Title and Approval Page (A1)

Quality Assurance Project Plan for Maidford River Restoration Pilot Project Monitoring Program

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Lead Organization: Southeast New England Program Network
Partner Organization(s): Aquidneck Land Trust, ESS Group, Inc., Rhode Island Department of Environmental Management, Save The Bay, Town of Middletown
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	September 23, 2021
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Warren Hall – Town of Middletown, Town Engineer	Date
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Section A: Project Management

The following section provides information regarding the background of the Maidford River Restoration Pilot Project Monitoring Program, the tasks involved in completing the project, and the names and responsibilities of key project team members.

A4: Project Task/Organization

See **Table 1** for a list of the specific members from each organization and **Figure 1** for an organizational chart.

Table 1. Project Participants

Name	Title	Organization	Primary Responsibility
Elizabeth Scott	Rhode Island Liaison	Southeast New England Program Network (Elizabeth Scott Consulting)	Project Manager Also tracks all program measures
Matt Ladewig	Senior Water Resources Scientist	ESS Group, Inc.	Project Lead Collection of the following watershed condition measures: pollutant loading and species of concern.
Jeff Hershberger	Senior Hydrogeologist	ESS Group, Inc.	Project QA Officer
Warren Hall	Town Engineer	Town of Middletown	Project Partner – Collection of the following watershed condition measures: incidence of flooding and floodplain map extent.
Alex Chuman	Director of Stewardship	Aquidneck Land Trust	Project Partner – Collection of the following watershed condition measures: riparian habitat.

Name	Title	Organization	Primary Responsibility
Jane Sawyers	Supervising Environmental Scientist	Rhode Island Department of Environmental Management	Project Partner – Collection of the following watershed conditions measures: pollutant loading.
Kate McPherson	Narragansett Bay Riverkeeper	Save The Bay	Project Partner – Collection/technical advisor for the following watershed conditions measures: species of concern.
Nora Conlon	EPA Quality Assurance	US EPA	Final review and approval of QAPP
Ian Dombroski	EPA Project Officer	US EPA	Project oversight and QAPP review

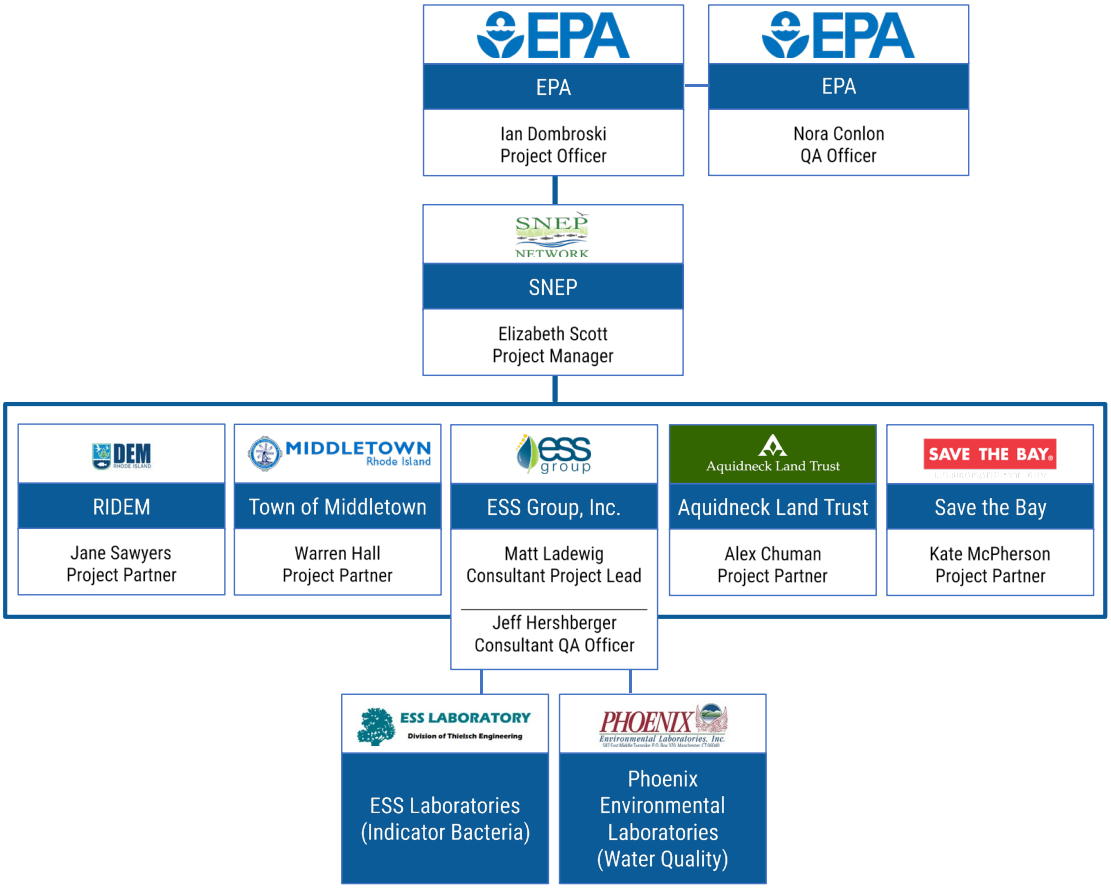


Figure 1. Project Organizational Chart

SNEP Network

The Southeast New England Program (SNEP) Network is the lead partner for the Maidford River Restoration Pilot Project (Pilot Project). To advance the project, the SNEP Network is providing consultant services in support of the design and engineering of the river and floodplain restoration project, and Network partner services to support project management, funding/financing assistance, communications, and community engagement (including direct outreach to riparian property owners). Rhode Island Liaison Elizabeth Scott (Elizabeth Scott Consulting) will provide project management services on behalf of the SNEP Network.

ESS Group, Inc.

ESS Group, Inc. (ESS) is the SNEP Network's technical consultant for development of this Quality Assurance Project Plan (QAPP). Additionally, ESS will implement the baseline streamflow and water quality portions of the monitoring program (the latter in cooperation with RIDEM) and develop the Baseline Conditions Report. ESS will also be performing the 2022 leopard frog call count survey. ESS Project Lead Matt Ladewig will oversee coordination with project partners, implementation of the field program, and development of project deliverables. ESS QA Officer Jeff Hershberger will provide quality assurance/quality control review of data deliverables.

Aquidneck Land Trust

Aquidneck Land Trust (ALT) with the Town of Middletown, co-leads the Maidford River Restoration Project and is a SNEP Network partner for the Pilot Project Monitoring Program. ALT will complete the Maidford River baseline buffer condition analyses.

Rhode Island Department of Environmental Management

The Rhode Island Department of Environmental Management (RIDEM) is a SNEP Network partner for the Pilot Project Monitoring Program. RIDEM will implement the baseline water quality portion of the monitoring program at the Berkeley Avenue, Green End Avenue, and Prospect Avenue stations (in cooperation with ESS).

Save The Bay

Save The Bay is a SNEP Network partner for the Pilot Project Monitoring Program. Save The Bay will serve as a technical advisor on the baseline surveys for species of concern and will provide assistance as requested by ALT in completing the Maidford River baseline buffer condition analyses.

Town of Middletown

The Town of Middletown (Town), with the Aquidneck Land Trust, co-leads the Maidford River Restoration Project and is a SNEP Network partner for the Pilot Project Monitoring Program. The Town will lead collection of the following watershed condition measures: incidence of flooding and floodplain map extent.

EPA

EPA funds the Southeast New England Program, which was launched in 2012 to find, test, and adopt a variety of new and innovative approaches to build resilient

ecosystems and connect communities through action, collaboration, innovation, and seed funding.

