project is following National Sanitation Foundation (NSF) protocol, store leftover aliquot in refrigerator for 24 hours.

12.11. If interrupted during pouring process, return to step 12.4.

13. Procedure for Influent Channel Grab Samples (Sterile)

13.1. Obtain equipment needed (see section 7.4)

13.2. **Double-check that you have all bottles and that they are for the correct location and date.**

13.3. Two methods for sampling are acceptable:

13.3.1. By hand (with proper PPE):

13.3.1.1. Remove the sample bottle cap, being careful not to touch the inside or the cap or bottle with your hand or other object.

13.3.1.2. Dip the sample approximately 5-10 inches into the wastewater at a 45-degree angle.

13.3.1.3. When full, bring the bottle to the surface, and pour off a small amount to leave an air gap at the top of the bottle.

13.3.1.4. Immediately re-cap the bottle.

13.3.2. Using a dipper pole:

13.3.2.1. Place a capped (sterile, if for biological analysis) 100mL sample bottle in the dipper pole cup.

13.3.2.2. Remove the sample bottle cap, being careful not to touch the inside or the cap or bottle with your hand or other object.

13.3.2.3. Dip the sample bottle approximately 5-10 inches into the wastewater.

13.3.2.4. When full, bring the bottle to the surface, and pour off a small amount to leave an air gap at the top of the bottle.

13.3.2.5. Immediately re-cap the bottle.

13.4. To the extent possible, keep the filled sample bottle out of sunlight if doing biological analyses.

14. Procedure For Project-Specific Grab Samples (Non-Sterile)

14.1. Consult *MASSTC-FRM-040 – Sampling Plan* to confirm location of sample and any particular instructions.

14.2. Secure the correct bottles for the particular sampling location to be sampled. **Double-check that you have all bottles and that they are for the correct location and date.**

14.3. Obtain equipment needed (see section 7.5)

14.4. Bring clean water to rinse equipment if sampling more than one site in the field.

14.5. Go to sample location and collect sample. Use the following in the order of preference unless directed otherwise by client (check the Sampling Plan!):

14.5.1. Collect sample by uncapping bottle(s) and placing under free-falling stream of liquid.

14.5.2. If free-falling stream has very slow rate of flow, an alternative, approved, and thoroughly-cleaned container may be used to collect liquid. Take care to place container so that it is not contaminated by another other sources until collection and pouring of bottles.

14.5.3. If it is not possible to either safely or adequately place bottles under free-falling stream, collect sample by using bottle secured to dipper pole and pouring into uncapped sample bottles. Rinse bottle secured to dipper pole at least three times with sample liquid before pouring into sample bottles to minimize any contamination or dilution from bottle secured to pole.

14.5.4. Use whaler pump to collect liquid from a pooled source (such as from a sump or a lysimeter). Ensure that proper purging has been completed per *MASSTC-FRM-040 – Sampling Plan* if necessary for site.

14.6. Recap sample bottles, ensuring that preserved caps are placed on preserved bottles and unpreserved caps are placed on unpreserved bottles. **Double-check that the sample bottles have the correct label.**

14.7. Carefully bring bottles inside as soon as possible.

14.8. Rinse sample bottles in clean tap water, followed by a disinfecting rinse, and one final rinse with tap water.

14.9. Place completed sample bottles in refrigerator. Record time of sample on chain of custody, along with initials of sampler.

14.10. Consult *MASSTC-SOP-015 Sample Preparation and Transportation*.

14.11. Clean any sample retrieval equipment before returning.

15. Procedure For Project-Specific Grab Samples (Sterile)

15.1. Consult *MASSTC-FRM-040 – Sampling Plan* to confirm location of sample and any particular instructions.

15.2. Secure the correct sterile bottles for the particular sampling location to be sampled. **Double-check that all bottles are for the correct location and date.**

15.3. Obtain any sampling equipment needed. **Clean all sampling equipment before use, including disinfection if needed.**

15.4. Commonly used sample retrieval equipment includes:

15.4.1. Dipper pole

15.4.2. Whaler pump and tubing (long or short) attached to battery pack.

15.4.3. Peristaltic pump.

15.5. Bring any clean water needed to rinse equipment if sampling more than one site in the field.

15.6. Go to sample location and collect sample. **Ensure that cap and bottle do not touch anything besides the liquid sample. If bottle or cap is compromised, discard the bottle and obtain a new sample in a new bottle.**

15.7. Use the following in the order of preference unless directed otherwise by client:

15.7.1. Collect sample by uncapping bottle(s) and placing under free-falling stream of liquid.

15.7.2. If it is not possible to either safely or adequately place bottles under free-falling stream, collect sample by using dipper pole.

15.7.3. If free-falling stream has very slow rate of flow, an alternative, approved, and thoroughly-cleaned container may be used to collect liquid. Take care to place container so that it is not contaminated by another other sources until pouring.

15.7.4. Use whaler pump to collect liquid from pooled source (such as from a sump or a lysimeter). Ensure that proper purging has been completed per *MASSTC-FRM-040 – Sampling Plan* if necessary for site.

15.8. Be sure to leave an air gap at the top of the bottle to ensure adequate headspace.

15.9. Recap sample bottles, ensuring that nothing touches the cap or bottle, as this can contaminate the aseptic sample. **Double-check that the sample bottles have the correct label.**

15.10. Carefully bring bottles inside as soon as possible. **Keep bottle in as dark of a condition as possible while still in field, as many pathogen samples are sensitive to ultraviolet light.**

15.11. Rinse sample bottles in clean tap water, followed by a disinfecting rinse, and one final rinse with tap water.

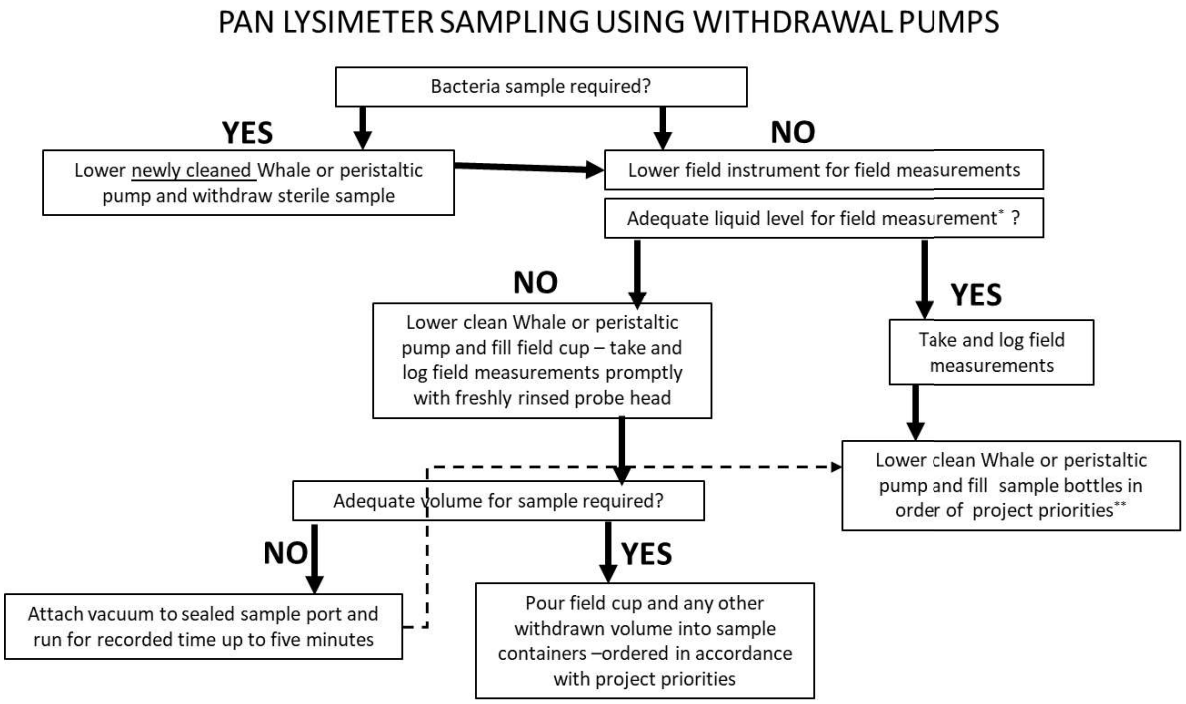
15.12. Place completed sample bottles in refrigerator. Record time of sample on chain of custody, along with initials of sampler.

15.13. Consult *MASSTC-SOP-015 Sample Preparation and Transportation*.

16. Procedure for Samples from Lysimeter

16.1. Obtain any sampling equipment needed. **Clean all sampling equipment before use, including disinfection if needed. See section 7.5.**

16.2. Take samples with whaler pump according to flow chart:



- To determine if there is adequate level for field measurements, use the specific conductance. In most wastewater samples specific conductance should exceed 200 uS. If less, in most instances conclude "inadequate liquid level" or use wetness of probe as indicator.

** In some instances where preserved bottles are required, it is preferable to fill clean one-liter no-preservative bottles and pour into preserved bottles from these. This also allows easier apportionment of samples to priority analyte bottles if necessary.

16.3.

17. Procedure for Ozone Samples

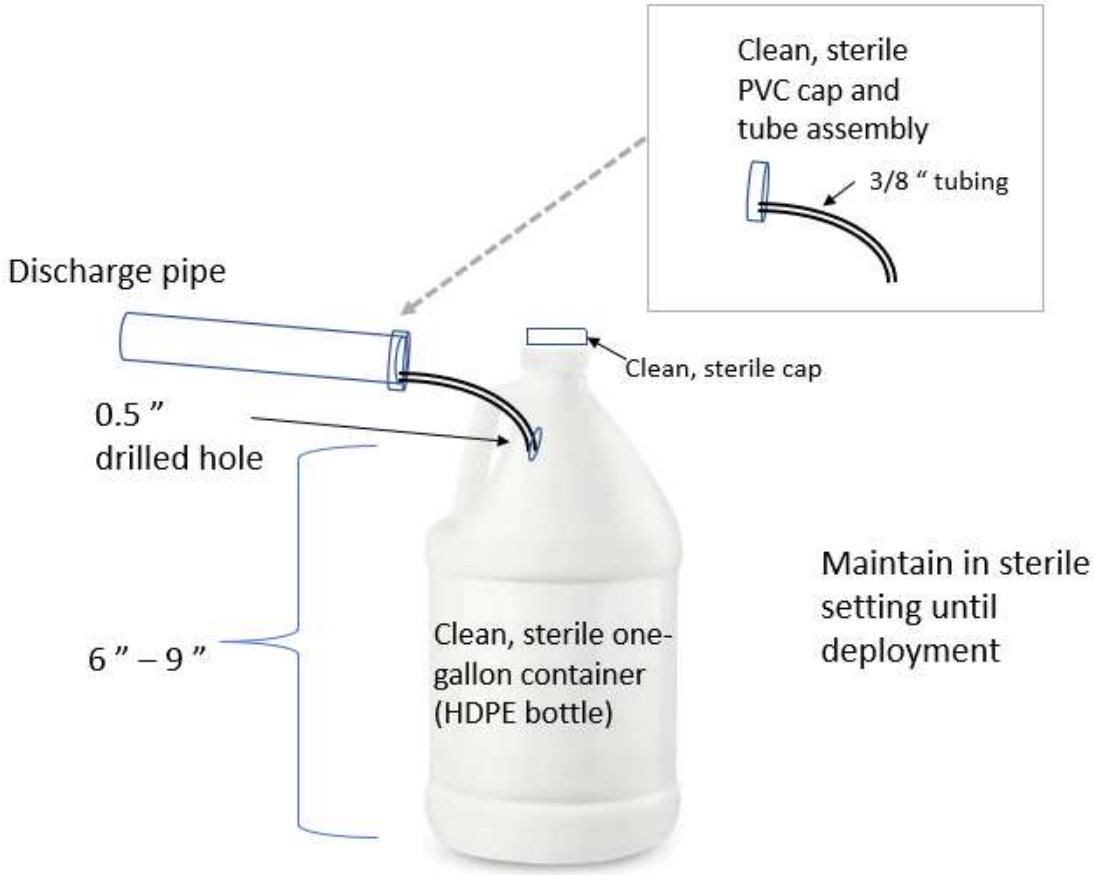
17.1. Consult *MASSTC-SOP-014 – Ozone Measurement.*

18. Procedure for Field samples

18.1. Consult *MASSTC-SOP-016 – YSI ProDSS Field Meter.*

19. Procedure for Virus Samples

19.1. Samples under this section can be retrieved from any location where there is a discharge location comprised of a two-inch PVC pipe below which is a five to six-inch free space or drop. The locations include standard stone-trench systems, shallow-placed soils-based systems, wood-based systems and others where the discharge point is comprised of a two-inch PVC pipe. The completed setup of the sampler is shown here:



19.1.1.

19.2. Ensure that all equipment has been properly disinfected.

19.2.1. Disinfection is performed by adding 1/3 cup of bleach to enough tap water to achieve the full volume of the one-gallon container HDPE bottle and cap.

19.2.2. The solution must remain in the bottle for at least ten minutes and be swirled to contact all surfaces.

19.2.3. Following emptying, the HDPE bottle is then subjected to at least a four-volume full rinse with tap water and stored in a clean, dry location until use.

19.2.4. The cap-and-tube assembly is submersed in a bleach solution (1/3 cup of bleach per gallon) for a minimum of ten minutes followed by a rinse with tap water.

19.2.5. These devices should be stored in a clean, dry location until use.

19.3. Ensure proper sampler deployment.

19.3.1. Prior to deploying the sampler device, the distal end of the two-inch discharge pipe should be cleaned with an alcohol wipe and dried with a paper towel.

19.3.2. The cap-and-tube assembly should be affixed to the discharge pipe with the tube on the bottom round of the pipe. Allow the flow to establish through the 3/8" tube. If you observe any leakage around the cap, tape around the cap to stop the leakage.

19.3.3. Once flow from the tube is observed, direct the tube through the drilled hole in the collection bottle. Make sure the cap is on the bottle to prevent contamination. Make sure that there is no discharge from adjacent pipes near the hole in the collection bottle.

19.3.4. Allow the bottle to fill up to the drilled hole in the bottle. The bottle may be allowed to overfill and spill out the hole in the sample bottle.