EPA is also responsible for review and approval of this Quality Assurance Project Plan (QAPP).

A5: Problem Definition/Background

The Maidford River Restoration Project is one of two pilot projects underway by the Southeast New England Program Network. SNEP Network partners are helping the Town of Middletown and Aquidneck Land Trust to advance use of nature-based solutions to mitigate flooding and restore water quality and habitat. The project entails an initiative to restore the river's riparian buffer along its length, as well as a river and floodplain restoration component in one reach of the river. To advance the project, the SNEP Network is providing consultant services in support of the design and engineering of the river and floodplain restoration project, and Network partner services to support project management, funding/financing assistance, communications, and community engagement (including direct outreach to riparian property owners). The project's goals are to mitigate flooding of local roads (Berkeley Avenue and Berkeley Avenue Extension) and farm fields, and to improve water quality and both riparian and aquatic habitat.

The Maidford River is located on Aquidneck Island in lower Narragansett Bay. Its watershed drains the southeastern corner of Middletown, which like most of Aquidneck Island has a long history of active agriculture (**Figure 2**). It is a highly-altered, small low-gradient coastal stream. The watershed is predominantly composed of agricultural and residential land uses. (**Table 2**).

Table 2. Maidford River Watershed Facts

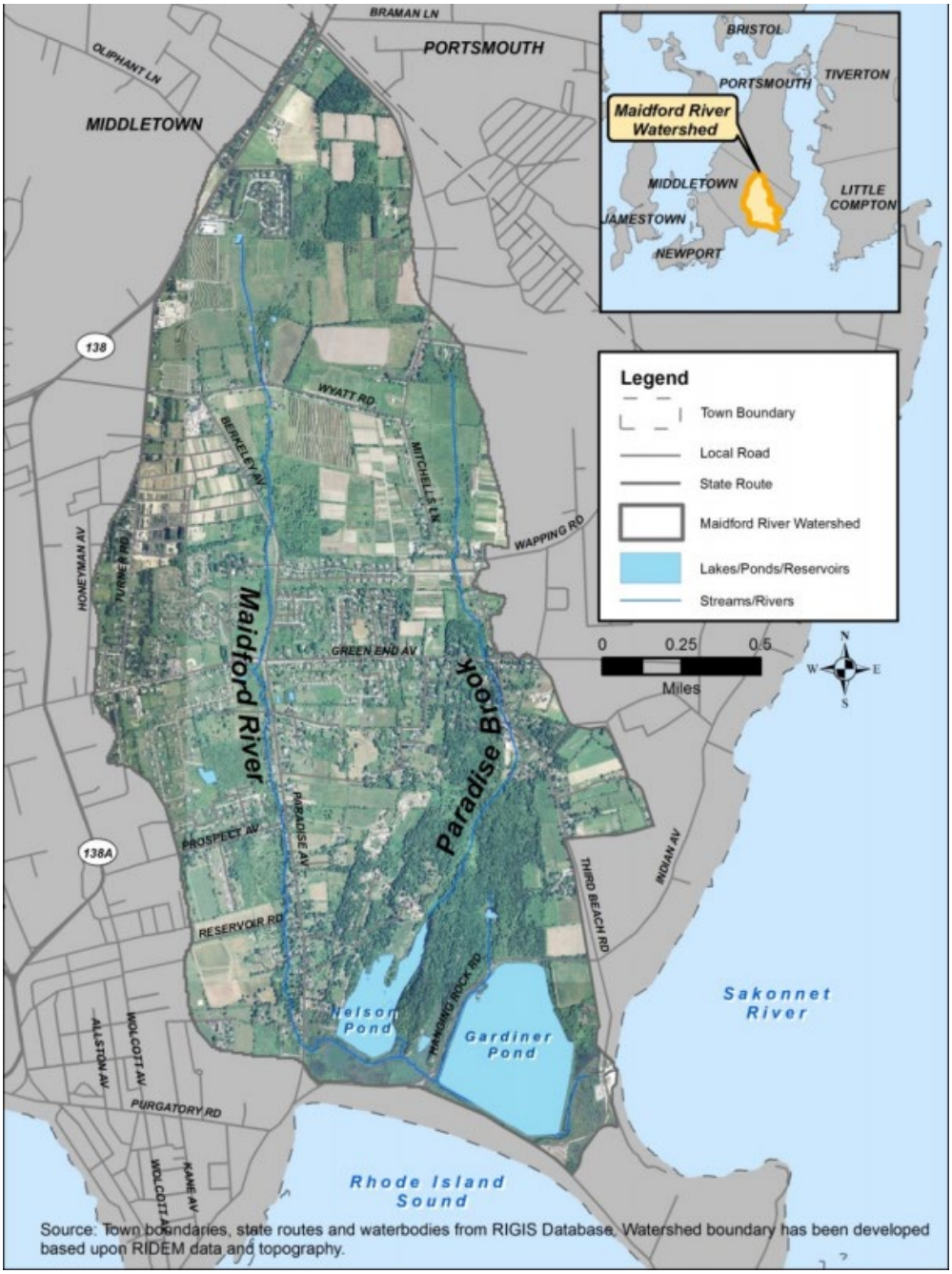
Measure	Value
Area	1,821 acres (2.85 square miles)
Length	4.6 miles
Elevation Drop (Headwaters to Mouth)	262 ft
Impervious Cover	10.7%
Major Land Uses	Agriculture: 40% Residential: 27% Forest: 16% Other: 17%
Population	1,787

Adapted from Maidford River Watershed Assessment & BMP Design (Fuss & O'Neill, 2017).

Water Resources Concerns

An underlying condition contributing to water resources concerns and affecting the Maidford River's ecological integrity is the channelization, straightening, and culverting that has occurred over decades. These alterations, along with development that has occurred near the river, severely impact natural stream processes. The river experiences both flashy stream flows and periods of de-watering and disconnected flows (in the upper portion). It routinely spills out of the river channel flooding farm fields, lawns and roads within the reach bound by the Berkeley Avenue culvert and Green End Avenue. Here, flooding of Berkeley Avenue and Berkeley Avenue Extension may occur several times a year during which time the Town of Middletown (Town) must close Berkeley Avenue to traffic, diverting public safety vehicles departing from the Fire Station on Wyatt Road from traveling south on Berkeley Avenue, and creating a public safety concern for homes and neighborhoods located in this part of town. This area is identified as A/AE FEMA Flood Zone on the Town's Flood Hazard Map. The flooding of roads, farm fields and lawns contributes to the observed increase in total suspended solids, phosphorus, and bacteria concentrations and loads documented by RIDEM (2017) and URI (Frazar et al. 2019).

The SNEP Network Pilot Project is focused on advancing nature-based solutions on the river's upper reach from its headwaters to the Newport Water Supply Diversion, defined by the RIDEM's assessment unit extending from the river's headwaters to the Newport Water Supply Diversion (RI0007035R-02A). The river's water quality is impacted by the watershed's agricultural and suburban land uses, the frequent flooding of farm fields and lawns, and scouring of riverbank along its channel with higher flows, as evidenced by bank sloughing and undercut streambanks in various locations. RIDEM has identified this upper reach of the Maidford as impaired for Total Phosphorus, Turbidity, Fecal Coliform, Benthic-Macroinvertebrate Bioassessments, and Lead.



Source: Maidford River Watershed Assessment & BMP Design (Fuss & O'Neill 2015).

Figure 2. Map of Maidford River Watershed

The Maidford River is the principal source of water to Nelson and Gardiner Ponds, two of nine surface water reservoirs owned by the City of Newport Water Division (NWD). While some households have their own wells, the system of reservoirs is the sole source of public drinking water for Aquidneck Island's communities. Newport Water Division operates a concrete diversion structure at the lower end of the upper

reach which diverts flow into Gardiner and/or Nelson Paradise Ponds. RIDEM has determined that the Maidford River's sediment and nutrient laden waters contribute to cyanobacteria blooms and related water quality impairments (Total Phosphorus and Total Organic Carbon) in these reservoirs. The impaired quality of these and other Newport water supply reservoirs necessitates advanced treatment for drinking purposes, the first of its kind in New England. The reservoirs are the subject of the draft Total Maximum Daily Load, prepared by RIDEM to address Total Phosphorus and Total Organic Carbon impairments (http://www.dem.ri.gov/programs/benviron/water/quality/rest/pdfs/tmdl-newport-reservoirs.pdf).

The lower Maidford River supports a small but important 43.5 acre saltmarsh adjacent to the river's outflow to the Sakonnet River. The river's poor water quality also contributes to degraded habitat conditions in the saltmarsh which, in combination with high water levels created by poor circulation and sea level rise, has led to reduced productivity of the Saltmarsh Sparrow (*Ammospiza caudacuta*), a species of high conservation concern (USFWS 2020). Lastly, the river's elevated bacteria levels potentially threaten closures of a coastal beach (Third Beach) and shellfish harvesting in the vicinity of the river's outlet into the Sakonnet River.

Project Description

The Maidford River Restoration Project, initiated in November 2019, complements ongoing efforts by the Town and ALT by supporting implementation of recommendations from prior watershed plans to restore the river's floodplain and riparian buffer, where feasible. With funding from EPA's Southeast New England Program, SNEP Network partners (Save The Bay, Throwe Environmental, NE Environmental Finance Center, and Elizabeth Scott Consulting) with consultant assistance from Fuss & O'Neill have been working with the Town, ALT, and Eastern Rhode Island Conservation District (ERICD) to advance riparian buffer restoration and use of nature-based solutions to mitigate flooding, improve water quality, and restore riparian and aquatic habitat. The Maidford River Restoration Pilot Project boundary extends from the river's headwaters to the Newport Water Supply Diversion, and the river and floodplain restoration project area (Pilot Project Area) is bounded upstream by the Berkeley Avenue culvert and downstream by the intersection with Green End Avenue.

Riparian Buffer Restoration

SNEP Network partner, Save The Bay is leading efforts to restore the river's riparian buffers to a natural vegetated condition along the length of the river from its headwaters to the Newport Water Supply Diversion where feasible through:

- Direct mail to all riparian property owners offering assistance from planning to planting to restore the buffer.

- On-site assistance to property owners by trusted local partners (ALT, Save The Bay, ERICD) to discuss options to restore riparian buffers. Five property owners have been provided assistance, to date.
- Development of the Riparian Buffer Guidance posted on the SNEP Network website (www.snepnetwork.org).

River and Floodplain Restoration

Guided by the project's objectives to mitigate flooding, improve water quality and habitat, the Pilot Project Team has developed a conceptual design which entails re-alignment and deepening of the reach extending from Berkeley Ave culvert to Green End Road, addition of meanders, creation of flood benches and floodplain, construction of diversion berms, replacement of one (and possibly two culverts), and raising of Berkeley Avenue (to optimize flood mitigation benefits). The conceptual design was presented to the property owners who have provided verbal endorsement for the project to advance to 30% design and have agreed to have wetland edges on their property verified by RIDEM as a preliminary step in the project design. Upon agreement from the property owners and Town and the procurement of additional funding, the Pilot Project will advance to 60% design and permitting.

Environmental Outcomes and Expected Benefits

Expected environmental improvements resulting from implementation of the selected restoration alternative and restoration of riparian buffers include the following:

- Reduced flooding and increased stream corridor stability
- Reduced frequency and duration of flooding of Berkeley Avenue and Berkeley Ave Extension
- Reduced occurrences of flooding affecting other infrastructure, residential properties, and farm fields
- Reduced flood hazard risk area (identified as A/AE FEMA Flood Zone)
- Unchanged or reduced peak flows downstream of Green End Avenue
- Reduced flashiness of river downstream of Green End Avenue
- More stable stream channel and reduced bank scour

Habitat Improvements

Expected habitat improvements resulting from implementation of the selected restoration alternative and restoration of riparian buffers include the following:

- Increased area of riparian buffer restored and/or maintained in a more complex naturally vegetated condition
- Improved aquatic community composition
- Improved aquatic habitat
- Protection of any state-listed species of concern, potentially including Leopard Frog (*Lithobates pipiens*)

Water Quality Improvements

- Reduced pollutant concentrations (total phosphorus, total suspended solids)
- Reduced sediment and nutrient loads from Pilot Project Area and to sensitive receiving waters, including Gardiner and Nelson Ponds, Sachuest Marsh and Sakonnet River

Project Measures

The SNEP Network has developed a set of measures to evaluate the impact of the Pilot Project on the condition of the watershed (Watershed Condition Measures). These include water quality, habitat, and flooding measures, each of which is more specifically described in Section A6.

Additionally, the SNEP Network has developed measures to evaluate the community's capacity to advance a watershed scale project (Program Measures). These are also described in more detail in Section A6.

The purposes of the measures are to:

- Collect data that helps us understand the benefit of the project(s)
- Align measures with EPA SNEP Program and its 5-year Strategy
- Select measures that can be practically collected (use of volunteers and partnerships)
- Build capacity at local level to collect data
- Engage partner agencies, community members, and volunteers in the data collection (where feasible)

A6: Project Task Descriptions

An overview of project tasks and schedule is presented in **Table 3**.

Table 3. Project Tasks and Schedule

Task	Deliverable	Timeline	Relevant Details/Comments
1. Develop QAPP	Approved QAPP	September 2021	Requires approval by EPA
2. Document Baseline Watershed Conditions	Baseline Streamflow, Water Quality, and Biological Data Report	June 2022	Data report specific to data collected by ESS
	Baseline Conditions Report	September 2022	Presents watershed condition measures collected by all project partners
3. Document Post-Implementation Watershed Conditions	Post-Implementation Conditions Report	Approximately one year following implementation	Presents watershed condition measures collected by all project partners
4. Document Program Measure Outcomes	Program Measure Tracking Sheet	Approximately one year following implementation	Tallies totals for all program measures over the course of the Pilot Project

Task 1: QAPP Development

This QAPP describes the quality management system and procedures, as well as the roles and responsibilities of the Project Team. The QAPP provides an overview of the project and quality assurance related to data used for the project.

The Project Manager, Elizabeth Scott, or her designee will be responsible for maintenance and distribution of the approved QAPP. The QAPP will be provided electronically as needed.

Task 2: Document Baseline Watershed Conditions

The Watershed Condition Measures to be documented during baseline watershed conditions are as follows:

A. Annualized Phosphorus, Nitrogen, and Sediment Loads

B. Area of Riparian Buffer Habitat

C. Annual Number of Road Closures Due to Flooding

D. Area Designated as A/AE Flood Zone

E. Presence/Absence of Species of Concern (Leopard Frog)

The approach for documenting each of these is detailed in the following sections:

Measure A. Annualized Phosphorus, Nitrogen, and Sediment Loads

Documentation of this measure will involve the collection of water quality and stream discharge data to be collected by RIDEM and ESS, as further explained below. Further details are provided for each component in the following sections:

Water Quality.