19.4. Ensure proper sample collection.

19.4.1. When collecting the sample, be careful not to allow contamination to enter the bottle.

19.4.2. Pull the bottle away from the sample tube and place tape across the hole in the sample bottle.

19.4.3. Use a fresh piece of tape from the roll that has not been exposed prior to removal.

19.4.4. Label the sample with the field location designator and record on chain of custody.

19.4.5. Replace the sample bottle with a container so that field parameters can be measured.

19.4.6. Deliver the sample to the laboratory immediately and place in the refrigerator.

19.5. Collect a field blank each time samples are collected.

19.5.1. Obtain clean, sterile one-gallon container (HDPE bottle) as used for virus analysis.

19.5.2. Label the sample with the field blank designator and record on chain of custody.

19.5.3. Fill with unchlorinated tap water and cap it.

19.5.4. Bring one-gallon container with tap water to sample location and leave among other set ups.

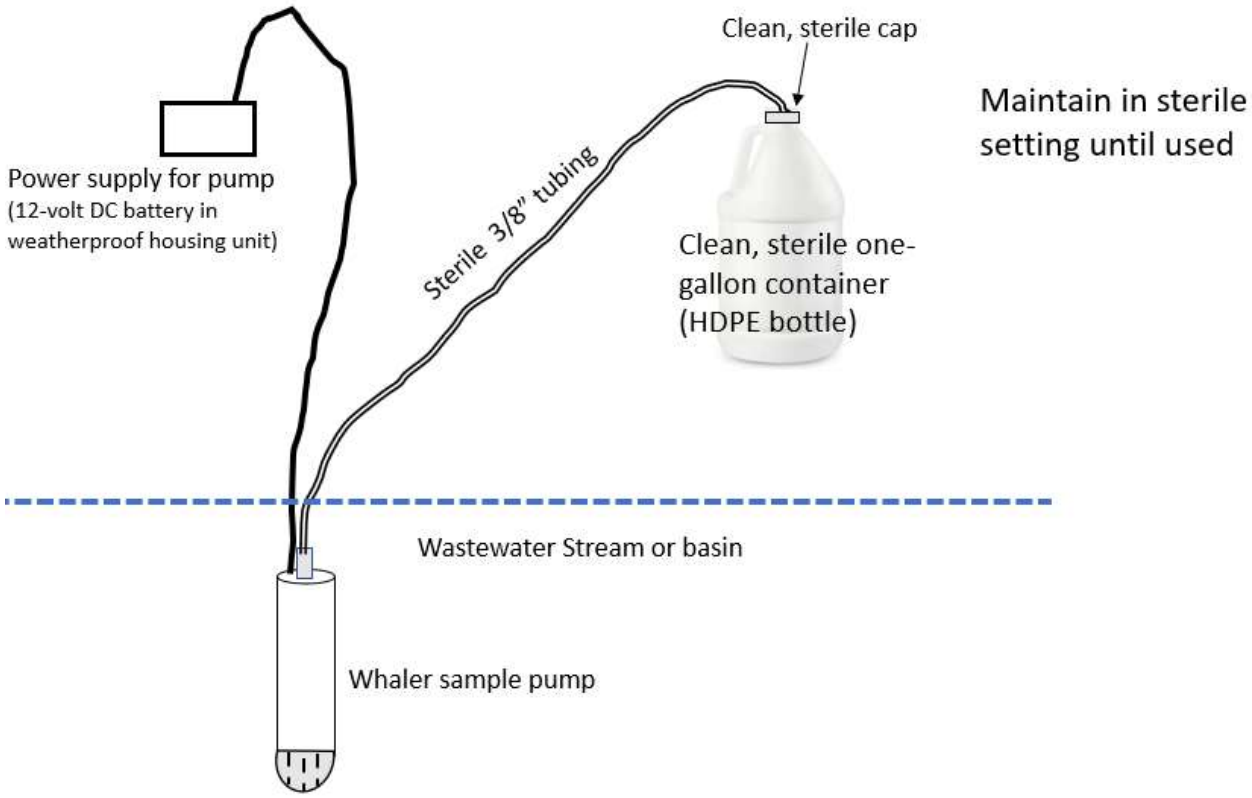
19.5.5. Subject the field sample to the same conditions (travel, temperature, etc) as other samples.

19.5.6. Deliver to laboratory with samples for virus analysis and place in refrigerator.

20. Procedure for Wastewater Collection from Locations at External Wastewater Treatment Plants

20.1. Samples under this section can be taken at any external location that provides a representative sample of the influent or mid process locations at wastewater treatment plants.

20.2. The sampling set up is shown as follows:



20.3. Ensure that all equipment to be used is properly disinfected.

20.3.1. Disinfection is performed by adding 1/3 cup of bleach to enough tap water to achieve the full volume of the one-gallon HDPE container and capping.

20.3.2. The solution must remain in the bottle for at least ten minutes and be swirled to contact all surfaces.

20.3.3. Following emptying, the HDPE bottle is then subjected to at least a four-volume full rinse with tap water and stored in a clean, dry location until use.

20.3.4. The cap-and-tube assembly is submersed in a bleach solution (1/3 cup of bleach per gallon) for a minimum of ten minutes followed by a rinse with tap water.

20.3.5. Pump should be run in a bleach solution for one minute.

20.3.6. Devices should be stored in a clean dry location until use.

20.4. Ensure Proper Sample Collection

20.4.1. Submerse pump with attached 3/8" tubing into the liquid to be sampled.

20.4.2. Run pump for 15 seconds, discarding discharge to area not impacting sampling location (could be discharge container).

20.4.3. Carefully uncap one-gallon HDPE bottle and fill to desired volume (at least 0.75 gallons).

20.4.4. Cap bottle and place immediately on ice and out of direct sunlight.

20.4.5. Label sample and record on chain of custody.

20.4.6. Complete field sampling log and field parameter measurement according to *MASSTC-SOP-016 – YSI ProDSS Field Meter*.

20.4.7. Sample other chemical biological samples as prescribed.

20.4.8. Deliver to laboratory within two hours of collection.

21. Data Management/Records Management

21.1. Aliquot temperature and sample volume should be recorded in the aliquot log book.

21.2. Ensure that the time of sample and initials of sampler are recorded promptly, legibly, and in indelible ink on the chain of custody.

21.3. Record all observations and data according to *MASSTC-SOP-003 – Data and Records Management*.

22. Quality Control

22.1. Always consult *MASSTC-FRM-040 – Sampling Plan*.

23. Internal and External References

23.1. MASSTC-SOP-003 – Data and Records Management.

23.2. MASSTC-SOP-014 – Ozone Measurement.

23.3. MASSTC-SOP-015 – Sample Preparation and Transportation.

23.4. MASSTC-SOP-016 – YSI ProDSS Field Meter.

23.5. MASSTC-SOP-017 – Sample Equipment Maintenance and Sterilization

23.6. Standard Methods (2017) Standard Methods for the Examination of Water and Wastewater, 23rd Edition American Water Works Association (AWWA, WEF and APHA)

24. Forms and Data Sheets

24.1. MASSTC-FRM-020 – Daily DC West Sampler Logs

24.2. MASSTC-FRM-040 – Sampling Plan

APPENDIX 6:
SAMPLING PLAN

Massachusetts Alternative Septic System Test Center

Barnstable, Massachusetts

Form

Title: Sampling Plan

Effective Date: 2021-03-02	**Number:** MASSTC-FRM-040	**Revision:** 000

Authors

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Sampling Plan

Document ID#: MASSTC-FORM-040
Revision#: 000
Released: 2021-03-02
Released By: Brian Baumgaertel

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site

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SAMPLING PLAN

Project : Date: End Date: Initials:

Location:

Parameters	S	M	T	W	T	F	S	Comments

Location:

Parameters	S	M	T	W	T	F	S	Comments

Parameters	S	M	T	W	T	F	S	Comments

Rush information:
Influent sample information:
Other notes:

Document ID#: MASSTC-FORM-040
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Released By: Brian Baumgaertel

OVERHEAD SKETCH

Instructions: Sketch *all* above-ground components. Labels should match the location names in the sampling plan. Include an arrow indicating the direction of the channel. Additional reference points are preferred.

Project: Date of Sketch: Staff Initials:

DISCHARGE

HEADWORKS

APPENDIX 7:
BARNSTABLE COUNTY DEPARTMENT OF HEALTH AND ENVIRONMENT LABORATORY QULIATY ADDURANCE PLAN

Quality Assurance Plan

(Revision 026)
Revised
29 December 2020

County of Barnstable
Barnstable County Department of Health and the Environment
Water Quality Laboratory
Superior Court House
Route 6A
P.O. Box 427
Barnstable, MA 02630

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1. Introduction

This Quality Assurance (QA) manual has been developed to describe the overall quality assurance program employed by the Barnstable County Department of Health and the Environment Water Quality Laboratory. The County Laboratory performs environmental analyses of volatile organic compounds, metals, and wet chemistry parameters using *Methods and Guidance for the Analysis of Water* (Version 2, June 1999)[1] approved by the United States Environmental Protection Agency, and *Standard Methods for the Examination of Water and Wastewater* (22th Edition, 2012)[2] approved by American Public Health Association, American Water Works Association and Water Environment Federation. The County Lab also performs analyses of total coliform, fecal coliform, e. coli, and heterotrophic plate count in potable and/or non- potable water using the Standard Method[2]. The County laboratory has also carried out its testing and calibration activities in such a way as to meet the requirements of the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC) 17025:2005 [3]. The Laboratory specializes in analysis of drinking water and groundwater whose continuous testing and quality maintenance are of great interest to the residents of the County.

Annually, all laboratory employees must complete a conflict of interest law and ethics online training program.
Access to the training is through the Commonwealth's Ethics Website https://www.mass.gov/online-conflict-of-interest-law-training. The Human Resources Department of Barnstable County requires all departments, including the Barnstable County Water Quality Laboratory, to submit and retain the records regarding the completion of this and other employee trainings relating to workplace policies.
In addition, every year all state, county and municipal employees must be provided with the summary of the conflict of interest law. All public employees are required to sign a written acknowledgment that they have been provided with the summary and records of this receipt are kept with the Department of Health and Environment administrative office.

On an annual basis, all personnel participate in the laboratory ethics training program. The objective of the program is to assess and maintain the quality, accuracy, and precision of the generated data and to provide a permanent record of instrument performance and overall data quality and reliability by implementing well defined QA / Quality Control (QC) procedures. The materials covered in our program addresses the proper procedures to ensure data integrity, recognition and prevention of improper laboratory practices, the promotion of objectivity and impartiality in the generation and reporting of analytical data, and procedures for confidential reporting of data integrity concerns to the laboratory director. The program describes our quality assurance organization and responsibilities, our quality assurance objectives for precision and accuracy, the format for generating our standard operating procedures, and the procedure for maintaining our records. The laboratory documents the content of the training and date of participation for each staff member. This documentation is kept available for review during an inspection.

2. Quality Assurance Policy and Quality Assurance Objectives

2.1 Statement of Policy

It is the policy of the Barnstable County Health Laboratory to provide the possible highest quality analysis of drinking water. We are committed to maintaining a strict QA program and adhering to all policies required by regulatory and accrediting agencies and other organizations[3]. To achieve this high standard of quality, we have implemented the following quality assurance plan for our analytical services.

Our laboratory employs modern analytical instrumentation and is fully automated to provide results of high quality in a timely fashion. The analytical professionals and technicians are well trained with many years of work experience in the respective area of analysis. Therefore, this program assures that the results provided by the County Laboratory are as accurate as possible and highly reliable.

2.2 Quality Assurance Objectives

In order to ensure the production of high quality data and clients' full satisfaction, Barnstable County Laboratory has established the following Quality Assurance Objectives:

- Full compliance with certification requirements;
- Full compliance with regulatory agencies;
- Full compliance with contract requirements;
- Full compliance with published methodologies;
- All personnel concerned with analysis and calibration activities in the Laboratory are required to familiarize themselves with the quality documentation and implement the policy as stated above.

2.3 Subcontracted Laboratories

Barnstable County Laboratory also subcontracts some analyses to other laboratories. Barnstable County Laboratory makes sure these subcontracted laboratories also meet Quality Assurance Policy and Quality Assurance Objectives as stated above. Especially Barnstable County Laboratory makes sure that the subcontracted laboratories must comply with ISO/IEC 17025:2005[3] when the samples for National Sanitation Foundation internal projects are analyzed.

3. Accommodation and Environmental Conditions

The Laboratory is in a newly renovated building. Major renovations have been completed at the former County Jail gymnasium which now has two floors: the first floor serves as the County's Water Quality Laboratory and the second floor is for the Human Services Department.

3.1 Laboratory Layout:

Water Quality Laboratory includes the following sections:

- Reception and Sample Receiving;
- Microbiology;
- Organic Analysis;
- Inorganic Analysis;
- Inorganic Instrumentation;
- Wet Chemistry;
- Data Storage and Reporting;
- Offices

Laboratory map is attached for reference.

3.2 Emergency Eyewashes and Showers

There are two emergency showers and three emergency eyewashes in the laboratory. All laboratory staff well know where they are all located and how to use them. Eyewashes are flushed weekly by laboratory staff to ensure they are operating correctly. Safety showers are tested weekly by laboratory staff too. Weekly check for emergency eyewashes and showers are recorded in a log book.

3.3 Fire Safety Equipment

There are six different sizes of ammonia phosphate base dry chemical fire extinguishers in the laboratory. Four of them is in four rooms respectively, and two in the hall.

4.0 Responsibilities and Authorities

Barnstable County does have its Personnel Policies and Procedures[5] for all County employees to follow. Two of major purposes of the Personnel Policies and Procedures are:

- To ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work;
- To avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity.

Barnstable County laboratory employees must follow not only the County's Personnel Policies and Procedures, but also the requirements of the Laboratory's quality assurance and quality control program. The quality assurance and quality control program requires an effective chain of command within the laboratory. The maintenance of this requirement is the responsibility of the laboratory analysts, office staff and the Laboratory Director.

4.1 Laboratory Director

The Laboratory Director supervises all analysts, technicians, and laboratory administrative personnel. Additional responsibilities include supporting the implementation of the quality assurance plan within the laboratory, maintaining and enforcing standard operating procedures, and maintaining good laboratory practices. The Director may substitute for any analyst when questions arise and performs any analysis as required.

The Laboratory Director also acts as the Quality Assurance Officer of the laboratory. All data are finally reviewed by the Laboratory Director prior to release. Data that fall outside of quality control limits can be accepted if in the judgement of the Director there are suitable technical reasons for these to be accepted. However, these cases are well documented and the reasons for acceptance are fully explained.

If the Laboratory Director is not around for a few days, the organic chemist will conduct final review and signing of reports. All other issues will go to Department Director. If there is still anything else that must need the Laboratory Director, the Laboratory Director will be contacted immediately, and the Laboratory Director will respond as soon as possible to make sure that the laboratory is operated smoothly.

4.1.1 Internal Audits:

One of the major responsibilities of the Laboratory Director is to conduct the laboratory audit once a year. The main purpose of the internal audit is to verify that the laboratory operations continue to comply with the requirements of the management system, quality assurance policy and quality assurance objectives. The internal audit covers sampling, sample log-in, sample analysis and data reporting, etc. The checklist of the Laboratory internal audits is attached (Attachment 1).

4.1.1.1 If there is any finding from the internal audit which affect any associated data, the following measures must be taken:

- The root cause of the finding must be found out and recorded in Lab Corrective Action Log Book;
- Any measures to be taken to prevent happening again must be recorded in Lab Corrective Action Log Book;
- All data affected by this finding will be flagged, and the lab reports will be revised with a lab narrative;
- The customers will be notified in writing with the revised Lab Reports.

4.1.2 Management Reviews

Laboratory Director also work with Department Director together to conduct a review of the laboratory's management system and testing and/or calibration activities to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. The review will take account of the following areas:

- The suitability of policies and procedures;
- Reports from managerial and supervisory personnel;
- The outcome of recent internal audits;
- Corrective and preventive actions;
- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;
- Changes in the volume and type of the work;
- Customer feedback;
- Customer complaints;
- Recommendations for improvements;
- Other relevant factors, such as quality control activities, resources and staff training.

A period for conducting a management review is once a year.

4.2 Laboratory Analysts

All analysts conduct sample analysis and maintain quality assurance by following the laboratories quality assurance plan. This is achieved by a thorough knowledge of the appropriate standard operating procedure of each method employed by the analyst. Especially analysts must closely track the holding times of all analyses, and it is analysts' primary responsibility to make sure that all analyses are done within their holding times. Additional responsibilities include complete and accurate work records, immediate notification of quality control problems, and the authority to accept or reject data based on defined quality control acceptance criteria.