Water quality samples will be collected at each of the three Pilot Project monitoring locations during wet and dry weather flow events. Wet weather events will be defined as those occurring during a storm that produces at least 0.25" of precipitation. Dry weather events will be defined as those occurring after at least three days with 0.25" or less of precipitation. Wet and dry weather determinations will be based primarily on precipitation measured at Newport State Airport, which is located near the Maidford River watershed (less than half a mile to the northwest) and is close enough to provide accurate quantitative precipitation data for this project. However, publicly accessible personal weather stations or Project Partner observations in or near the watershed may also provide useful supplemental data to help understand how precipitation intensity or duration varied during wet weather events.

During wet weather events, samples will be targeted for the rising limb, peak flow, and receding limb of the hydrograph. Sampling will target the following key parameters and analytes:

- Field-measured parameters
    - Water temperature
    - Dissolved oxygen (% saturation and concentration)
    - Turbidity
    - pH
    - Conductivity/specific conductance
- Laboratory analytes
    - Total phosphorus
    - Dissolved phosphorus
    - Total suspended solids
    - Total nitrogen (total Kjeldahl nitrogen and nitrate/nitrite-nitrogen)

- Ammonia
- Enterococci

Although SNEP's focus is anticipated to be on wet weather water quality, dry weather water quality data will also be collected to provide a measure of water quality during baseflow conditions.

Water quality sampling conducted by RIDEM will be completed in accordance with a separate QAPP (RIDEM 2021). Otherwise, sampling and analysis will be conducted in accordance with the attached SOPs (**Appendix A and Appendix B**).

Discharge.

To collect continuous instream water level data, a submerged water level and temperature logger (Solinst Levelogger 5 or similar) will be installed at each of the three stream monitoring stations. These will be programmed to collect data at the same time interval. A barologger (Solinst Barologger) will be deployed out of the water in a single watershed location to collect barometric data at the same time interval. This will allow for correction of raw water level data from the unvented loggers to account for varying atmospheric pressure over time.

Data will be downloaded from the dataloggers an average of once every 10 weeks over the course of the study, including one verification download event shortly after logger deployment.

Water level data (stage) can be converted to discharge where the relationship between the two variables has been established. To establish this relationship (known as a rating curve), in situ measurements of streamflow must be collected over a range of water levels.

Therefore, depth, width, and current velocity measurements will be collected across a representative cross section for each of the three monitoring locations in accordance with SOPs (**Appendix A**). At least 11 streamflow measurements will be collected during wet and dry weather events at each cross section. Velocity will be measured using a current meter where water depth allows. A time-of-travel method will be used in cases where flow is perceptible but too shallow to accommodate an instrument.

Once a rating curve has been finalized for each cross section, the curve equation will be used to convert the continuous stage data to discharge. The total phosphorus, TSS, and total nitrogen water quality results will then be applied to the discharge record to calculate loads of each over the course of the Pilot Project Monitoring Program.

Measure B. Area of Riparian Buffer Habitat

ALT will use high resolution aerial imagery and ground-checking to assess the condition of a 200 ft buffer around the Maidford River, as further detailed below:

Area of River Assessed

Assessment of the river will begin at the headwaters at its northernmost points, those being the northern branches adjacent to Newport Vineyards and through land of Newport National Golf Club in Middletown. The study area will follow the river south until it ceases at the Newport Water diversion structure just west of Paradise Avenue in Middletown. Total distance of streambed, which includes the river itself and tributaries covers 2.5 miles. The river south of the diversion structure will not be assessed as this area does not feed the Aquidneck Island drinking water supply and is outside of the study area.

A 200-foot buffer will be assessed on either side of the stream bed. The total area of such a buffer along this section of the stream is approximately 144 acres.

Data/Software Used

- Rhode Island currently has 3-inch pixel resolution aerial flyover imagery available for free to the public. Aerials are published as an image service that can be accessed via ArcGIS Online . This has been flown 3 times a year in different seasons beginning in 2018 to present (Spring 2021).
- To determine the stream buffer assessment area, the Freshwater Rivers and Streams (1:5,000) data layer available from the state GIS database , modified and corrected to the aerial imagery to accurately follow the Maidford River, will be used.
- The latest version of ESRI ArcMap or ArcGIS Pro will be used for the assessment. All assessment will be completed in RI State Plane (NAD_1983_StatePlane_Rhode_Island_FIPS_3800_Feet). All analysis will be completed in feet/acres.

Assessment Workflow

- Using the corrected stream layer for the Maidford River, a 200 ft buffer will be generated using ArcGIS around the stream to designate the assessment area
- The buffer will be overlaid on to aerial imagery from 2020 (spring, winter, fall) and 2021 (spring). Spring 2021 imagery will be used as a base layer with 2020 imagery used as necessary if the Spring 2021 imagery was unclear in any way. Land cover will then be digitized within the stream buffer area according to the categories listed below. Digitizing involves manually tracing the points, lines, or polygon features of interest over an aerial image (in this case) to create a vector file that can be measured. Toggling between the seasonal data in each year will

help ensure accuracy of classification of the land cover. Land cover will be categorized into the following categories:

- Water Bodies (ponds, standing water)
- Hayfield
- Active Farmland
- Residential and/or Commercial Lawn
- Impervious Surface (such as roads, driveways, and structures)
- Natural Vegetation (vegetated wetlands, meadow, forest, scrub, etc)

These categories were chosen because they represent the primary land covers in the watershed and are easily distinguished between one another on the aerial imagery. Each land cover has a distinct look and the resolution of the aerials allows for easy decision-making when classifying land cover. While digitization in such a manner is typically a labor intensive process, the relatively small linear length of the stream combined with the high resolution aerial imagery available make such a process achievable and will ensure the greatest level of accuracy. Performing the analysis in this way also enables the most up-to-date assessment as the high-resolution aerials are current. A sample area of the stream assessed is provided in **Figure 3**.

- For a more generalized analysis, the six land cover categories can be further summarized and grouped into the following categories:

Classification	Grouping
Natural/Vegetated	Water Bodies Natural Vegetated
Farmland/Hayfield	Active Farmland Hayfield
Degraded	Impervious Residential and Commercial Lawn

- Once the buffer is assessed, the area of each category will be tabulated in GIS and a visually appealing series of maps of the conditions will be completed.
- This analysis can be replicated in any subsequent year there is aerial flyover data available, ideally with similar resolution aerial imagery. After completion in subsequent years, the area and stream length restored could be overlaid with this 2020-2021 analysis and re-calculated to determine changes in buffer condition.