4.2.1 The bacteriologist is the primary microbiological resource for the Laboratory. The primary duties of the bacteriologist include performing or supervising all bacteriological analyses, all media and buffer preparation, and all QA/QC record keeping for the microbiology laboratory. The bacteriologist also performs and oversees sample container preparation, preservation, sterilization, and distribution. The bacteriologist also generates the final reports. If there is any positive identification of total coliform in drinking water, the bacteriologist will inform customers right away.

4.2.2 The primary duties of the Inorganic Chemist and Inorganic Analyst are performing all metal analyses by flame or graphite furnace AA and Inductively Coupled Plasma Mass Spectrometer (ICP-MS), all inorganic anion analyses by ion chromatography, and all wet chemistry analyses by closely following Standard Operating Procedures (SOP). The Inorganic Chemist and Inorganic Analyst also generate final data reports and conduct data reviews. Secondary duties include supervision of sample container preparation, preservation, and distribution.

4.2.3 The Organic Chemist is the primary resource for questions regarding organic methodology and analyses. The primary duties of the organic chemist are to analyze volatile organic compounds using Gas Chromagraph – Mass Spectrometer (GC/MS) and Total Organic Carbon (TOC) Analyzer. The organic chemist is responsible for maintaining proper documentation for any modified methods and SOP's. The Organic Chemist also generates data reports and conducts data reviews. Secondary duties include performing sample container preparation, preservation, and distribution.

4.2.4 Analytical training is required for all analysts, and training includes new analyst training and cross training. When a new analyst is hired, a systematic training will be conducted by an experienced analyst and/or laboratory Director. New analyst will complete an Initial Demonstration of Capability if the pertinent analytical method requires it. All analysts are required and encouraged to be cross trained for each other to ensure the analysis is done properly when its primary analyst is absent for a period of time. All training processes will be recorded in Lab Staff Training Log Book (Attachment 2).

4.2.5 All analysts need to enter their own sample data to the Laboratory Information Management System (LIMS), and also cross review data entry to ensure data information are correct in sample received date and time, customer ID, lab ID, analytical date and time, units, and analytical method.

4.3 Information Specialist/Billing Clerk

Information specialist/billing clerk has the following three kinds of duties:

- Maintains customer accounts receivable records and processes payments as received.
- Prepare invoices as scheduled and as needed basis.
- Develop and maintain database applications as needed and directed.

4.4 Administrative Staff

The administrative staff is primarily responsible for sample log-in, inquiries of sample status, final report generation, taking and filling bottle order requests, keeping track of inventory and ordering supplies, and typing and calling in purchase order. Some of the staff's secondary responsibilities include sample container preparation and sample pick-up.

4.5 Laboratory Personnel

Name	Initial	Title
Dan White	DW	Laboratory Director/Chemist
Ryan Lucier (Grady)	RG	Microbiologist
Bethany Traverse (part-time)	BT	Microbiologist
Yuankun "Ken" Ni (retired)	YN	Chemist
Chris Long	CL	Chemist
Liping Xun	LX	Chemist
Andrew Barker	AB	Chemist
Leonard Pitts (part-time)	LP	Chemist
Lacey Adams Prior (part-time)	LAP	Chemist
Vacant – TBD (part-time)		Analyst
Katarina Soldatov	KS	Laboratory Assistant
Patricia Palmer (part-time)	PP	Laboratory Assistant
Veronica Tavares (part-time)	VT	Laboratory Assistant

5. Quality Assurance for Precision and Accuracy

Quality assurance is used to establish and maintain confidence in the precision and accuracy of the data generated by the Laboratory. The routine procedures utilized in assessing precision and accuracy are based on established SOPs.

5.1 All data are recorded in the pertinent logbooks, charts, and Laboratory Information Management System (LIMS). These records are periodically boxed and stored for easy retrieval. LIMS data are archived yearly. All the data are maintained for a period of ten years.

5.2 Forms used in reporting results are generated by the LIMS and are designed to convey all pertinent information to the client. Report forms include date of collection, date of receipt by the laboratory, date of analysis, client name, client I.D. name or number, and laboratory sample I.D. number. All analyses required by the

Massachusetts Department of Environmental Protection (DEP), Division of Water Supply (DWS) are reported on forms supplied by DWS. Included in any report, if requested by the client, are the recoveries of all QC samples and matrix spikes.

5.3 If any analyte exceeds its MCL, the local Health Agent is informed by the report and the client is telephoned as soon as possible and advised of the results. All recommended limits exceeded are explained on the report form sent to the client along with the recommended limit. If any MCL is exceeded for an analysis required by a State or Federal Agency, the client is contacted immediately (24 hours) and advised of the result. The client has the primary responsibility to contact the regulatory agency involved; however, the Laboratory will make the contact within 48 hours if based on follow up conversations with the client, there is no indication of the results being reported.

5.4 A few different methods for internal quality control checks are used. The internal controls include daily instrument blanks, daily method standards, matrix spikes, duplicates, and annual Proficiency Testing (PT) samples analyses, and PT samples are ordered from an accredited PT vendor meeting the criteria of the current policies made by Massachusetts Department of Environmental protection, Laboratory Certification office. Please check the following website for the detailed information: http://www.mass.gov/dep/bspt/wes /wespubs.htm.

5.5 Calibration curves must consist of at least three points and are used to calculate analyte concentration. A separate calibration curve is generated for each analyte included in the analytical method. Instrument calibration is performed each day samples are analyzed. The instrument may be completely calibrated with at least three calibration standards or a single calibration standard may be used to check an existing calibration curve. Again, all analytes of interest are included in the calibration.

5.6 Method blanks are analyzed daily and represent all sample preparation procedures excluding any target analytes. The method blank contains all appropriate surrogate and internal standards, diluents, and modifiers.

5.7 Laboratory fortified blanks (LFB) or Laboratory Control samples (LCS) are analyzed to monitor accuracy of the method. The LFB is a spiked method blank sample containing all the analytes of interest. The recoveries of the analytes are charted and used as a diagnostic tool to monitor system performance. When these recoveries approach either the upper or lower limits established by the method, corrective action is taken before the system fails to meet calibration criteria.

5.8 Quality Control (QC) check samples or Continuing Calibration Verification (CCV) are analyzed to monitor the accuracy of the method. QC check sample is from an alternative source containing all the analytes of interest. The recoveries of the analytes are charted and used as a diagnostic tool to monitor system performance.

When these recoveries approach either the upper or lower limits established by the method, corrective action is taken before the system fails to meet calibration criteria.

5.9 Duplicate analyses are performed on each batch of samples analyzed. The frequency is generally 10 % of all samples in the batch, but at least one sample if less than 10 samples are analyzed. The duplicate samples are prepared and analyzed using the same procedures as the original sample. The recovery of the duplicate analysis is used to monitor the reproducibility of the entire procedure.

5.10 Matrix spikes are performed to account for any matrix effects in an environmental sample. The frequency of matrix spike analysis is 10 % of a sample batch. Duplicate matrix spike analyses are used to monitor reproducibility.

5.11 Quality Control for Purchasing Reagents, Standards and any Other Supplies:

5.11.1 The Laboratory Director must make sure all reagents, standards, containers and any other supplies used in each analytical method meet their minimum Quality Control requirements described in the Method. All the names of vendors, Catalog numbers must be included in the Standard Operating Procedure of the Analytical Method.

5.11.2 All purchasing must be approved by the Laboratory Director to make sure the right items such as reagents, standards, containers, etc are ordered.

5.11.3 Once the ordered items are received, lab assistants and analysts will double check if the received items are right or any damages occurred during transportation. If there is any item received which has been damaged or wrong item has been shipped, the laboratory will contact the vendors right away, and the Laboratory Director will be notified too.

5.11.4 Analysts must log in all received reagents and standards into the laboratory Primary Standard Logbook. The Certificate of Analyte is stored in a three-hole binder.

5.11.5 Bottle sterility check for microbiology laboratory: The laboratory checks at least one bottle per lot of commercially prepared sample containers for sterility by adding approximately 25 ml of sterile non-selective broth to each bottle. The bottle is capped and rotated so that the broth comes in contact with all surfaces and is incubated at 35±0.5ºC and checked for growth at 24 and 48 hours and the results are recorded.

5.12 Precision Criterion of Duplicate Analysis:

In order to determine the acceptability of duplicate analysis of Fecal Coliform, E.coli, Enterococci, and Hetertrophic Plate Count (HPC), their Precision Criterion of Duplicates analysis are obtained by calculating the range of logs of most recent 15 samples and their corresponding duplicates as follows:

5.12.1 Collect the most recent 15 sets of the results of the original samples and their duplicates.

5.12.2 Calculate the Logarithms of each set of the results, and record them as *L1* and *L2*. If any result is <1, add 1 to both values before calculating the Logarithms.

5.12.3 Range of Logarithms ($R_{\log}$) is calculated using the following equation:

$$R_{\log} = |L1 - L2|$$

5.12.4 The mean ($\overline{R}$) of $R_{\log}$ is calculated as follows:

$$\overline{R} = \frac{\sum R_{\log}}{n}$$

$\sum R_{\log}$ = the sum of the range of Logs;
n = the number of sets of duplicates.

5.12.5 Precision Criterion is calculated as follows:

$$\text{Pr}\,ecision Criterion = 3.27 \times \overline{R}$$

5.12.6 If any Range of Logarithms is greater than the precision Criterion, there is a greater than 99% probability that the analysis has exceeded variability limits. For any samples that fall outside the acceptable limits, the acceptability of the imprecision will be determined. If the data are not acceptable, all results since the last precision check must be rejected. The analytical problems will be determined and corrective actions will be taken to resolve problem.

Corrective action based on internal quality control samples or external samples such as performance evaluation sample is used to maintain precision and accuracy of the analytical results. The process for corrective action includes a review of the history of the problem by checking standard preparation logs, instrument maintenance logs, and QC charts. Based on the historical information the cause of the problem is narrowed. The next steps are to change a defective part, clean a dirty part, or if necessary call a service engineer for advice or a visit.

6. Control Charts[2]

Two types of control charts are used in the County Laboratory: (1) accuracy (or means) chart; and (2) precision (or range) chart.

6.1 Accuracy Chart:

The accuracy chart for QC samples is constructed from the average and standard deviation of a specified number of measurements of the analyte of interest. The accuracy chart includes upper and lower warning levels (WL) and upper and lower control levels (CL). ±2s and ±3s are used for the WL and CL, respectively, where s represents standard deviation. These values are derived from stated or measured values for reference materials. The number of measurements, n or n-1, used to determine the standard deviation, s, is specified relative to statistical confidence limits of 95% for WLs and 99% for CLs. The County Laboratory is using the Accuracy Chart for Laboratory Control Sample (LCS)/Laboratory Fortified Blank (LFB), Matrix Spike (MS), and sample surrogate recovery. A chart is constructed for each analytical method. The results are entered on the chart each time the QC sample is analyzed. Everything is done in EXCEL Spreadsheet.

6.2 Precision Chart:

The precision chart also is constructed from the average and standard deviation of a specified number of measurements of analyte of interest. Precision chart is used for percent differences of LCS/LCSD, MS/MSD, and sample and sample duplicate. Perfect agreement between replicates or duplicates results in a difference of zero when the values are subtracted, so the baseline on the chart is zero. Therefore, for precision charts, only upper warning limits and upper control limits are meaningful. A chart is constructed for each analytical method. The results are entered on the chart each time the QC sample is analyzed. Everything is done in EXCEL Spreadsheet.

6.3 Updating of Control Charts:

If measurements never or rarely exceed the WL, recalculate the WL and CL using the 20 to 30 most recent data points. Trends in precision can be detected sooner if running averages of 10 to 20 are kept. Trends indicate systematic error; random error is revealed when measurements randomly exceed warning or control limits.

6.4 Application:

- Control Limit – If one measurement exceeds a CL, repeat the analysis immediately. If the repeat measurement is within the CL, continue analyses; if it exceeds the CL, discontinue analyses and correct the problem.
- Warning Limit – If two out of three successive points exceed a WL, analyze another sample. If the next point is within the WL, continue analyses; if the next point exceeds the WL, evaluate potential bias and correct the problem.
- Trending – If seven successive samples are on the same side of the central line, discontinue analyses and correct the problem.
- Barnstable County Laboratory has calculated our own in-house control limits and warning limits for drinking water and wastewater. Each analyst closely reviews his or her in-house control limits for each analysis based on the procedures stated as above. The laboratory director must review control

charts monthly.

7. Lab Corrective Actions

Quality control data outside the acceptance limits or exhibiting a trend are evidence of unacceptable error in the analytical process. The County laboratory takes corrective action promptly to determine and eliminate the source of the error. The laboratory does not report data until the cause of the problem is identified and either corrected or qualified. The customers will also be notified.

7.1 The County Laboratory records any problems and issues that occur and affect data integrity, lab safety and lab operation. If there are any unusual things happened in the Lab, the Lab Director is informed right away, and the Originator must describe the detailed information on these problems and issues in the Laboratory Corrective Action Log Book (See Attachment 3). The Originator needs to address the following three things:

(1) Describe what happened;
(2) Describe how and why they happened;
(3) What actions should be taken to prevent or eliminate them happening again?

7.2 The following three categories of problems and issues can be written down in the lab Corrective Action Log Book:

(1) Improper Lab Practices:
Definition: A scientifically unsound or technically unjustified omission, manipulation, or alteration of procedures or data that bypasses the required QC parameters, making the results appear acceptable.

Any alteration of data such that the data are unauthentic or untrue representations of the experiment or test performed.

Peak integrations are not done properly;
Quality Control Samples do not meet criteria;
Initial Calibrations do not meet criteria;
Sample holding times are out;
Method Blank is contaminated;
Loss of sample;
Equipment malfunction;

(2) Standard Operating Procedures (SOP) Modifications:
Incorporate new equipment into the SOP;
Correct the wrong or inappropriate procedures in the SOP;
Etc.

(3) Any other actions affecting data quality and lab operations:

7.3 Corrective Actions:

Corrective actions begin with the analyst, who is responsible for knowing when the analytical process is out of control. The analyst must initiate corrective action when a QC check exceeds the acceptance limits or exhibits trending and must report an out of control event to the supervisor. The corrective actions to be used when QC data are unacceptable are as follows:

- Check data for calculation or transcription error. Correct results if error occurred.
- Check to see if sample(s) was prepared and analyzed according to the approved method and SOP. If it was not, prepare and/or analyze again.
- Check calibration standards against an independent standard or reference material. If calibration standards fail, reprepare calibration standards and/or recalibrate instrument and reanalyze affected sample(s).
- If a LFB fails, reanalyze another laboratory-fortified blank.
- If a second LFB fails, check an independent reference material. If the second source is acceptable, reprepare and reanalyze affected sample(s).
- If a LFM fails, check LFB. If the LFB is acceptable, qualify the data for the LFM sample (Table 1 lists the data qualifiers) or use another method or the method of standard addition.
- If a LFM and the associated LFB fail, reprepare and reanalyze affected samples.
- If reagent blank fails, analyze another reagent blank.
- If second reagent blank fails, reprepare and reanalyze affected sample(s).
- If the surrogate or internal standard known addition fails and there are no calculation or reporting errors, reprepare and reanalyze affected sample(s).