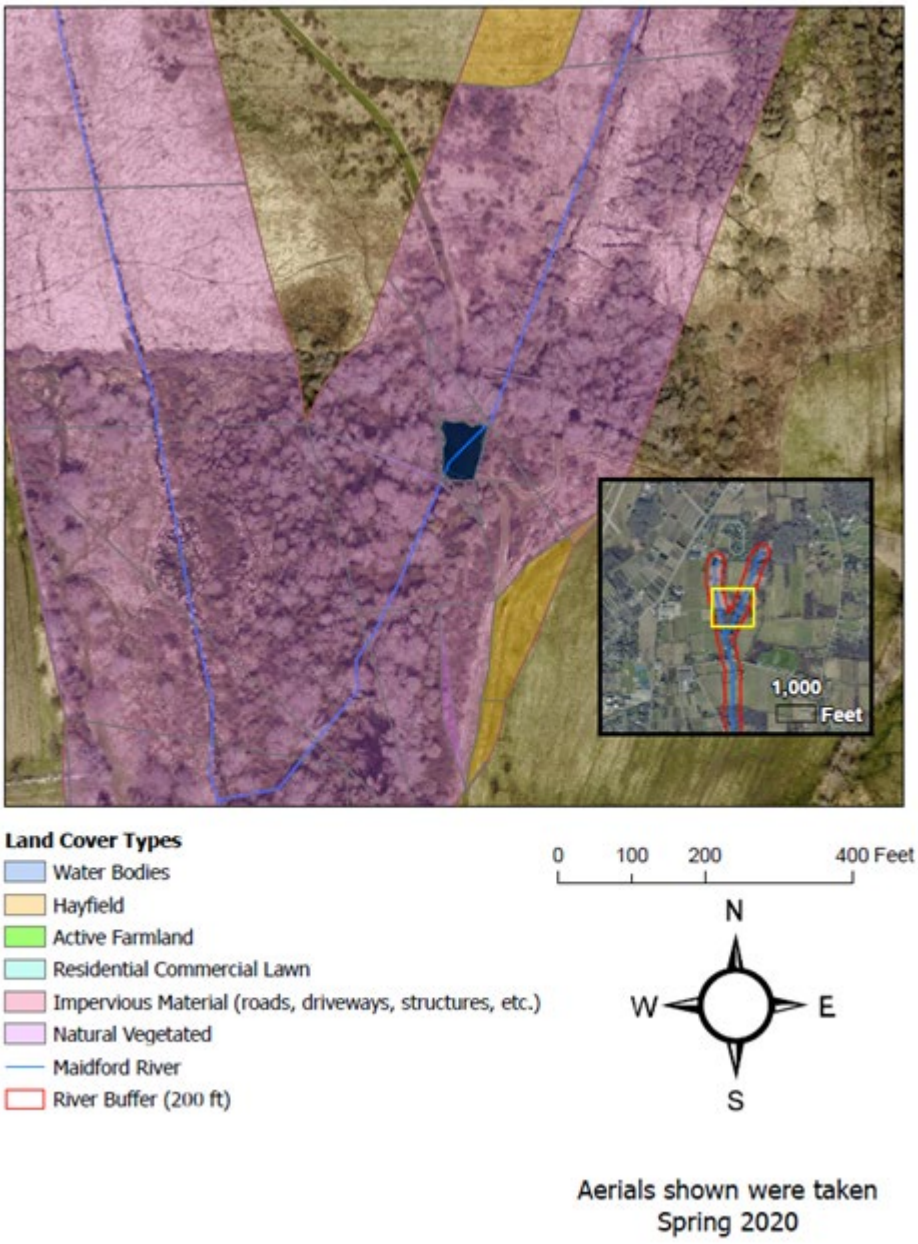


Figure 3. Example of Riparian Buffer Assessment Map Derived from Aerial Imagery

Measure C. Annual Number of Road Closures Due to Flooding

Each road closure event at Berkeley Avenue, Berkeley Avenue Extension, or Green End Avenue due to flooding of the Maidford River will be documented. Documentation will include the period of closure, a description of the extent of flooding, and photographs of the flooding event, where possible. The Town will track information in the Town of Middletown Record of Flooding Spreadsheet

Road closure events will be tallied on an annual basis over the course of the Pilot Project Monitoring Program (**Appendix C**).

Measure D. Area Designated as A/AE Flood Zone

The Pilot Project Monitoring Program will identify A/AE Zone floodplain areas during both the Baseline and Post-Implementation Monitoring periods, using FEMA's National Flood Hazard Layer. Any changes in the extent of areas designated as A/AE Zone will be measured and described.

Measure E. Presence/Absence of Species of Concern (Leopard Frog Call Count Surveys)

Anuran (frog) call count surveys will be used to monitor for the presence of state-listed northern leopard frogs (*Lithobates pipiens*) in and near the project area. Anuran call count survey methods will be adapted from Paton et al. (2003) and the North American Amphibian Monitoring Program (NAAMP).

Surveys will be conducted once every two weeks during the Baseline and Post-Implementation Monitoring periods, beginning the first week of April and ending the last week of June. Each event will begin approximately 30 minutes after sunset and end prior to midnight.

To avoid detecting individuals at multiple stations, monitoring stations will be spaced at least 173 meters (570 feet) apart within the monitoring area. Stations will be located to provide the greatest possible coverage of the study area without overlap. However, the actual layout of the stations will depend on access conditions and landowner permission. The order in which stations are visited will be reversed in alternating survey nights to avoid time bias of results.

During each survey, anuran calling activity will be monitored for a set period at each survey station. This duration will be determined by the final number of survey stations; however, it is currently anticipated that no less than five minutes of survey will occur at each station. For each anuran species heard, the observer will record the maximum calling index during one-minute intervals. The calling index ranges from 0 (no calls heard) to 3 (individuals too numerous to count, chorus is constant and overlapping).

Additionally, the following environmental abiotic data will also be recorded for each survey:

- Air temperature
- Wind speed
- Estimated cloud cover
- Days since the last rainfall

- Current precipitation
- Ambient noise level, as measured by qualitative index ranging from 1 (slightly affecting sampling – e.g., one car passing nearby) to 4 (profoundly affecting sampling – e.g., construction noise)

Although these surveys will be focused on determining the presence of northern leopard frog, they will also provide a basis for measuring changes in the anuran community at-large over the course of the Pilot Project. Additionally, incidental observations of other wildlife will also be collected to help document the presence/absence of species using the Maidford River and adjacent riparian habitats.

Task 3: Document Post-Implementation Watershed Conditions

The process for documentation of post-implementation watershed conditions will generally attempt to replicate that used to document baseline conditions under Task 2. This will help to ensure that the post-implementation data collected are comparable to those collected during baseline conditions.

Task 4: Document Program Measure Outcomes

In addition to the quantitative data generated to document baseline and post-implementation watershed conditions, the Project Manager or her designee will also document program measure outcomes using a tracking spreadsheet. The Program Measures to be documented are as follows:

1. Number of Partner Organizations on the Project
2. Number of Monitoring Programs Established or Underway
3. Number of Workshops, Webinars, and Stakeholder Meetings Given
4. Number of Volunteers Participating in the River's Restoration
5. Dollars Leveraged from Partners on the Project

These are included in the Program Measure Tracking Spreadsheet (**Appendix D**).

A7: Quality Objectives and Criteria

Description of Data Acceptance

The data quality objectives (DQOs) for this project will be met if the data collected can be used to assess or determine the value or change in value of each of the Watershed Condition Measures and Program Measures.

DQOs can be described in terms of the following performance criteria: precision, accuracy and bias, representativeness, comparability, and completeness.

Precision:

Water Quality. Water quality laboratory sample and field measurement results will be sufficiently precise to ensure reliable detection of each analyte or parameter within its expected environmental range.

Field duplicates will be collected at a 10% rate and submitted as blind duplicates (i.e., not labeled as "duplicate") for quality control purposes. Laboratory duplicates will be tested in accordance with the requirements of the laboratory SOPs and/or Quality Manual/Quality Assurance Manual.

Precision will be measured using Relative Percent Difference (RPD) between duplicates. The precision objective is presented in **Table 4**.

Discharge. Permanent cross section transects will be established at each station to maximize precision over the course of the Pilot Project Monitoring Program. This will avoid errors due to varying transect angles or positioning. Additionally, duplicate discharge measurements will be made at a 10% rate. The duplicate will consist of repeat measurements at the same cross section immediately following the first measurement.

Other Watershed Condition Data. The Project Manager or her designee will check other data to ensure that they are of sufficient precision to meet data quality objectives.

Accuracy and Bias:

Water Quality. Water quality laboratory sample and field measurement results will be sufficiently accurate and unbiased to ensure results that reflect the true value of the analyte or parameter being measured.