7.4 Customer Complaints:

Barnstable County laboratory is totally customer-focused organization, and it has been understood that customer complaints represent valuable information about recurrent problems. Laboratory secretary and assistant are always front-line staff to handle customer complaints, and they must give customers their full and undivided attention. Laboratory secretary and assistant must resolve complaints promptly if they are able to. Laboratory secretary and assistant must report any customer complaints to laboratory director. If the customer complaints can not be resolved right away, laboratory secretary and/or lab assistant must report to laboratory director right away. The laboratory director must talk to the customers to find out the root causes for complaints if necessary. Laboratory director must take actions to have any mistakes corrected properly and promptly, and call back to customers to explain the final resolutions for the complaints. All these processes must be recorded in the Laboratory Customer Complaint Logbook (Attachment 4).

If there are any customers of Barnstable County Test Center to complaint to the Test

Center Staff about laboratory testing, the Test Center Staff will pass the complaints to the laboratory. Barnstable County Test Center has its own Customer Complaint Logbook. All the resolutions of the Customer Complaints for the Test Center must be reviewed by Department Director.

7.5 Notification of Customers

The Laboratory will notify the customers regarding any issues which trigger the laboratory corrective action, and which affect their sample and data integrity and quality. The final Laboratory Corrective Actions will need to be submitted to the customers for their approval.

8. Analytical Methods

The analytical methods performed by the Barnstable County Laboratory have come from two sources[1, 2]. The first one is *Methods and Guidance for the Analysis of Water* (Version 2) by EPA, and the second one is *Standard Methods for the Examination of Water and Wastewater*, 22th Edition, 2012 by American Public Health Association, American Water Works Association and Water Environment Federation. Barnstable County Laboratory has its own modified standard operation procedures (SOP) based on the methods stated above. These SOP including any modifications are approved and certified by the Laboratory Certification Office (LCO) of Massachusetts Department of Environmental Protection. All non-approved methods used are for informational purposes only and are documented as such. The Massachusetts Laboratory Certification Identification Number of Barnstable County Laboratory is M-MA009.

8.1 A list of certified methodologies used for specific analysis is noted below:

Parameters	**Methodologies [1, 2]**	
Bacteria	**Potable**	**Non-Potable**
Total Coliform	MF-SM9222B	
Total Coliform	ENZ.SUB.SM9223	
Total Coliform	EPA 1604	
Total Coliform	SM 9223B-COLILERT	
Fecal Coliform		SM9223B-COLILERT-18
Fecal Coliform	MF-SM9222D	MF-SM9222D
E. Coli	NA-MUG-SM9222G	EPA 1604
E. Coli	EPA 1604	EPA 1603
E. Coli	ENZ.SUB.SM9223	
E. Coli	SM 9223B-COLILERT	SM 9223B-COLILERT
Heterotrophic Plate Count	SM9215B	
Enterococci	EPA 1600	EPA 1600
Enterococci	ENTEROLERT	ENTEROLERT
Metals	**Potable**	**Non-Potable**

Aluminum	EPA 200.8	EPA 200.8
Antimony	EPA 200.8	EPA 200.8
Arsenic	EPA 200.8	EPA 200.8
Barium	EPA 200.8	
Beryllium	EPA 200.8	EPA 200.8
Cadmium	EPA 200.8	EPA 200.8
Chromium	EPA 200.8	EPA 200.8
Calcium	SM3111B	SM3111B
Cobalt		EPA 200.8
Copper	EPA 200.8, SM 3111B	EPA 200.8, SM 3111B
Iron		SM 3111B
Lead	EPA 200.8	EPA 200.8
Magnesium		SM 3111B
Manganese	EPA 200.8	EPA 200.8, SM 3111B
Mercury	EPA 200.8	
Nickel	EPA 200.8	EPA 200.8
Potassium		SM 3111B
Selenium	EPA 200.8	EPA 200.8
Sodium	SM 3111B	SM 3111B
Thallium	EPA 200.8	EPA 200.8
Vanadium		EPA 200.8
Zinc		EPA 200.8
Inorganics		
Total Alkalinity	SM 2320B	SM 2320B
Specific Conductivity		120.1, SM2510B
Chemical Oxygen Demand		HACH 8000
Total Organic Carbon		SM 5310B
Chloride		EPA 300.0
Fluoride	EPA 300.0	
Nitrate-N	EPA 300.0	EPA 300.0
Nitrite-N	EPA 300.0	
Sulfate	EPA 300.0	EPA 300.0
Ammonia-N		EPA 350.1
Kjeldhal-N		EPA 351.2
pH	SM 4500-H-B	SM 4500-H-B
Non-Filterable Residue (TSS)		SM 2540D
Total Dissolved Solids	SM 2540C	SM 2540C
Turbidity	EPA 180.1	
Total Hardness ($CaCO_3$)		SM 2340B
Perchlorate	EPA314.0	
Organics		
Volatile Organic Compounds	EPA 524.2	
Volatile Halocarbons		EPA 624.1
Volatile Aromatics		EPA 624.1

Trihalomethanes EPA 524.2

8.2 In order to clearly distinguish in the analytical reports between those analyses for which it holds Massachusetts Department of Environment Protection Certification and those for which it does not hold Massachusetts Department of Environmental Protection Certification, Barnstable County Laboratory has started attaching a summary of the laboratory certifications as stated in Section 8.1 to each of analytical reports to all customers.

8.3 Notification of Customers for Any Changes of Analytical Methods:
After Barnstable County Laboratory receives samples from customers, Barnstable County Laboratory realizes certain analytical methods must be changed or modified to proceed with the analyses. The Laboratory will notify and discuss with the customers first for the changes of the methods before conducting analyses.

9. Sample Management

9.1 The generation of quality data begins with the collection of the sample. The integrity of the sample collection is therefore of importance to the laboratory. Samples must be collected in such a way so as not to disrupt the integrity of the sample by the introduction of foreign material or the release of any material of interest. The laboratory maintains sample integrity by supplying the appropriate sample containers, ensuring that the sample containers are properly cleaned and contain the appropriate preservative, enforcing sample holding times to allow adequate time for analysis, and ensuring that adequate volumes of the sample are collected.

9.2 Upon receipt of a batch of samples, the Sample Reception Office examines the samples for breakage or damage while checking the accompanying documents for conformance with sampling procedure. The Sample Receiving Person also insures that the type of preservative is noted. Then the samples are logged into the LIMS by the Sample Receiving Person, and a unique laboratory ID is assigned to each sample. The Sample Receiving Person must ensure all information is entered into the LIMS correctly. All information and documentation are relayed to the analyst for his\her review and if any further sample manipulation is required, it is performed in a timely fashion.

9.3 All chain-of-custody samples received by the laboratory are examined for breakage or damage and sample integrity. Once the chain-of-custody form has been reviewed for clarity and accuracy, it is signed and the samples are received into the laboratory. After receipt, the samples are logged into the sample log book by the Sample Receiving Person, given a laboratory identification number, and stored in a secured area. The internal report form follows the sample through the laboratory until all analyses are complete. At the end of each day when the sample was being analyzed, it is returned to the secured area until all analyzes have been completed.

9.4 If the samples are not properly collected, preserved and handled, they will not be accepted by the laboratory. The following are the Laboratory's policies for the sample rejection:

- The samples are stored in wrong containers such as non-sterile bottles for total coliform;
- The samples are out of the holding times;
- There is no clear identification of the sample matrix;
- There are Bubbles in VOC vials;
- There are no enough sample volumes such as less than 100 ml for Total Coliform analysis;
- The samples are preserved with wrong preservatives;
- Perchlorate samples are not filtered with sterile filters and syringes;
- The samples are not kept in coolers when they are received.

NOTE: When the samples are rejected, the samples receiving officers will make notes on the chain of custody for any rejection reasons.

9.4.1 Notification of Customers

- If the customer is dropping off the samples and the customer's samples are not accepted, the Laboratory assistants shall clearly, respectfully and professionally explain to the customer why the samples should not be accepted, and what the customer could do better to make the laboratory accept its samples next time.
- If Once the samples are rejected for acceptance and the Laboratory assistants could not talk to the customer face-to-face, the Laboratory will notify the customer either by phone or by e-mail right away.

9.5 Subtracting of Samples for Analyses

9.5.1 Prior to subtracting out samples to other labs for analyses, Barnstable County Laboratory must notify its customers by e-mail, discuss with its customers and obtain preapproval from its customers. All these processes must be documented too.

9.5.2 There are the following circumstances under which the received samples need to be subcontracted to another certified laboratory:

- There are too many samples, and the number of the samples received exceeds the capacity of the laboratory.
- The instruments break down, and they cannot be fixed right away. Then the samples may need to be subcontracted out.
- The samples received contain the uncertified parameters, and they will be

subcontracted out.

- The samples requiring total metals will be subcontracted out for the time being.

9.5.3 If there are any contaminants detected which exceed their Maximum Contaminant Levels, Maximum Residual Disinfectant Level or reportable concentration, the subcontracted laboratories are required to notify Barnstable County Laboratory within 24 hours of obtaining valid data. When it prepares the chain of custody to the subcontracted laboratory, Barnstable County Laboratory will stamp on it with "NOTIFY FOR ANY MCL EXCEEDANCES".

9.5.4 If the final analytical reports need to be submitted to Massachusetts Department of Environmental Protection, Barnstable County Laboratory will stamp on the chain of custody to the subcontracted laboratory with "STATE FORM". The subcontracted laboratory will have to report the data with MA DEP required format.

10. Laboratory Equipments and their Calibration Procedures

Calibration of the laboratory's equipment is performed on a regular basis. The calibration is performed in accordance with the manufacturer's instruction or in accordance with the calibration procedures outlined in the appropriate analytical methodology and, as needed.

Each piece of equipment requires preventive maintenance to ensure optimal performance of the instrument. The preventive maintenance schedule is supported by vendor service maintenance contracts and an inventory of spare parts. All major instruments have separate service contracts that include a yearly preventive maintenance visit. The minor instruments and equipment are covered by a general laboratory preventive maintenance contract where a service representative will annually check and calibrate all ovens, incubators, thermometers, refrigerators, autoclaves, UV-VIS spectrophotometers, and fume hoods.

10.1 Flame Atomic Absorption - This instrument is calibrated daily if samples are analyzed with five calibration standards prepared fresh. The calibration is checked with a QC check sample of an alternative source and a LFB sample. Both of these analyses occur before any samples are analyzed and at the end of the analysis. Also, interferences are analyzed for with a method blank.

10.2 Total Organic Carbon Analyzer (TOC-V $_{CPH/CPN}$) - This instrument is calibrated daily with a six point calibration curve and checked for interferences with a method blank. The calibration is checked with QC and LFB samples at a frequency of 10% of the sample load.

10.3 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS):

There are the following few tuning steps must be done before the ICP-MS is calibrated for analysis:

- Tuning for sensitivity daily by using a 2 wt% nitric acid solution containing 10 ng/ml of each of the elements: Li, Co, Y, Ce, and Tl. Sensitivity after tuning must meet criteria set by ICP-MS manufacturer;
- Tuning for detector daily by setting P/A factor of each target element;
- Checking of mass calibration, mass resolution and instrument stability.

Initial calibration is composed of six different levels of standards, and initial calibration verification standards are run following the initial calibration to verify the accuracy of the initial calibration. Please refer to the SOP for EPA Method 200.8.

10.4 Ion Chromatograph - Each day a five-point calibration curve of the analytes of interest is generated from freshly prepared standards for nitrite-N, o-phosphate, bromide, nitrate-N, sulfate, chloride, and fluoride standards. Each day a one point calibration standard (continuing calibration) is analyzed to check an existing calibration curve or as the daily single point standard. Interferences are checked with reagent water blank. Checks include QC check and LFB samples at a frequency of 20% and matrix spikes to identify any environmental sample matrix effects.

10.5 GC\MS - Each day the mass spectrometer tune settings are checked with 25 ng of p-bromofluorobenzene (method 524.2) or, 50 ng of p-bromofluorobenzene (for method 624). The background subtracted spectra with the highest abundance is compared to and must meet established criteria for analysis to continue. When a successful BFB analysis is achieved, a single point calibration standard (Continuing Calibration) is analyzed to check the validity of the existing initial calibration curve. Whenever the MS is tuned, a new five point calibration curve may need to be generated. A method blank is analyzed daily to check for interferences and matrix spikes are analyzed for matrix effects.

10.6 PC-Titrate – The PC-Titrate is used to analyze pH, conductance, and alkalinity in aqueous samples:

- PH is calibrated daily using standard solutions of pH = 4.0, pH = 7.0, and pH = 10. A beginning and an end QCs (pH = 7.0) are run every ten samples.
- Alkalinity measurement is based on the pH calibration. A beginning and an end QCs (Alk = 25 mg/L) are run every ten samples.
- Conductance probe is calibrated using seven levels of standards. A beginning and an end QCs (Cond = 100 µmohs/cm) are run every ten samples.

10.7 Flow Injection Analysis (FIA) System (HACH QuikChem 8500 Series)--- The Flow Injection Analysis (FIA) System is equipped with XYZ Autosampler, Data Quality Management (DQM) software and Auto-Dilutor. The FIA is used for analysis of

Total Kjeldahl Nitrogen (TKN), ammonia, and total cyanide in aqueous samples.

- The instrument is calibrated daily per specified requirements of each analytical method.

10.8 Refrigerators - Each day the refrigerator is used, the temperature is recorded and must be within $\pm$ 2°C of the required 4°C. The thermometers used to measure the temperatures are calibrated yearly with a NIST certified thermometer.

10.9 Balances - Each day the balance is used, the calibration of the balance is checked with two class S weights that bracket the expected weight to be measured. The deflection test is performed for the top-loader balances. The analysts make sure the balances are capable of detecting 100 mg at 150 g. The results of the deflection test are recorded daily. The range of weights available is 0.10 mg to 100 g, and they are all NIST traceable. The accuracy of the reference weights are verified annually.

10.10 Incubators (bacteriology) - The temperature for each incubator is recorded twice each day with readings separated by at least four hours. The temperatures must be within $\pm$ 0.2°C for the 44.5°C water bath and $\pm$ 0.5°C for the 35°C incubator and $\pm$ 0.5°C for the 41.5°C incubator. The thermometers used to measure the temperatures are calibrated yearly with a NIST certified thermometer.

10.11 Incubators (BOD, CBOD) - Each day the incubator is used, the temperature is recorded twice per day. The temperatures must be within $\pm$ 0.5°C for incubators to be maintained at 20°C. The thermometers used to measure the temperatures are calibrated yearly with a NIST certified thermometer.

10.12 Ovens - During use the temperature is monitored to ensure that the required temperature is reached and maintained for the appropriate length of time.

10.13 Thermometers - All thermometers are made of glass material and are calibrated yearly with a NIST certified thermometer. Any correction factor to be used in reading the temperature of the thermometer is indicated on the record sheet taped to the refrigerator or incubator where the thermometer is located. The reference thermometers are calibrated annually.

10.14 Reagent Grade Water – The distilled water is produced by using EMD Millipore water purification system. There are three water purification systems from EMD Millipore:

- Two Milli-Qs (Model);
- One Direct-Q (Model)

The quality of the purified reagent water used in microbiology laboratory as stated in the following paragraph must be met[1]:

- The specific conductivity of the reagent water is monitored and recorded

daily, and the values of specific conductivity must be less than 2 μmhos/cm at 25°C.

- The resistivity of the reagent water must be ≥ 16.5 megohm-cm (temperature compensated to 25ºC), and the old cartridges will be replaced if the reading is below 16.5 megohm-cm. The daily resistivity is recorded.
- Residual chlorine level is tested monthly. The amount of the chlorine detected must be less than 0.01 mg/L.
- Heterotrophic plate count (HPC) is performed monthly to check if HPC is less than 500 colony forming units (CFU)/ml. If HPC ≥ 500 colony forming units (CFU)/ml, the distilled water system will be checked and maintained.
- Heavy metal content is tested annually, and no single metal (Cd, Cr, Cu, Ni, Pb and Zn) may be present at ≥0.05 mg/L. Total heavy metals must be less than 0.1mg/L.
- Biosuitability is tested annually, and it must have a ratio of 0.8-3.0.

10.15 Reagents and Other Supplies:

- All inorganic reagents shall be ACS Reagent Grade or equivalent unless the analytical procedure specifies a different grade.
- All organic reagents used to prepare standards shall be of the highest quality obtainable. Organic reagents used to prepare general reagent solutions shall be free of detectable interferences as demonstrated by the analysis of acceptable method blanks.
- All organic solvents shall be free of detectable residue as demonstrated by the analysis of acceptable method blanks. For organic analyses, contamination shall not be restricted to target analytes.
- Supplies such as filter paper, glass wool and boiling beads must be free of contamination as demonstrated by the analysis of acceptable method blanks. For organic analyses, contamination shall not be restricted to target analytes.
- All desiccants must contain moisture indicators.

10.16 Autoclaves: There are three autoclaves in the laboratory:

- Market Forge: Model: STM-E;
- Tuttnauer: Model: 3870M;
- Tuttnauer: Model: 3850E-B/L.

The following parameters are recorded in the lab Autoclave Logbook when any autoclaves are used for sterilization:

- Start time;
- Pressure;
- Temperature;
- Items which are placed in the inside of the autoclaves for sterilization;
- Cycle length;
- Stop time;
- Maximum Temperature
- Initial and date

10.17 Pipettors: All pipettors used for quantitative purposes will be calibrated annually,

either through a certified professional service or in-house. In-house calibration will be conducted gravimetrically, with correction made for temperature and pressure. Calibration records will be kept for 10 years.

11. Preventive Maintenance

Each analytical system or piece of equipment is required to be maintained according to the manufacturer's recommendations. Regular maintenance checks ensure that the systems are able to operate properly and efficiently on a consistent basis.

Each major piece of equipment is covered by a service contract offered by the manufacturer (or a similar company). These contracts include an annual preventive maintenance visit. In addition, more frequent maintenance is performed as recommended by the manufacturer on a regular basis by the laboratory staff as needed.

Maintenance logbooks are utilized to document major preventive as well as emergency maintenance procedures as these are performed. These logbooks are also used to document any routine maintenance / repair procedures.

The following outlines the major and minor preventive maintenance routines for each analytical system in operation within the laboratory:

11.1 Gas Chromatograph
Cut the chromatographic column on a regular basis.
Change injection port septum, o-ring and the glass wool in the liner, when necessary.
Clean by baking, and/or solvent rinsing the Electron Capture Detector, when signal is high.
Service the Electron Capture Detector, when signal high after cleaning the detector.
Cycle/Bake out the entire system once a week, when not in use.

11.2 Mass Spectrometer
Check rough pump oil leads, routinely.
Clean the source when the instrument fails to tune.
Change the electron multiplier when the applied voltage is too high.
Routinely verify that adequate calibration fluids are available for automatic instrument tuning.

Change the o-rings and transfer lines when inspection indicates these are degrading
or weakening.

11.3 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) -
See attachment 5 for preventive maintenance of the ICP-MS.

11.4 Purge and Trap Apparatus -
Purge each line prior to analysis.
Bake the trap once all lines are purged.

Check for leaks in the purge and trap system using the pressurization technique when air leaks are detected.
Change the ferrules when leak check supports a leak; this procedure may be done frequently at most susceptible sites.
Replace the trap when data indicate its degradation.

11.5 Ion Chromatograph

Regularly check the eluent reservoir to see if it needs to be filled.
Daily check the component-mounting panel for leaks or spills. Wipe up spills. Isolate and repair leaks. Rinse off any dried eluent with deionized water.
Weekly check fluid lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.
Check the pump and replace the pump piston rinse seals and piston seals if necessary.
Replace the AS40 Automated sampler tip and tubing once a year.

11.6 Flame Atomic Absorption

11.6.1 Burner Head:

Clean and polish the top surface using mild abrasive soap cleaner;
Clean the burner slot with the Burner Cleaning tool supplied or a piece of stiff card. Do not use an abrasive material inside the slot;
Wash the Burner with detergent solution and rinse with deionized water. Dry the Burner carefully before using it again;
To clean the interior surface of the Burner head, dismantle and use an ultrasonic bath with deionized water, dilute detergent solution or 5% (v/v) solution of nitric acid.

11.6.2 Spray Chamber:

Aspirate a solution of 1% (v/v) hydrochloric acid, or a solution of laboratory detergent to clean the Spray Chamber. Aspirate the solution for 5 minutes, then aspirate deionized water.
For more thorough cleaning, dismantle the Spray Chamber, clean the individual components with lab detergent, rinse thoroughly with deionized water, dry and reassemble.

11.6.3 Nebulizer:

Use the cleaning probe and push through the nebulizer to dislodge to remove any blockage in the nebulizer capillary.

11.7 pH Meter

Clean the electrode prior to and after analysis.
Change the electrode, when necessary.
Store the electrode in pH 7 buffer solution between analyses.
Clean and maintain the magnetic stirrer and magnetic stirring bars, whenever used.
Examine the KCl level in the electrode prior to use, add more solution, when

necessary.

11.8 PC-Titrate
Clean the electrodes/probes prior to after analysis.
Change the electrodes/probes when necessary.
Replace all tubings once a year.

11.9 Water Purification Unit
Check the conductivity of the finished water daily.
Change the cartridges when resistivity starts to decrease, approximately every 2-4 months.

11.10 Quebec Colony Counter and Microscope
Microscopes and colony counters are maintained in a clean condition and checked for defects on each use. All glass surfaces are periodically cleaned as needed. Moving parts of all microscopes are lubricated on an-needed basis.

11.11 Total Organic Carbon Analyzer (TOC-V $_{CPH/CPN}$)

11.11.1 Daily Inspection:
Check the level of dilution water;
Drain vessel water and humidifier water;

11.11.2 Periodic Inspections:
Catalyst Regeneration;
Washing or replacing catalyst;
Replacing the carrier gas purification tube and catalyst;
Washing/replacing the combustion and carrier gas purification tubes;
Replacing the CO2 absorber;
Replacing the Halogen Scrubber;
Syringe replacement.

11.11.3 Autosampler (ASI-V) Maintenance

Please see the *USER's MANUAL* [5] for the details.

11.12 Flow Injection Analysis (FIA) System (QuickChem 8500 Series)

11.12.1 The FIA is composed of the following parts:
- Autodiluter;
- Autosampler;
- Pump;
- Valves;
- Manifolds;
- Detectors;
- Flow Cells;

- Interference Filters;
- Leak Detector;
- Computer.

10.12.2 Maintenance Guide for these parts as listed above could be found in Attachment 6.

Generally, if any instruments have problems, the analysts or technical director will try fix them first. If the problem still persists, service maintenances will be called.

12. Standard Operating Procedures (SOPs)

Each method utilized by the laboratory has a standard operating procedure (SOP) developed by the laboratory (Attachment 7). The analyst follows this procedure at all times to ensure proper operation accuracy and precision. The SOP's include the analytes to be measured, the detection limits of the method, and the applicable matrices. Each SOP also includes a detailed description of the method procedure.

The safety issues involved in the analysis are discussed to insure the safety of the operator and the laboratory environment. All reagents and standards are described as well as the recipes for their preparation. When applicable, sample holding times, appropriate containers, preservatives, and methods of collection are discussed. A step-by-step procedure is detailed with all sample preparation, instrument calibration, and sample analysis steps described.

After completion of the analysis, the SOP describes the calculation procedure and the reporting procedure for the analysis to ensure accurate reporting of the results. Following this section is a description of the QA/QC requirements of the method. Described within this section are the QC samples to be analyzed, the acceptance criteria for each QC sample, and potential corrective action to be taken if the criteria are not met.

12.1 Review, Revision, and Approval

12.1.1 Each SOP or sections subject to revision, will be opened for comments to those who are familiar with its content and/or those who will use it. All proposed SOP revisions must be submitted to the Laboratory Director for review, approval and subsequent distribution.

12.1.2 The following information trigger the revision of the SOP:

- The original methods are revised and approved by regulatory agencies such as US Environmental Protection Agency and MA Department of Environmental protection;
- A new instrument is purchased and used for the Method;
- Any different supplies such as reagents, standards, gases, etc are

changed;
- Initial calibration range is changed due to certain reasons;

12.2 Distribution and Maintenance

12.2.1 Hardcopy SOPs are distributed as controlled documents for project specific external (out of lab) distribution or uncontrolled documents for external review. The distribution of controlled documents must be recorded to ensure that they are updated when new or revised SOPs released.

12.2.2 SOPs for use within Barnstable County Lab are available as uncontrolled hardcopies, controlled hardcopies in binders.

12.2.3 SOPs submitted to regulatory agencies in support of certification or submitted to clients for contract compliance must be controlled for the duration of the certification or contract.

12.2.4 The Laboratory Director shall be responsible for maintaining updated masters for all documents.

12.2.5 All obsolete SOPs are collected and archived and stored away.

13. Data Report, Validation and Review

13.1 Data reporting is performed through the Laboratory Information Management System and a custom created system to report data to Massachusetts Department of Environmental Protection (MA DEP), Drinking Water Program. There are three sections on any lab reports produced by the Lab: (1) Customer Information, (2) Analytical Information, and (3) Signature and Date.

13.1.1 Regarding the Customer Information, the following items must show on any report:

- Reporting mailing address;
- Public Water Supply (PWS) ID# (for reporting to MA DEP);
- PWS Name (for reporting to MA DEP);
- City/Town (for reporting to MA DEP);
- Class: COM, NTNC or TNC (for reporting to MA DEP);
- Multiple or Single (for reporting to MA DEP);
- Raw or Finished (for reporting to MA DEP);
- Date collected;
- Collected by (or Sampler);
- Routine Sample or Special Sample (for reporting to MA DEP);

- Original, Resubmitted or Confirmation (for reporting to MA DEP);
- Reason for resubmission (for reporting to MA DEP);
- Collection Date of Original Sample (for reporting to MA DEP);
- Sample Notes.

13.1.2 Regarding the Analytical Information, the following items must show on any report:

- Primary Laboratory Name;
- Primary Laboratory Massachusetts Certification Number (Barnstable County Health Laboratory: M-MA 009);
- Subcontracted Laboratory Name if any samples are sent out;
- Subcontracted Laboratory Massachusetts Certification Number;
- Sample Matrix;
- Specific Analytes and their Respective Results, Maximum Contaminant Levels (MCL), Maximum Detection Limits (MDL), Analytical Methods, Analytical Dates and time, Analysis Lab MA Certification Numbers, Analysis Lab Name, and Lab Sample ID#;
- Information on the sample composited by the Lab and lab notes;

13.1.3 Raw Data from Any Instruments and Copies from Original Results Recording Log Books:

When analysts have done their analyses, analysts print out the raw data from instruments or get a copy from the original data log book. After analysts enter these raw data into LIMS, analysts submit the raw data with the reports for review.

These raw data will be provided with final laboratory reports to customers if customers request these raw data.

13.2 Data validation is the process by which data are accepted or rejected based on a set of specific criteria. This process is performed to insure possible accuracy of the data and the calculations in data reduction process.

13.2.1 The initial review of all data is performed by the analyst. The analyst checks the raw sample results and compares them to the daily standard(s), daily method blank, and all QC results. If all the QC results meet the criteria established in the method SOP, the completed report sheet for that sample is submitted for validation. All relevant daily QC samples (standards, blanks, check samples, and LFBs) are included with the report sheet. This information is reviewed by the Laboratory Director. Any analysis performed by the Director will be submitted to the Chemist in charge of that area.

13.2.2 The secondary review of all data is preformed by an Analyst or the laboratory

director who did not conduct the associated analyses. The review covers the following areas:

- All raw data supporting the report are included;
- All data in LIMS are correctly entered;
- All criteria of QA/QC are met;
- The subcontract lab name is indicated on the report if any parameters were subcontracted out;
- Any comments on the report are properly addressed.

13.2.3 The final review is conducted by the Laboratory Director, and the content of review is the same as ones described in Section 13.2.2. The reports are then signed and checked off in the LIMS as being completed.

13.2.4 Data reduction includes adjusting reporting limits for sample amount and any dilutions required, rounding of the results occurs after all calculations have been made, all results are reported in two or three significant figures, and no results are reported below the method reporting limit unless requested by customer. Volatile organic results are reported in ug/L or ug/Kg, metal results are reported in mg/L, and bacteriological results are reported in CFU/100 mL (CFU = colony forming units). Inorganic results are generally reported in mg/L except for pH (0.1 pH units), specific conductivity (umhos/cm), alkalinity and total hardness (mg/L $CaCO_3$), and turbidity (NTU or nephelometric turbidity units).

13.2.5 For customers' reference, Barnstable County Laboratory attaches its certified parameter list approved by Massachusetts Department of Environmental Protection to any analytical report.

13.3 Reporting and Recording of Any Non-Conforming Data

13.3.1 Any non-conforming data found during data review and validation will be flagged, explained and recorded in lab narrative. The customer will be notified by phone and through lab narrative.

13.4 Reporting Turn Around Time

13.4.1 Data will be reported to in a timely manner as meets the client's requirements. Turn around times for data reporting will be clearly specified at the time samples are received.

14. Record Keeping, Logbook Review and Standard Traceability

14.1 As part of the QA/QC plan, the procedure for maintaining the records for all aspects of the laboratory operation are well established. Each instrument has a log book in which each analysis is recorded. Also, the printouts of each analysis are filed

according to the intra-laboratory identification number. Included in these files are copies of the appropriate chain-of-custody forms, quality control reports, and all calculations of the data. All this material is available upon request by the client.

14.2 The results of quality control checks such as temperature records for ovens and refrigerators, quality control results for laboratory glassware, distilled water, and microbiological media are stored as permanent records to maintain quality control in the laboratory. Instruments such as pH meters, analytical balances, and thermometers are calibrated daily or prior to use, and the records of these calibrations are also maintained.

14.3 Standard Traceability and Logbook Review:

14.3.1 All standards and reagents received will be called as primary standards that will be recorded in the laboratory primary logbook. Any standards made from the primary standards will be called as either intermediate or working standards which will be recorded in the laboratory working standard logbooks. All primary standards, intermediate and working standards will be assigned unique identification numbers (ID) which will be also recorded. These unique IDs will be recorded on their Certificate of Analyte will be kept in a three-hole binder. The intermediate, working standards and primary standards will be labelled clearly on their containers with the unique IDs.

14.3.2 There are also the following logbooks:

Instrument running logbook
Instrument maintenance logbook
Media preparation logbook
Refrigerator and freezer temperature logbook
Conductance logbook for lab water purification logbook
Buffer preparation logbook
pH meter calibration logbook
Incubator and water bath temperature logbook
Lab corrective action logbook
Customer complaint logbook
Lab staff training logbook

14.3.3 Logbook Review

Laboratory Director or an analyst assigned by the Laboratory Director will review the logbooks quarterly to make sure all recordings are done correctly and properly.

14.4 Document Control: The laboratory maintains the originals and copies of all analytical reports, logs, charts, used SOPs and any other documentation for a minimum of 10 years.