Laboratory accuracy will be measured using Percent Recovery of one or more of the following quality control samples: laboratory control samples (LCS) and matrix spikes (MS). Method blanks and other checks on accuracy may also be used. The accuracy objective for this project is presented in **Table 4**.

Stream pollutant loading calculations may be affected by hysteresis effects, which result in higher loading rates during the rising limb than at identical discharges during the falling limb of the hydrograph. To avoid this bias, water quality samples will be collected at three points in the hydrograph for each wet weather event. One sample will be collected during the rising limb of the hydrograph, one will be collected near the event peak flow, and one will be collected during the falling limb.

Discharge. Discharge measurements will be sufficiently accurate and unbiased to allow for the development of rating curves for each station and estimation of pollutant loads. To avoid bias generated by collection of discharge data primarily at low flows with few measurements at high flows, wet weather discharge data will be collected at three different stages during each event.

Continuous Water Level Loggers. Water level logger raw data will be corrected for atmospheric pressure using a barometric logger that is set to collect and log data at the same interval. Additionally, water level at each logger location will be manually measured by the field crew during each visit. The directly measured depth will be compared against the logged depth at the nearest timestamp. Additionally, the measurements collected at both deployment and download will be used to assess any "drift" in the logger readings. This will serve as an accuracy check.

Table 4. Laboratory Performance Objectives

Analyte	Units	Laboratory Minimum Detection Limit	Expected Range	Precision Objective	Accuracy Objective
Phosphorus (Total and Dissolved)	mg/L	0.01	0.01 – 2.0	±20%	±15%
Total Suspended Solids	mg/L	5.0	5.0-20	±20%	±15%
Total Kjeldahl Nitrogen	mg/L	0.1	0.2-2.0	±20%	±1%
Nitrate-Nitrogen	mg/L	0.01	0.02-1.0	±20%	±10%
Nitrite-Nitrogen	mg/L	0.01	0.005-0.02	±20%	±10%
Ammonia-Nitrogen	mg/L	0.05	0.05-0.5	±20%	±10%
Enterococci	MPN/100 mL	1	10-24,200+	±30%	N/A

Other Watershed Condition Data. To ensure accuracy of the buffer assessment, a single ALT staff person trained in GIS and land use classification will complete the digitizing with a second person performing an accuracy check of the designated classification areas within the stream buffer. ALT staff members completing the analysis have experience assessing aerial imagery and have extensive on-the-ground knowledge of the watershed and land uses within the watershed.

This on-the-ground knowledge is based on past monitoring visits of conservation easements on both public and private lands on the stream, frequent drive-bys of the project area, research for the Maidford River Conservation Plan, and walking the full length of the Maidford River streambed in 2016.

In cases where the land cover is still unclear based on the aerials or existing knowledge, ALT or a project partner will perform a ground accuracy check. Ground checking in this watershed is done fairly easily as the majority of the stream is visible from public roadways and properties ALT has access to.

The Project Manager or her designee will also check all other data to ensure that they are not unreasonably biased and are of sufficient accuracy to meet data quality objectives.

Representativeness:

Water Quality: Water quality samples will be collected at stations selected to be representative of incoming flows to the Pilot Project Area, outgoing flows from the Pilot Project Area, and flows located well downstream of the Pilot Project Area. The same wet and dry weather sampling criteria will be used for each sampling event.

Sample results are expected to be representative provided that samples are collected in accordance with relevant SOPs and analytical laboratory sampling protocols, including use of appropriate containers, collection of sufficient volume, use of preservatives as directed, and adherence to sample hold times. Deviations from the established protocols will be noted on the chain-of-custody form or field books by field personnel and included in data reporting.

Discharge. Measurement stations have been selected to be representative of incoming flows to the Pilot Project Area, outgoing flows from the Pilot Project Area, and flows located well downstream of the Pilot Project Area.

Discharge measurements will spread out over the course of the study to be representative of the range of flows expected to occur. This will include measurements of low flows experienced during dry weather conditions, as well as higher flows that are generated by wet weather conditions in this runoff-driven system.

Continuous Water Level Loggers. Water level logger locations have been selected to be representative of incoming flows to the Pilot Project Area, outgoing flows from the Pilot Project Area, and flows located well downstream of the Pilot Project Area. Water levels will be continuously recorded at 15-minute intervals to allow for collection of data representative of the rapid changes in discharge within this flashy system.

Other Watershed Condition Data. The Project Manager or her designee will check other data to ensure that they are adequately representative of the targeted measure and will meet data quality objectives.

Comparability:

Water Quality. Water quality samples collected from different monitoring locations and events will be sufficiently comparable provided that samples are collected,

stored, and transported in accordance with relevant SOPs and the laboratory sampling protocols. Additionally, the same wet and dry weather sampling criteria will be used for each sampling event. Deviations from the established protocols will be noted on the chain-of-custody form by field personnel and included in data reporting.

Discharge. Discharge measurements collected from different monitoring locations and events will be sufficiently comparable provided that measurements are completed in accordance with relevant SOPs and are collected at the same channel cross section during each visit to each measurement location. If possible, the same equipment should be used to measure velocity during all visits. In cases where bank failure or other substantial geomorphic change has occurred that would alter the suitability of the location for continued monitoring, a replacement cross section that is suitable for measurement (per the SOP), should be established as closely as possible to the original cross section.

Continuous Water Level Loggers. Water level logger measurements collected from different monitoring locations and events will be sufficiently comparable provided that the devices used have similar specifications and are deployed in a similar manner among the various monitoring locations.

Other Watershed Condition Data. The Project Manager or her designee will check other data to ensure that they are sufficiently comparable to meet data quality objectives.

Completeness:

Water Quality. Data will be considered complete if water quality samples are collected from each of the proposed stations during each anticipated sampling event and analyzed for the intended analytes. If data cannot be obtained for an event (e.g., due to inadequate flow), this will be reported to the Project Manager or their designee in a timely fashion to determine whether this is likely to affect completeness of the dataset in a substantive way. If the effect is determined to be substantive, the Project Manager or their designee will consider alternatives to improve completeness of the dataset (e.g., through additional sampling event[s]).

Discharge. Discharge measurements will be considered complete if collected from each of the proposed stations during each anticipated measurement event. If data cannot be obtained for an event (e.g., due to inadequate flow), this will be reported to the Project Manager or their designee in a timely fashion to determine whether this is likely to affect completeness of the dataset in a manner that will substantively impact the development of a representative rating curve. If the effect is determined to be substantive, the Project Manager or their designee will consider alternatives to improve completeness of the dataset (e.g., through additional sampling event[s]).

Continuous Water Level Loggers. Water level logger measurements will be considered complete if no more than 10% of the anticipated measurements are

missing from the data record for each of the monitoring stations over the course of deployment.

Other Watershed Condition Data. The Project Manager or her designee will check other data to ensure that they are sufficiently complete to meet data quality objectives.

A8: Special Trainings/Certification

No special training or certification courses were specifically attended in preparation for this project.

ESS personnel involved with this project have received formal education and in-house training in necessary field methods, including water quality sampling, hydrologic investigations, taxonomic identification, and GIS analysis from previous academic study, routine participation at conferences, as well as during in-house training associated with a variety of similar projects throughout New England. Additional in-house training will be provided for ESS field staff, as necessary, to meet project requirements.

ALT staff completing the riparian buffer condition assessment will be trained in GIS and land use classification. ALT staff members completing the analysis have experience assessing aerial imagery and have extensive on-the-ground knowledge of the watershed and land uses within the watershed.