14.5 Document and Record Storage: All originals and copies of analytical reports, logs, charts, used SOPs, and any other documents are stored on site in laboratory data reporting area. The data in LIMS are stored in a server managed by the County IT department, and the LIMS data are backed up daily and carried off site daily by the County IT Department.

15. Safety

A formal safety program for the Laboratory is issued to each new employee. All personnel are introduced to the safety equipment available in the laboratory and instructed as to its use. All analysts are made aware of the safety consideration of the chemicals they use by Right-to-Know training and their review of the individual material safety data sheets. If some requests additional safety measures, the request is acted on immediately.

15.1 All gas tanks such as Helium, Argon, Oxygen, Hydrogen, Acetylene, and Air must be chained and secured.

16. References

1. Environmental Protection Agency, *Methods and Guidance for the Analysis of Water*, Version 2, June 1999.

2. American Public Health Association, American Water Works Association and Water Environment Federation, Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, and 22th Edition, 2012.

3. International Organization for Standardization (ISO) and International Electrotechnical Commission (IEC), General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025:2005(E), Second Edition, 2005-05-15.

4. Barnstable County, *Personnel Policies and Procedures*, Effective July, 2005.

6. SHIMADZU CORPORATION, *User's manual* for Total Organic Carbon Analyzer (TOC-V CPH/CPN) (For TOC-Control V Ver.2), Part# 638-94536,

Laboratory Map

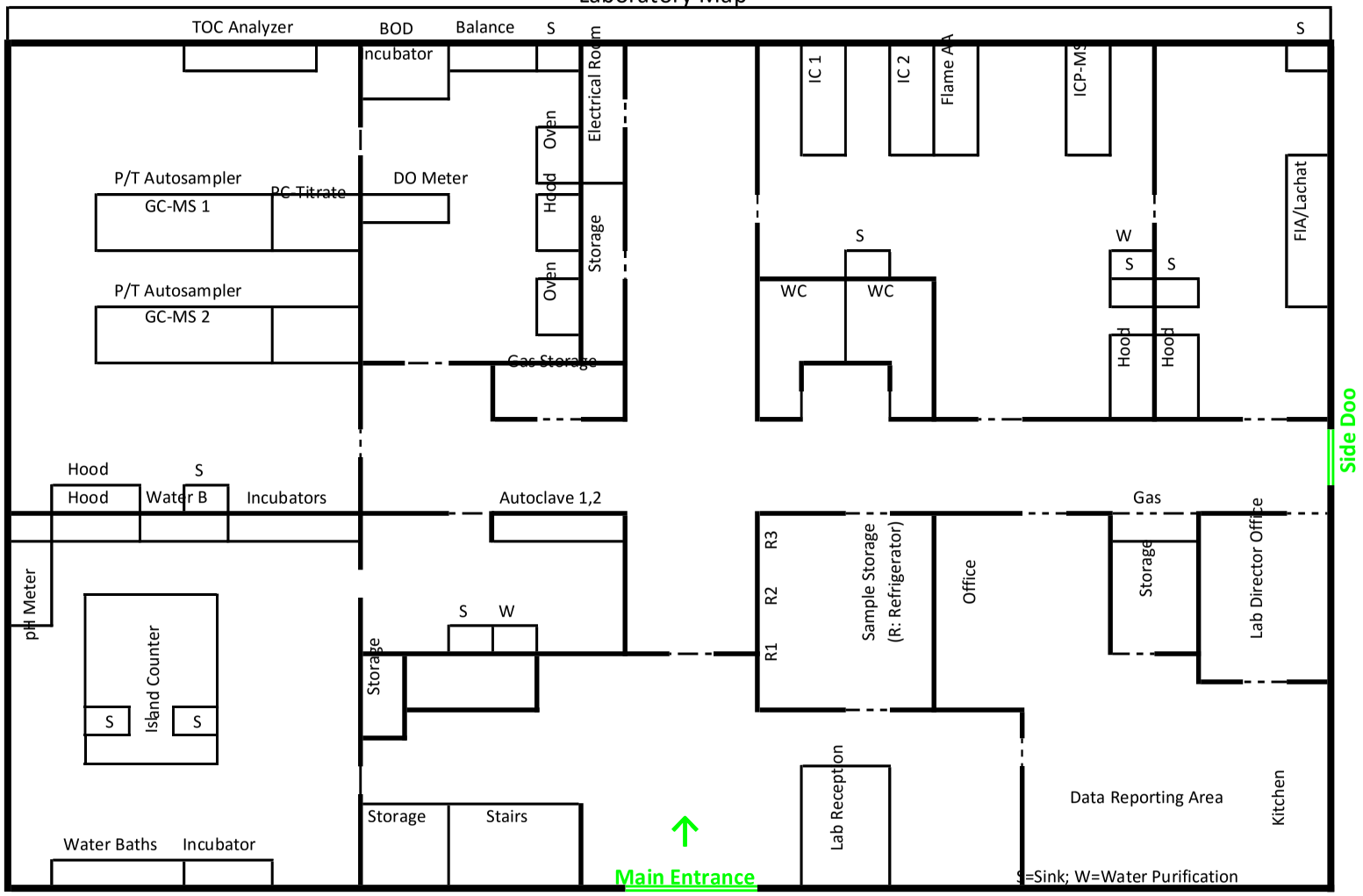

Table 1: QC Data Qualifiers

Symbol	Explanation
B	Analyte found in reagent blank, Indicate possible reagent or background contamination.
E	Reported value exceeded calibration range.
J	Reported value is an estimate because concentration is less than reporting limit and greater than method detection limit or because certain QC criteria were not met.
N	Organic constituents tentatively identified. Confirmation is needed.
PND	Precision not determined.
R	Sample results rejected because of gross deficiencies in QC or method performance. Re-sampling and/or reanalysis is necessary.
RND	Recovery not determined.
U	Compound was analyzed for, but not detected.

ATTACHMENT 1: The Checklist of Laboratory Internal Audits

1. Sample Management

    - Sampling
    - Shipping
    - Receiving
    - Holding times
    - Preservatives
    - Temperature
    - Log-in
    - Storage

2. Analysis

    - Certified methods
    - Standard operating procedures (SOPs)
    - Holding times

3. Quality Assurance and Quality Control (QA/QC)

    - Runlog/Sequence
    - Method blank, Lab Control Sample and Lab Control sample Duplicate (LCS/LCSD), and Matrix Spike and Matrix Spike Duplicate (MS/MSD)
    - Method Detection Limit (MDL) study
    - Accuracy and Precision
    - Control Charts
    - Second Sources
    - Continuing Calibration Verification (CCV)
    - Maintenance logbook
    - Corrective Action Logbook

4. Data Entry, Data Reporting and Data Backup

    - Data entry
    - Report generation
    - Data backup

5. Data Review

    - Analyst level
    - Cross review
    - Final review

6. Report Mailing, Filing and Storage

ATTACHMENT 2: Lab Staff Training Log Book

DATE:	TRAINEE:	TRAINER:
TRAINING START DATE:		
TRAINING ENDING DATE:		
PURPOSES:		
GOALS:		
SPECIFIC TRAINING ACTIONS:		
DOES THE TRAINING MEET THE INITIAL GOALS? EXPLAIN IF NECESSARY.		
COMMENT:		

ATTACHMENT 3: Lab Corrective Action Log Book

Corrective Action Log Book

Date:	Lab:	Originator:

Problem/Issue Description:
Reasons for the Problem/Issue:
Actions Taken to Eliminate The Problem/Issue in the Future:

ATTACHMENT 4: Customer Complaint Log Book

Customer Complaint Log Book

Date:	Name of Customer:	Originator:

Content of the Complaint:

Reasons for the Complaint:

Actions Taken to Satisfy the Customer/Make Things RIGHT:

Comment by Lab Director or Department Director:

ATTACHMENT 5: ICP-MS Routine Maintenance Schedule

Time*	Component	Task	Subsystem
D	Argon gas	Check for argon gas pressure and volume	
D	Peristaltic pump tube	Check for damage	
D	Sampling cone, Skimmer cone	Check orifice	
WN	Sampling cone, Skimmer cone	Clean/replace	Interface
WN	Nebulizer	Clean/replace	Sample introduction
WN	Pelistaltic pump tube	Replace	Sample introduction
WN	Torch	Clean/replace	Torch box
WN	Water filter	Check/replace	Cooling support
1 W	Torch, spray chamber, End cap	Clean	
1 W	Nebulizer	Clean	
1 W	Cooling water	Check water volume and for contaminants	
1 M	Rotary pump	Check oil level and color	Vacuum
1 M	Cooling water filter	Check	
1 M	Extraction Lens (If necessary)	Check	
1 M	Sample tubing	Replace	
6 M	Rotary pump	Change oil	vacuum
1 Y	Oil mist filter of Rotary pump	Check/replace mist filter	vacuum
2 Y	Argon gas filter	Replace (2 years after installation)	

* D = day; WN = when needed; W = week; M = month; Y = year

Attachment 6: Maintenance Guide for Flow Injection Analysis (FIA) System							
Component	**Service**	**Daily**	**Weekly**	**Bi-weekly**	**Monthly**	**6 Months**	**Yearly**
Autodiluter	Clean surfaces	X					
	Prime with DI water	X					
	Replace flared tubing						X
	Replace seals						X
Autosampler	Clean surfaces	X					
	Clean worn gears with dry cloth and alcohol				X		
Pump	Clean rollers with dry cloth and silicone spray				X		
	Replace pump tubes				X		
	Replace pump tubes adapters				X		
Valves	Flush with DI water and air	X					
	Clean ports and O-rings		X				
	Replace valve fitting						X
Manifolds	Flush with DI water and air	X					
	Clean unions and tees			X			
	Replace O-rings					X	
Detectors	Dry and clean surfaces	X					
	Clean tips of fiber optics					X	
Flow cells	Flush with DI water and air	X					
	Replace flares and O-rings					X	
Interference filters	Clean surfaces with cotton swab and isopropanol				X		
System unit	Keep clean	X					
Computer	Clean hard drive					X	
Leak detector	Clean with DI water				X		

Attachment 7: A List of Standard Operating Procedures					
Lab	Method Code	Description	Current Version Number	Current Version Date	Initial
Microbiology	EPA 1604	Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)	002	1/07/2019	RL
Microbiology	SM 9222B, SM9222G	Determination of Total Coliform in Potable and Non Potable Water Using SM 9222B and SM9222G	005	9/30/2019	RL
Microbiology	SM 9223	Determination of Total Coliform Bacteria and *Escherichia coli* (*E.coli*) in Potable Water	003	1/07/2019	RL
Microbiology	SM 9222D	Determination of Fecal Coliform in Potable and Non Potable Water Using SM 9222D	006	9/30/2019	RL
Microbiology	SM 9215B	Heterotrophic Plate Count for Potable Water Using SM 9215B	003	1/07/2019	RL
Microbiology	EPA 1600	Determination of Enterococci in Water using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI)	006	9/30/2019	RL
Microbiology	EPA 1603	Determination of *Escherichia coli* (*E.coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (Modified mTEC)	005	9/30/2019	RL
Microbiology	SM 9223B	Determination of Total Coliform and *E.coli* in Water Using the following Method: Idexx Colilert-18, Colilert-24 and Quanti-Tray	001	4/23/2020	RL
Microbiology	SM 9223B	Determination of Fecal Coliform in Waste Water Using the following Method: Idexx Colilert-18, Colilert-24 and Quanti-Tray	001	4/24/2020	RL
Microbiology	SM 9230B	The Detection of Enterococci in Water Using the following Method: Enterolert and Quanti-Tray	001	4/23/2020	RL
Organics	EPA 524.2	Determination of Volatile Organic Compounds in Aqueous Samples Using Gas Chromatography/Mass Spectrometry	013	6/15/2020	YN
Organics	EPA 624.1	Determination of Volatile Organic Compounds in Non-potable Water Using Gas Chromatography/Mass Spectrometry	002	6/18/2020	YN
Organics	SM 5310 B	Determination of Total Organic Carbon (TOC) in Aqueous Samples Using High-Temperature Combustion Method	012	10/28/2020	LX
Organics	HACH	Determination of Chemical Oxygen	015	2/01/2019	LP

	8000	Demand (COD) in Aqueous Samples			
Inorganics	EPA 200.8	Determination of Trace Elements in Aqueous Samples by Inductively Coupled Plasma – Mass Spectrometry	012	10/29/2020	CL
Inorganics	SM 3111B	Determination of Sodium, Copper, Iron, Manganese, Zinc, Nickel, Potassium, Calcium, Magnesium in Aqueous Samples Using SM 3111B	014	10/29/2020	CL
Inorganics	EPA 314.0	Determination of Perchlorate in Aqueous Samples Using Ion Chromatography	015	10/29/2020	LP
Inorganics	EPA EPA 300.0	Determination of Inorganic Anions in Aqueous Samples Using Ion Chromatography	015	5/11/2018	LP
Inorganics	SM 2320B	Determination of Alkalinity in Aqueous Samples	010	6/16/2020	YN
Inorganics	SM 2510 B EPA 120.1	Determination of Conductance in Aqueous Samples	011	6/17/2020	YN
Inorganics	SM 4500-H-B	Determination of pH in Aqueous Samples	010	6/17/2020	YN
Inorganics	SM 2540 C	Determination of Total Dissolved Solids in Aqueous Samples	010	9/06/2019	LP
Inorganics	SM 2540 D	Determination of Total Suspended Solids in Aqueous Samples	010	9/06/2019	LP
Inorganics	SM 2540B	Determination of Total Solids in Aqueous Samples	010	9/06/2019	LP
Inorganics	EPA 180.1	Determination of Turbidity in Aqueous Samples	008	8/29/2020	AB
Inorganics	SM 2340B	Determination of Hardness in Aqueous Samples (Refer to SM 3111B)	012	12/22/2017	LAP
Inorganics	EPA 351.2	Determination of Total Kjeldahl Nitrogen in Aqueous Samples by Semi-Automated Colorimetry	005	11/09/2018	KK
Inorganics	EPA 350.1	Determination of Ammonia Nitrogen in Aqueous Samples by Semi-Automated Colorimetry – Gas Diffusion Method	006	3/05/2019	KK
Inorganics	SM 2120B	Determination of Color in Aqueous Samples	003	6/15/2018	DB
Inorganics	SM 2150B	Determination of Odor in Aqueous Samples	003	6/15/2018	DB

APPENDIX 8:
SAMPLE PREPARATION AND TRANSPORTATION

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: Sample Preparation and Transportation

Effective Date: 2021-07-15	**Number:** MASSTC-SOP-015	**Revision:** 000

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Sample Preparation and Transportation	Document ID#: MASSTC-SOP-015 Revision#: 000 Released Date: 2021-07-15 Released By: Brian Baumgaertel

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1. Scope and Application

1.1. This protocol describes the procedures used to prepare bottle labels and chains of custody for sampling as well as for the preparation of transportation of samples.

2. Definitions

2.1. None.

3. Health and Safety Warnings

3.1. Biohazard – Contact with wastewater can result in sickness, injury or death. **Wear goggles and gloves**.

3.2. Chemical – Contact with preservatives (such as sulfuric acid) can result in sickness, injury or death. **Wear goggles and gloves and hold bottles upright.**

4. Cautions

4.1. Ensure that the correct bottle size, type, and preservative is being used. Consult MASSTC Director or Water Quality Lab Director if needed.

4.2. Ensure the correctly-labeled bottle is used at the corresponding sample location.

5. Interferences

5.1. Always consult MASSTC-FRM-040 – Sampling Plan to ensure that the correct parameters and frequency of sampling are followed.