At a minimum, all personnel involved in field sample collection or data analysis will be provided with a copy of this QAPP and attachments.

Analytical laboratories shall maintain certification through the Rhode Island Department of Health for analysis of non-potable water microbiology and/or non-potable water inorganic chemistry, as applicable to the analytes processed under this QAPP.

A9: Documentation and Records

The Project Manager for the SNEP Network will maintain the current approved version of the QAPP. Only the Project Manager or their designee will update or amend the QAPP. All versions of the QAPP will be retained in the Project Manager or her designee's electronic files for this project for a period of at least three years after the end of the project.

Should any change to the QAPP be necessary, the current version of the QAPP will be saved in the Project Manager or designee's electronic archive folder for the project before any changes are made, and the updated version will be assigned the next consecutive version number. The Project Manager or designee will distribute the new version of the QAPP to the individuals listed on the Title and Approvals

page in Section A1 of this QAPP for approval before distributing the new version of the QAPP to the individuals identified on the Distribution List in Section A3.

The following data will be included as part of the Baseline and Post-Implementation Conditions Reports:

- Chain-of-custody forms, laboratory analytical reports, and electronic data deliverables for water quality samples
- Continuous datalogger outputs
- Rating curves developed for each discharge monitoring station
- GIS data created to support development of map figures or spatial analysis
- GIS data and map(s) of digitized land cover in the 200 ft buffer and other documentation used to calculate buffer condition length by category
- Tracking spreadsheet containing information about dates and extent of road flooding
- Calculations of mapped floodplain area
- Anuran survey point count results
- Tracking spreadsheet containing tallies of Program Measure data

Because this project is expected to extend over several years, it is likely that changes in key personnel or organizations will occur. To ensure consistency in methodology and data quality, new personnel or organizations who implement any component covered by this document will be required to review and acknowledge the QAPP. The Project Manager will maintain signed copies of the QAPP acknowledgment form (**Appendix E**) for each new entity.

The Project Manager or their designee will review all forms and reports for accuracy and completeness and contact the party responsible for completing the form or report in the event that any omissions or suspected inaccuracies are found. In the event that any corrections are made, they will be written on the form, dated and initialed by the party responsible for completing the form, or a new report will be issued with a new version number and date. All project data will be retained in the Project Manager's electronic files for this project for a period of at least three years after the end of the project.

Section B: Data Generation and Acquisition

The description of data generation and acquisition contained in this QAPP was developed with guidance from EPA's Requirements for Quality Assurance Project Plans (QA/R-5).

B1: Sampling Process Design (Experimental Design)

Rationale for Design.

Water Quality and Discharge. Water quality sampling and water level logging/discharge stations were primarily selected to bracket the Pilot Project Area. The Berkeley Avenue station is located just upstream of this area and the Green End Avenue station is located just downstream. The Prospect Avenue station is the sampling location for RIDEM's Ambient River Monitoring Program and URI's RI EPSCoR project (from 2014-2016). It was selected to provide a location to measure water quality changes reflective of Project Partners' efforts to restore riparian buffers, improve stormwater management, and encourage enhanced stewardship more broadly throughout the watershed. A map showing the approximate locations of these sampling stations is provided as **Figure 4**.

Additionally, to take advantage of ongoing water quality monitoring data being collected by RIDEM as part of the state's Ambient River Monitoring Program (at Prospect Street) and the National Water Quality Initiative (at Berkeley Avenue and Green End Avenue) under separate QAPPs (RIDEM 2021). The specific sampling stations at Berkeley Avenue, Green End Avenue, and Prospect Avenue were co-located with RIDEM's existing sampling stations. RIDEM will complete all sampling, analysis, and quality control and assurance of data collected in accordance with their approved QAPPs. They will make these water quality data available to the Project Manager to be utilized in calculating annualized loads.

Presence/Absence of Species of Concern (Leopard Frog Call Count Surveys). Sampling locations for the anuran surveys will be located in the Pilot Project Area to focus on detection of frogs within the area where habitat restoration will occur. The 570-foot spacing of monitoring stations was selected to minimize the potential overlap of detections. Additionally, the seasonal timeframe for the surveys (April to June) was selected to capture the period when leopard frogs are most likely to be detected by call.

Other Watershed Condition Data. Other data will be measured or counted directly. The locations and specific measures were selected to allow for representative tracking of watershed conditions in the Pilot Project Area or immediately downstream (e.g., incidence of flooding).

Figure 4. Water Quality and Discharge Monitoring Locations

Types and Number of Samples Required.

Water Quality. Water quality samples will be analyzed for the analytes identified in **Table 4**. Water quality sample collection will be split between dry weather and wet

weather monitoring events. The number of wet weather water quality samples to be collected is shown in **Table 5**. This table also identifies the responsible party for wet weather sample collection.

Table 5. Anticipated Number of Wet Weather Water Quality Samples*

	ESS			RIDEM		
Location	**# Visits**	**Samples/ Visit**	**Total Samples**	**# Visits**	**Samples/ Visit**	**Total Samples**
Berkeley Avenue	1	3	3	3	1	3
Green End Avenue	1	3	3	3	1	3
Prospect Avenue	3	3	9	0	0	0
Grand Total			15			6

*Reflects baseline sampling breakdown. Total number of samples anticipated to be similar for post-implementation sampling.

The number of dry weather water quality samples to be collected is shown in **Table 6**. This table also identifies the responsible party for dry weather sample collection.

Table 6. Anticipated Number of Dry Weather Water Quality Samples*

	ESS			RIDEM		
Location	**# Visits**	**Samples/ Visit**	**Total Samples**	**# Visits**	**Samples/ Visit**	**Total Samples****
Berkeley Avenue	2	1	2	0	0	0
Green End Avenue	2	1	2	0	0	0
Prospect Avenue	2	1	2	5	1	5
Grand Total			6			5

*Reflects baseline sampling breakdown. Total number of samples anticipated to be similar for post-implementation sampling.

**Three samples will include all water quality parameters and two will be bacterial indicators only

The overall split between dry weather and wet weather monitoring events anticpated over the course of the Program is identified in **Table 7**.

Table 7. Anticipated Total Number of Water Quality Monitoring Events

Location	# Dry Weather Visits	# Wet Weather Visits	Total # Visits
Berkeley Avenue	2 Baseline 2 Post	4 Baseline 4 Post	6 Baseline 6 Post
Green End Avenue	2 Baseline 2 Post	4 Baseline 4 Post	6 Baseline 6 Post
Prospect Avenue	7 Baseline* 2 Post	3 Baseline 3 Post	10 Baseline* 5 Post
Total	11 Baseline* 6 Post	11 Baseline 11 Post	22 Baseline* 17 Post

*Eight baseline (five dry and three wet weather) visits include sampling for all water quality parameters. The other two visits only include bacteria sampling. Baseline visits includes planned sampling by both RIDEM OWR and ESS.

Additionally, several water quality parameters will be measured in the field, as identified in **Table 8**. These will be measured during wet and dry weather at the frequency identified in **Table 5** and **Table 6**.

Table 8. Anticipated Water Quality Field Parameters

Parameter	Units	Expected Range
Temperature	°C	0 to 30
Dissolved Oxygen	mg/L % saturation	0 to 15 0 to 110
Turbidity	NTU	0.1 to 50
pH	SU	5.5 to 8.0
Specific Conductance	µS/cm	100 to 1,000

Discharge. Discharge measurements will be collected during dry weather and wet weather monitoring events, as identified in **Table 9**.