5.2. Ensure that the correct lab report turnaround time as requested by client is being followed and is specified on the chain of custody.

5.3. Sample locations must be grouped together on the Chain of Custody to minimize confusion of Barnstable County Water Quality Lab Staff. For example, do not create an order for “Port 1 TSS”, then an order for “Final Effluent nitrate”, then go back to “Port 1 alkalinity.”

5.4. Samples must have unique sample numbers (no duplicates) on the day on which they are received by the Water Quality lab.

5.5. When samples are being packed into a cooler for transportation, a MASSTC Staff Member must initial next to the sample as it is placed in the cooler to minimize the chance of losing a sample.

6. Personnel Qualifications

6.1. Personnel are required to be knowledgeable of the procedures in this SOP.

6.2. Personnel are required to be knowledgeable of the Sampling Plan and Sampling Sketch (MASSTC-SOP-030).

7. Special Apparatus and Materials

7.1. Coolers.

7.2. Ice packs or ice.

8. Instrument or Method Calibration

8.1. None.

9. Procedure for Creation of Lab Orders and Preparation of bottles

9.1. Prepare bottle labels and chains of custody. This should be done no later than Friday afternoon for the week before so that all MASSTC staff have the opportunity to double-check bottles and Chains of Custody.

9.1.1. Consult Sampling Schedule calendar, found electronically on Sharepoint in MASSTC-Documents – Documents – Uncontrolled Documents – Chains, as the Excel sheet "Sample Schedule [*dates*]."

9.1.1.1. Sampling Schedule will state which project and location must be sampled on that day of the week.

9.1.1.2. If any clients have requested non-routine samples, be sure to include these.

9.1.2. Create the Chain of Custody.

9.1.2.1. Open the MASSTC Database, located at http://10.14.20.130:31983/

9.1.2.2. Under the left-hand column sections, click on Sampling, and then click on Chains.

9.1.2.3. Click on Add Chain to create a new Chain of Custody. Alternatively, find the most recently used Chain of Custody, click on the downward facing arrow, and click on Duplicate.

9.1.2.4. Consult MASSTC-FRM-040 – Sampling Plan in the Sampling Plans Binder or the most updated electronic copy and ensure that every sample parameter named in the Sampling Plan appears on the Chain of Custody.

9.1.2.4.1. If MASSTC-FRM-040 – Sampling Plan does not exist for the project in question, consult MASSTC-SOP-030 – Sampling and Maintenance Plans to complete this form.

9.1.2.4.2. Include any Rush Order on lab turnaround. This should be chosen from drop-down menu on electronic Chain of Custody and also should be stamped with the red "Rush" stamp. Any sample parameters that have different rushes must be put on separate Chains of Custody.

9.1.2.4.3. Include any influent samples required by client on Chain of Custody.

9.1.2.5. **Sample locations should appear on the Chain of Custody grouped by sample location.** Do not interchange sample locations out of order on the Chain of Custody. This minimizes errors made by Water Quality Lab Staff. If a sample parameter was missed at one location, delete any sample parameters of other locations and then re-add them.

9.1.2.6. Ensure that the Sample Date is correct. If changed, make sure to click Apply Changes.

9.1.2.7. Ensure that the Starting Chain Sample Number is correct. There should never be duplicate numbers of samples brought to the Water Quality Lab on a single day. If the starting number needs to be changed, key in the correct number and click Apply Changes.

9.1.2.8. **As much as possible, try to start samples at #1 and go chronologically without number gaps.** If known in advance that samples will be delivered on a different day than they are taken, try starting at 100, 200, etc so as to ensure no duplicates.

9.1.2.9. **Separate any BOD_5 or $cBOD_5$ order from other parameters.** This helps the Barnstable County Water Quality Lab staff, and also increases likelihood of timely reports.

9.1.2.10. Make sure the "Send Report To" has the client's actual name (and does not say "Client"). Click Apply Changes if this was changed.

9.1.2.11. The fields for Send Report To and Bill To should be the same unless otherwise confirmed by the MASSTC Director.

9.1.2.12. Add the LIMS client ID into the comments section (preferably, to minimize errors of Barnstable County Water Quality Lab staff).

9.1.3. When the Chain of Custody is completed and you are certain that the Chain of Custody correctly matches the Sampling Plan's requirements, click on Print Chain.

9.1.3.1. Print a physical copy of the Chain of Custody and place it on the table where the sample bottles will be set up.

9.1.3.2. Save a copy of the Chain of Custody electronically in Sharepoint – MASSTC-Documents – Documents – Uncontrolled Documents – Chains. The name should automatically be created by the system but make sure to update the title date to the day that the samples will be taken.

9.1.4. Print the labels.

9.1.4.1. Click on Print Labels and save the labels.

9.1.4.2. Using Dymo Print software, print the labels on the Dymo Label Printer.

9.1.4.3. **When printing a large number of labels in one job (more than 4 labels), you must stand by the label printer and gently pull the labels as they are printed.** If this is not done, the label printer may jam and a label may be skipped without your noticing.

9.1.5. Place the labels on the bottles. If unsure of the correct bottle to use, consult the Chain of Custody which states the bottle size and preservative.

9.1.6. Finish all bottle preparation and Chains of Custody needed for the entire week based on the Sampling Board and Sampling Plans.

9.1.7. Make sure bottles and Chains of Custody are grouped together clearly and separated by day.

9.1.8. Consult another MASSTC Staff member to ensure nothing has been missed. If corrections need to be made, restart at 10.1.

9.1.9. By the end of the last business day of the week (usually Friday), after other MASSTC staff have looked over the bottles and Chains of Custody, e-mail all Chains of Custody to the Water Quality Lab Staff.

9.1.10. If any Chains of Custody need to be created or edited later during the week, restart at 9.1.2.

10. SAMPLE STORAGE

10.1. Samples should be analyzed as soon as possible after they are taken, but at the very least, within holding time.

10.2. Samples cannot be preserved for later analysis.

10.3. Samples must be bottled and stored according to the following table:

Parameter	Container Volume	Container	Processing/Storage	Holding Time
Alkalinity	60-125 ml	Polyethylene	Stored on ice (dark)	14 Days
Ammonium	60-125 ml	Polyethylene (H_2SO_4 Preserved)	Stored on ice (dark)	28 Days

BOD_5 or $CBOD_5$	500 ml	Nalgene	Stored on ice (dark)	48 Hours
COD	60-125 ml	Polyethylene (H_2S0_4 Preserved)	Stored on ice (dark)	28 Days
E. Coli	100 ml	Polyethylene	Collected aseptically by hand at discharge site; stored on ice (dark)	<6 Hours
Fecal Coliform	100 ml	Polyethylene	Collected aseptically by hand at discharge site; stored on ice (dark)	<6 Hours
Nitrate + Nitrite	60-125 ml	Polyethylene	Stored on ice (dark)	48 Hours
Ortho-phosphate	60-125 ml	Polyethylene	Stored on ice (dark)	48 Hours
Total Kjeldahl Nitrogen	60-125 ml	Polyethylene (H_2S0_4 Preserved)	Stored on ice (dark)	28 Days
Total Phosphorus	60-125 ml	Polyethylene (H_2S0_4 Preserved)	Stored on Ice (dark)	28 Days
Total Suspended Solids	1000 ml	Polyethylene	Stored on Ice (dark)	7 Days

10.4. Samples must be rinsed with tap water, disinfected, and rinsed again before placing in fridge.

10.5. See MASSTC-SOP-037 – Sample Collection for procedure on procuring samples.

11. SAMPLE TRANSPORTATION

11.1. On the appropriate day, collect and pour samples according to MASSTC-SOP-037 – Sample Collection.

11.1.1.1. Write the time at which the sample was taken and the initials of the person who took the sample in the Comment field on the Chain of Custody for each sample.

11.1.2. Store sample bottles in refrigerator until delivery.

11.1.3. When sample collection is complete, contact the courier service to confirm that samples are ready, or use schedule already arranged with courier for pick up deadline.

11.1.3.1. If a MASSTC Staff Member is delivering samples, alert the person delivering samples when ready.

11.1.4. Before delivery, MASSTC Staff must pack samples into coolers and add ice to ensure samples remain cold.

11.1.4.1. **The MASSTC Staff member who packing coolers must check off each sample as it is placed into the cooler and write his/her initials next to the number on the Chain of Custody as a written record.**

11.1.4.2. If any samples are missing, that MASSTC Staff Member must attempt to find the missing sample(s), or reprint the label and retake the sample(s).

11.1.5. Before delivery, MASSTC Staff Member must sign at bottom of Chain of Custody for release.

11.1.6. Samples must be delivered to the Barnstable County Water Quality Lab on the same day as taking samples or as soon as possible.

11.2. Staff must be aware of holding times to ensure that no samples are delivered to the Water Quality Lab outside of holding time.

12. Data Analysis/Calculations

12.1. None.

13. Data Management/Records Management

13.1. Save Chains of Custody in PDF format as stated in 10.1.3.2. Archived data are subject to official retention schedule contained in MASSTC-SOP-003, Records and Archives.

14. Quality Control

14.1. Always consult the MASSTC-FRM-040 – Sampling Plan to ensure proper sample order and chains of custody are created.

15. Internal and External References

15.1. MASSTC-SOP-003 – Data and Records Management

15.2. MASSTC-SOP-030 – Sampling and Maintenance Plans

15.3. MASSTC-SOP-037 – Sample Handling

16. Forms and Data Sheets

16.1. MASSTC-FRM-040 – Sampling Plan

APPENDIX 9:
ProDSS™ CALIBRATION CHECKLIST

Massachusetts Alternative Septic System Test Center

Barnstable, Massachusetts

Form

Title: ProDSS Calibration Checklist

Effective Date: 2021-10-18	**Number:** MASSTC-FRM-033	**Revision:** 002

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ProDSS Calibration Checklist

Document ID#: MASSTC-FORM-033
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History	Effective Date
Revision #002: Added Turbidity calibration section, and reformatted to fit to single page. Edits done by EMO	2021-10-18
Revision #001: Added pH 7 acceptable mV range. Added sections to write pH and ODO CCV. Updated by EMO.	2021-06-09
Revision #000: Original Issue	2020-08-21

ProDSS Calibration Checklist

Document ID#: MASSTC-FORM-033
Revision#: 002
Released: 2021-10-18
Released By: Brian Baumgaertel

Date (mm/dd/yyyy):					
Time:					
Meter color:					
Initials:					
Specific Conductance					
Conductivity reading in dry, room-temperature air:					
Conductivity Calibration - done today?	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
Conductivity Calibration - date last completed on this meter (mm/dd/yyyy):					
Turbidity					
Turbidity Calibration - done today?	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
If calibrated: Date Turbidity standards last changed?(mm/dd/yyyy)					
Optical Dissolved Oxygen					
ODO Calibration - did you successfully complete it today?	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
Today's Post-Cal ODO%					
pH Calibration					
pH Calibration - did you successfully complete it today? (yes or no)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
pH 7 mV reading was within -50 to +50 mV range? (MUST STOP IF OUT OF RANGE)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
Date pH standards were last changed:					
Date pH standards must be changed again (2/week):					
Continuing Calibration Verifications (CCV)					
Did you log pH 7 CCV? (yes or no)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
pH 7 CCV:					
Did you log ODO CCV *(and wait 5-15 minutes in ODO cup for saturation)* ? (yes or no)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
ODO CCV:					
Notes:					

When completed, please scan and place in: Sharepoint - MASSTC-Documents - Data - ProDSS Downloaded Data - Calibration Logs.
Put the hard copy in the ProDSS Calibration binder in the lab

ProDSS Calibration Checklist

Document ID#: MASSTC-FORM-033
Revision#: 002
Released: 2021-10-18
Released By: Brian Baumgaertel

Date (mm/dd/yyyy):					
Time:					
Meter color:					
Initials:					
Specific Conductance					
Conductivity reading in dry, room-temperature air:					
Conductivity Calibration - done today?	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
Conductivity Calibration - date last completed on this meter (mm/dd/yyyy):					
Turbidity					
Turbidity Calibration - done today?	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
If calibrated: Date Turbidity standards last changed?(mm/dd/yyyy)					
Optical Dissolved Oxygen					
ODO Calibration - did you successfully complete it today?	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
Today's Post-Cal ODO%					
pH Calibration					
pH Calibration - did you successfully complete it today? (yes or no)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
pH 7 mV reading was within -50 to +50 mV range? (MUST STOP IF OUT OF RANGE)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
Date pH standards were last changed:					
Date pH standards must be changed again (2/week):					
Continuing Calibration Verifications (CCV)					
Did you log pH 7 CCV? (yes or no)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
pH 7 CCV:					
Did you log ODO CCV *(and wait 5-15 minutes in ODO cup for saturation)* ? (yes or no)	o Yes o No	o Yes o No	o Yes o No	o Yes o No	o Yes o No
ODO CCV:					
Notes:					

When completed, please scan and place in: Sharepoint - MASSTC-Documents - Data - ProDSS Downloaded Data - Calibration Logs.
Put the hard copy in the ProDSS Calibration binder in the lab

APPENDIX 10:
ProDSS™ END OF DAY CHECKLIST

Massachusetts Alternative Septic System Test Center

Barnstable, Massachusetts

Form

Title: ProDSS End of Day Checklist

Effective Date: 2020-08-21	**Number:** MASSTC-FRM-034	**Revision:** 000

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ProDSS End of Day Checklist

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ProDSS End of Day Checklist

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Released By: Brian Baumgaertel

Date (mm/dd/yyyy):					
Time:					
Meter color:					
Initials:					
Rinse and check 7 pH?					
Cleaned DO cup (including **emptying and scrubbing, then refilling with water**)?					
Emptied and rinsed white tray?					
Emptied and rinsed pH discard/rinse cup?					
Cleaned conductivity probe using brush and clean water?					
Conductivity reading:					
Rise, gently pat dry, and check ODO?					
Wipe down screen and entire meter body?					
Wipe any dirt off of cord?					
Detangle cord?					
Cable hanging and off ground?					
Data has been dowloaded and saved on Sharepoint?					
(If not put on Sharepoint, why not?)					
Connected meter to power and charging?					
Calibration cup is sealed?					
Turned off meter?					
Notes					

When completed, please scan and place in: Sharepoint - MASSTC-Documents - Data - ProDSS Downloaded Data - End of Day Checklists.
Put the hard copy in the ProDSS Calibration binder in the lab

ProDSS End of Day Checklist

Document ID#: MASSTC-FORM-034
Revision#: 000
Released: 2020-08-21
Released By: Brian Baumgaertel

Date (mm/dd/yyyy):					
Time:					
Meter color:					
Initials:					
Rinse and check 7 pH?					
Cleaned DO cup (including **emptying and scrubbing, then refilling with water**)?					
Emptied and rinsed white tray?					
Emptied and rinsed pH discard/rinse cup?					
Cleaned conductivity probe using brush and clean water?					
Conductivity reading:					
Rise, gently pat dry, and check ODO?					
Wipe down screen and entire meter body?					
Wipe any dirt off of cord?					
Detangle cord?					
Cable hanging and off ground?					
Data has been dowloaded and saved on Sharepoint?					
(If not put on Sharepoint, why not?)					
Connected meter to power and charging?					
Calibration cup is sealed?					
Turned off meter?					
Notes					

When completed, please scan and place in: Sharepoint - MASSTC-Documents - Data - ProDSS Downloaded Data - End of Day Checklists.
Put the hard copy in the ProDSS Calibration binder in the lab

APPENDIX 11:
CHEMICAL RECEIPT LOG

Massachusetts Alternative Septic System Test Center

Barnstable, Massachusetts

Form

Title: Chemical Receipt Log

Effective Date: 2020-08-21	**Number:** MASSTC-FRM-014	**Revision:** 001

Authors

Name: Brian Baumgaertel
Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... Date: 8/21/2020

Approvals

Name: Brian Baumgaertel
Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... Date: 8/21/2020

Chemical Receipt Log

Document ID#: MASSTC-FORM-014
Revision#: 001
Released: 2020-08-21
Released By: Brian Baumgaertel

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Revision #001: Reformatted. Added "MSDS Check" column. Revisions by Brian Baumgaertel.	2020-08-21
Revision #000: Original Issue	2018-02-12

Chemical Receipt Log

Document ID#: MASSTC-FORM-014
Revision#: 001
Released: 2020-08-21
Released By: Brian Baumgaertel

Chemical Name	Lot/Batch Number	Date Rec'd	Rec'd By	Date Opened	Opened By	MSDS Checked	Comments

APPENDIX 12:
CONTROL OF NONCONFORMING WORK

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: Control of Nonconforming Work

Effective Date: 2021-11-18	**Number:** MASSTC-SOP-004	**Revision:** 001

Authors

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Title: MASSTC Director

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Title: Environmental Project Assistant/Quality Assurance Manager

Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

Approvals

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Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... **Date:** 11/18/2021

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Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

REVISION HISTORY

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the MASSTC Director on the MASSTC Sharepoint Site.

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Revision #001: Updated cover page to include Quality Assurance Manager approval. Updated SOP references. Edits done by EMO and BB.	2021-11-18
Revision #000: Original Issue	2019-10-02

Control of Nonconforming Work SOP	**Document ID#:** MASSTC-SOP-004 **Revision**: 001 **Released Date**: 2021-11-18 **Released By**: Brian Baumgaertel

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4. Procedure for Control of Nonconforming Work 4
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6. Records 5
7. Forms and Data Sheets 5

1. PURPOSE

1.1. This document defines the procedure used to identify, evaluate, and address any aspect of work conducted by MASSTC or results of work conducted by MASSTC which does not conform to the policies and procedures in the MASSTC Quality Management System or the agreed upon requirements of the customer.

2. SCOPE AND APPLICATION

2.1. This procedure applies to all work conducted by all MASSTC personnel.

3. IDENTIFICATION OF NONCONFORMING WORK

3.1. Identification of nonconforming work or problems with the management system or sampling, measurement or analytical activities can occur at various points within the management system and technical operations.

3.2. Nonconforming work may be identified several ways including customer complaints, quality control, instrument calibration, checking of consumable supplies, staff observations, report reviews, management reviews, and internal and external audits.

4. PROCEDURE FOR CONTROL OF NONCONFORMING WORK

4.1. A nonconformance occurs anytime there is a departure from the policies and procedures in the MASSTC Quality Management System or technical operations or when there is an absence of a specified requirement. Nonconformances will require a formal corrective action if 1) there is potential for the nonconformance to recur somewhere else in the quality system or, 2) if there is an adverse impact on the quality of the work generated.

4.2. MASSTC personnel will address nonconformances according to the following procedure.

4.2.1. When nonconforming work is identified, the Quality Assurance Manager or the manager on duty has the authority and responsibility to stop work if appropriate.

4.2.2. Whenever a nonconformance occurs, MASSTC personnel will take immediate action to correct the issue if appropriate.

4.2.3. The individual who identified the nonconformance will complete the Nonconforming Work Report (MASSTC-FRM-004) and notify the Quality Assurance Manager as soon as possible.

4.2.4. The Quality Assurance Manager, in consultation with staff, will evaluate the significance of the nonconformance.

4.2.5. The Quality Assurance Manager will determine if there is potential for the nonconformance to recur somewhere else in the quality system or if there is an adverse impact on the quality of the work generated. If so, it will be addressed through formal corrective action per the Corrective Action SOP (MASSTC-SOP-005).

4.2.6. If the nonconformance is not addressed through the corrective action process, it will be evaluated by the Quality Assurance Manager to determine if it is an opportunity for a Preventive Action or Quality Improvement.

4.2.7. The Quality Assurance Manager will determine if the results generated from the nonconforming work are acceptable and if the work should be repeated.

4.2.8. The Quality Assurance Manager will also determine if the client should be notified of the nonconformance and if any previously released data should be recalled.

4.2.9. If work was stopped due to the nonconformance, the Quality Assurance Manager will determine when it is appropriate for work to resume.

5. Recalled Data

5.1. If it is necessary to recall data due to a nonconformance, it will be the responsibility of the Quality Assurance Manager or MASSTC Director to contact the customer and notify them of the recalled data. This may be done via email or a memorandum. If it is necessary to issue another report, this will be handled in accordance the Standard Operating Procedure for Report Preparation and Distribution (MASSTC-SOP-042).

6. Records

6.1. When identified, information related to occurrences of nonconforming work will be recorded on the Control of Nonconforming Work Form (MASSTC-FRM-004). The Quality Assurance Manager will begin follow-up. The Quality Manager will maintain all records associated with occurrences of nonconforming work. These records may include:

6.1.1. Nonconforming Work Form (MASSTC-FRM-001)

6.1.2. Notification for stopping work (e-mails, memos, verbal communication)

6.1.3. Records of customer notification (e-mails, memos, verbal communication)

6.1.4. Records of recalled data (e-mails, memos, verbal communication)

6.1.5. Notification for resuming work (e-mails, verbal communication)

6.1.6. Corrective Action Report (MASSTC-FRM-002)

7. Forms and Data Sheets

7.1. MASSTC-FRM-001 – Control of Nonconforming Work Form

7.2. MASSTC-FRM-002 – Corrective Action Report

APPENDIX 13:
CORRECTIVE ACTION SOP

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Standard Operating Procedure

Title: Corrective Action SOP

Effective Date: 2021-11-18	**Number:** MASSTC-SOP-005	**Revision:** 000

Authors

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Title: MASSTC Director

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Title: Environmental Project Assistant/Quality Assurance Manager

Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

External SOPs and Authors

None

Approvals

Name: Brian Baumgaertel
Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... **Date:** 11/18/2021

Name: Emily Michele Olmsted
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Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

Corrective Action SOP	**Document ID#:** MASSTC-SOP-005 **Revision #:** 000 **Released Date:** 2021-11-18 **Released By:** Brian Baumgaertel

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Corrective Action SOP	**Document ID#:** MASSTC-SOP-005 **Revision #:** 000 **Released Date:** 2021-11-18 **Released By:** Brian Baumgaertel

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6. Records Management 5
7. Internal and External References 5
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1. PURPOSE

1.1. This document defines the procedure used to handle corrective action at MASSTC.

2. SCOPE AND APPLICATION

2.1. This procedure applies to the Quality Assurance Manager and Director of MASSTC.

3. DEFINITIONS

3.1. **Corrective Action** – An action initiated in response to an identified nonconformance, in order to define a problem, attempt to identify the root cause and determine how to prevent the problem from recurring

3.2. **Nonconformance** – Departure from the policies and procedures in the MASSTC Quality System or technical operations, or the absence of a specified requirement.

3.3. **Quality Assurance Manager** – Unless otherwise designated, the Director of MASSTC.

4. INITIATION AND TRACKING

4.1. Once the need for a corrective action has been identified, anyone within the MASSTC staff can initiate a corrective action request through the Quality Assurance Manager.

4.2. Corrective actions will be uniquely identified to facilitate tracking. A seven-digit identification number will be assigned to each corrective action by the Quality Assurance Manager. Tracking numbers will begin with MASSTC-CAR. The first four digits will represent the calendar year. The last three digits will follow and begin at 001 and increase sequentially with each additional corrective action. The last three digits will start over at 001 at the beginning of each calendar year (Ex. MASSTC-CAR-2020-001).

5. PROCEDURE

5.1. Upon identification or notification of the need for a corrective action, the Quality Assurance Manager will assign a corrective action identification number and begin documentation of the corrective action on the Corrective Action Report (MASSTC-FRM-002).

5.2. The Quality Assurance Manager, in consultation with MASSTC staff, will assess the issues surrounding the problem. Staff members consulted will be noted in the record.

5.3. The Quality Assurance Manager will investigate the issue to determine the root cause of the problem. A summary of the assessment with be included in the record on the Corrective Action Report (MASSTC-FRM-002).

5.4. Once the root cause of the problem has been identified, the Quality Assurance Manager will determine how to correct the problem and prevent it from recurring. A summary of the cause and solution will be included with the record.

5.5. If any policies or procedures require updates, the Quality Assurance Manager will ensure they are conducted in accordance with the MASSTC Document Control SOP (MASSTC-SOP-001).

5.6. The Quality Assurance Manager will notify all affected personnel in writing of any changes that result from the corrective action process. Any affected controlled documents (SOPs, forms, etc.) will also be updated should the need arise.

5.7. Management is responsible for ensuring all affected personnel are implementing any changes. Some ways that management may do this is through direct communication with their staff and reviews of project records.

5.8. The Quality Assurance Manager will formally monitor the effectiveness of corrective actions by conducting a review of the corrective action. The time frame for reviews will be determined by the Quality Assurance Manager and will be based on the magnitude and risk of the problem. Multiple follow-ups may be conducted to ensure the effectiveness of the corrective action.

5.9. If the Quality Assurance Manager determines that the corrective action is not effective, based on the magnitude and risk of the problem, the Quality Assurance Manager is to be tasked with reevaluating the problem and proposing another solution.

5.10. Once the problem has been adequately addressed, the Quality Assurance Manager will close-out the Corrective Action Report and the associated Nonconforming Work Report.

6. Records Management

6.1. The Quality Assurance Manager will maintain all records associated with corrective actions. The records may include but are not limited to:

6.1.1. Documentation associated with root cause analysis.

6.1.2. Documentation associated with solutions to the problem.

7. Internal and External References

7.1. MASSTC-SOP-001 – Document Control SOP

7.2. MASSTC-SOP-004 – Control of Nonconforming Work SOP

8. Forms and Data Sheets

8.1. MASSTC-FRM-001 – Nonconforming Work Report

8.2. MASSTC-FRM-002 – Corrective Action Report

APPENDIX 14: NONCONFORMING WORK REPORT

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Form

Title: Nonconforming Work Report

Effective Date: 2021-11-18	**Number:** MASSTC-FRM-001	**Revision:** 001

Internal Authors

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Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... **Date:** 11/18/2021

Name: Emily Michele Olmsted
Title: Environmental Project Assistant/Quality Assurance Manager

Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

External SOPs and Authors

None

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Name: Brian Baumgaertel
Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... **Date:** 11/18/2021

Name: Emily Michele Olmsted
Title: Environmental Project Assistant/Quality Assurance Manager

Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

Nonconforming Work Report	**Document ID#:** MASSTC-FRM-001 **Revision#:** 001 **Released Date:** 2021-11-18 **Released By:** Brian Baumgaertel

Revision History

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History	Effective Date
Revision #001: Converted to standard template. Removed "Entered into database" fields. Reformatted Section A table and added "How did you identify the nonconformance?" question. Added Section B and C. Added significance of nonconformance. Edits by BB. Added instruction on where to put electronic and paper copies. Edits by EMO.	2021-11-18
Revision #000: Original Issue	2019-10-02

Nonconforming Work Report	**Document ID#:** MASSTC-FRM-001 **Revision#:** 001 **Released Date:** 2021-11-18 **Released By:** Brian Baumgaertel

Filled out by Reporter:		**Quality Assurance Manager Only:**	
Nonconformance Date and Time		Nonconforming Work Report Number	
Reported By		Corrective Action Report Number	
Report Date and Time			

Section A - Nonconformance Details

Filled out by Reporter:

Where did the nonconformance take place? LIST ALL PROJECTS INVOLVED, LOCATION, ETC.
How did you identify the nonconformance?
☐ Internal Audit ☐ Customer Complaint ☐ Incidental ☐ Other: _____
What is nonconforming? EXAMPLE: CLOSING CALIBRATION VERIFICATION FOR D.O. WAS OUT OF SPECIFICATION.
Related SOP, Policy, Etc.
Why is it a nonconformance? EXAMPLE: D.O. MUST BE WITHIN +/- 2% OF THE CALIBRATION VALUE FOR THE DAY

Section B – Disposition

Quality Assurance Manager Only:

Data/Results Acceptable?	( ) No ( ) Yes	
Redo Nonconforming Work?	( ) No ( ) Yes	
Corrective Action Required?	( ) No ( ) Yes	CAR#
Preventive Action Required?	( ) No ( ) Yes	
Significance of nonconformance		
Comments INCLUDE PREVENTIVE ACTION OR QUALITY IMPROVEMENT NOTES HERE.		
Report Closure Date		

Quality Manager Signature Date	MASSTC Director Signature Date

Upon completion, this document must be stored electronically in Sharepoint's MASSTC-Data and Records – Quality Assurance – Nonconforming Work Reports. Paper copies must go in the Nonconforming Work Reports binder.

APPENDIX 15:
CORRECTIVE ACTION REPORT

Massachusetts Alternative Septic System Test Center
Barnstable, Massachusetts

Form

Title: Corrective Action Report

Effective Date: 2021-11-18	**Number:** MASSTC-FRM-002	**Revision:** 000

Internal Authors

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Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... **Date:** 11/18/2021

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Title: Environmental Project Assistant/Quality Assurance Manager

Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

External SOPs and Authors

None

Approvals

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Title: MASSTC Director

Signature: DocuSigned by: Brian Baumgaertel A809A6344B57407... **Date:** 11/18/2021

Name: Emily Michele Olmsted
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Signature: DocuSigned by: E5E55A7B2C05436... **Date:** 11/18/2021

Corrective Action Report	**Document ID#:** MASSTC-FRM-002 **Revision#:** 000 **Released Date:** 2021-11-18 **Released By:** Brian Baumgaertel

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Revision #000: Original Issue	2021-11-12

Corrective Action Report	**Document ID#:** MASSTC-FRM-002 **Revision#:** 000 **Released Date:** 2021-11-18 **Released By:** Brian Baumgaertel

Corrective Action Report Number		Nonconformance Report Number	
Report Date		Full Closeout Date	

Section A – Root Cause Analysis

Staff Members Consulted

Five Why's

Why 1
Why 2
Why 3
Why 4
Why 5

Root Cause Analysis Results

Section B – Corrective Actions to Be Taken

Corrective Action #1			
Responsible Parties			
Monitoring Period			
Follow-up (Include date and reviewer initials)			
Closeout Date			
Quality Manager Signature	Date	MASSTC Director Signature	Date

Corrective Action #2			
Responsible Parties			
Monitoring Period			
Follow-up (Include date and reviewer initials)			
Closeout Date			
Quality Manager Signature	Date	MASSTC Director Signature	Date

Corrective Action #3			
Responsible Parties			
Monitoring Period			
Follow-up (Include date and reviewer initials)			
Closeout Date			
Quality Manager Signature	Date	MASSTC Director Signature	Date

Corrective Action #4			
Responsible Parties			
Monitoring Period			
Follow-up (Include date and reviewer initials)			
Closeout Date			
Quality Manager Signature	Date	MASSTC Director Signature	Date

Upon completion, this document must be stored electronically in Sharepoint's MASSTC-Data and Records – Quality Assurance – Corrective Action Reports. Paper copies must go in the Corrective Action Reports Binder.