Table 9. Anticipated Number of Baseline Discharge Measurement Events*

Location	# Dry Weather Measurements	# Wet Weather Measurements	Total # Measurements
Berkeley Avenue	2	9	11
Green End Avenue	2	9	11
Prospect Avenue	2	9	11
Total	6	27	33

*The need for additional discharge measurements during post-implementation monitoring is currently uncertain. Whether additional discharge measurements are needed will depend on the degree of change due to restoration activities and natural events occurring between the end of the baseline monitoring period and the start of the post-implementation monitoring period.

Presence/Absence of Species of Concern (Leopard Frog Call Count Surveys). It is currently anticipated that up to seven surveys would occur during the Baseline Monitoring Program with another seven surveys completed as part of the Post-Implementation Monitoring Program. However, the number of anuran call surveys completed will depend on whether the number of individuals and species detected over the course of each field survey window (April to June). If detections decline and no new species are detected over multiple surveys, the Pilot Project Monitoring Team may elect to truncate the survey period.

Other Watershed Condition Data. Other data will be measured or counted directly. The locations and specific measures were selected to allow for representative tracking of watershed conditions in the Pilot Project Area or immediately downstream (e.g., incidence of flooding).

B2: Sampling Methods

Water Quality and Discharge. Field sampling of water quality and collection of discharge measurements will be conducted in accordance with SOPs (**Appendix A)**.

Laboratory sample containers, preservation methods, and hold times that are applicable to the analytes being sampled as part of the Pilot Project Monitoring Program are summarized in **Table 10**. Additionally, laboratory analysis will follow the SOPs provided (**Appendix B**).

Table 10. Laboratory Requirements for Collected Samples*

Analyte	Container	Hold Time	Preservative	Comments
Phosphorus (Total and Dissolved)	50 mL plastic	28 days	H_2SO_4	Dissolved phosphorus field-filtered with 45-micron filter
Total Suspended Solids	100 mL plastic	7 days	None	
Total Kjeldahl Nitrogen	250 mL plastic	28 days	H_2SO_4	
Nitrate-Nitrogen	50 mL plastic	48 hours	None	
Nitrite-Nitrogen	50 mL plastic	48 hours	None	
Ammonia-Nitrogen	500 mL plastic	28 days	H_2SO_4	
Enterococci	125 mL sterile	24 hours	None	

*Applies to samples collected by ESS. RIDEM samples will be collected using a comparable approach but under a separate QAPP.

B3: Sample Handling and Custody

Water Quality. Water quality samples will be collected in accordance with relevant SOPs for water quality field sampling and handling (**Appendix A and Appendix B)**. Sample collection personnel will wear previously unused nitrile gloves when collecting and handling samples. Bacteria samples will be collected using aseptic techniques to avoid contamination of the sample matrix or containers.

Sample collection personnel will use permanent marker to label all sample bottles with sample ID, date and time sampled, and initials of the sampler, at a minimum. Samples will be placed on ice or in a dedicated refrigerator to maintain sample temperature between 0°C and 4°C until transfer to the laboratory. Samples will be transferred to the laboratory prior to exceeding the hold time for any analyte.

All samples transferred to laboratory custody will be accompanied by a completed chain-of-custody (COC) form specific to the laboratory that will be performing the analyses (**Appendix A and Appendix B**). Personnel transferring samples to the laboratory will sign the COC and indicate the date and time of sample transfer.

B4: Analytical Methods

Laboratory analytical methods are summarized in **Table 11**. SOPs for each method are provided in **Appendix B**.

Table 11. Anticipated Water Quality Analyses*

Analyte	Laboratory	Analytical Method(s)
Phosphorus (Total and Dissolved)	Phoenix	SM4500PE
Total Suspended Solids	Phoenix	SM2540 D
Total Kjeldahl Nitrogen	Phoenix	351.1
Nitrate-Nitrogen	Phoenix	353.2
Nitrite-Nitrogen	Phoenix	353.2
Ammonia-Nitrogen	Phoenix	350.1
Enterococci	ESS/BAL	Enterolert

*Applies to samples collected by ESS. RIDEM samples will be collected using a comparable approach but under a separate QAPP.

B5: Quality Control

Laboratory analytical SOPs and quality control information relevant to each analyte are provided in **Appendix B**.

Additionally, field duplicates will be collected for water quality samples, field water quality measurements, and discharge measurements at an overall 10% rate. These field duplicates will be used expressly for quantifying the RPD between samples collected or measurements taken at the same location. To avoid biasing the dataset, duplicate sample data will not be included in the final data analyses.

The minimum field duplicate sampling rate for each laboratory analyte is summarized in **Table 12**.

Table 12. Water Quality Sample Field Duplicates*

Analyte	Minimum # Field Duplicate Samples
Phosphorus (Total and Dissolved)	2 Baseline 2 Post
Total Suspended Solids	2 Baseline 2 Post
Total Kjeldahl Nitrogen	2 Baseline 2 Post
Nitrate-Nitrogen	2 Baseline 2 Post
Nitrite-Nitrogen	2 Baseline 2 Post
Ammonia-Nitrogen	2 Baseline 2 Post
Enterococci	2 Baseline 2 Post

*Applies to samples collected explicitly under this QAPP. RIDEM will also collect field duplicate samples using a comparable approach but under a separate QAPP.

B6: Instrument/Equipment Testing, Inspection and Maintenance

Laboratory analytical equipment testing, inspection, and maintenance protocols are provided in **Appendix B**.

Field instrumentation will be cleaned, stored, and serviced in accordance with manufacturer recommendations.

B7: Instrument/Equipment Calibration and Frequency

Laboratory analytical equipment calibration protocols are provided in **Appendix B**.

Field instrumentation will be calibrated and/or checked in accordance with manufacturer recommendations.

B8: Inspection/Acceptance of Supplies and Consumables

Sample collection personnel will complete a bottle order request form with each analytical laboratory to help ensure that the number, type, and condition of sample containers are appropriate for the analyses to be conducted. Additionally, sample

collection personnel will also be responsible for visually inspecting sample containers and verifying their cleanliness, integrity, and consistency with this QAPP before collecting samples for laboratory analysis. Sample containers that are visibly damaged, soiled, or inappropriate for the required analysis will not be used for sample storage.

B9: Non-Direct Measurements (i.e. secondary data)

Geospatial data is the main type of secondary data that will be used to support the Pilot Project Monitoring Program. To ensure that the geospatial data are of sufficient quality to support the analyses identified in this QAPP, only data obtained through official government sources or otherwise meeting government standards will be used.

Publicly available geospatial information, including high resolution orthophotography, will be obtained to evaluate the extent of riparian buffer cover types in the Pilot Project Area during both the Baseline and Post-Implementation Monitoring periods. Geospatial data files may also be used to develop map figures of the Pilot Project Area or the greater Maidford River watershed. This information will be obtained through RIGIS, which is Rhode Island's official geospatial data hub. Geospatial data available through RIGIS meet minimum metadata standards (http://data.rigis.org/assets/docs/Metadata_Resources/RIGIS_Minimum_Metadata_Standard.pdf).

Additionally, the Pilot Project Monitoring Program will make use of the National Flood Hazard Layer to identify A/AE Zone floodplain areas during both the Baseline and Post-Implementation Monitoring periods. Geospatial data obtained will meet FEMA's Policy Standards for Flood Risk Analysis and Mapping (https://www.fema.gov/media-collection/standards-flood-risk-analysis-and-mapping-public-review).

B10: Data Management

Field Data Collection and Records

Field notes, forms, and other documentation (e.g., photographs) will be saved for each round of field sampling or survey. A copy of each will be retained by the Project Manager or her designee for at least three years following project completion in the electronic records for the project.

Information to be recorded for each laboratory analytical sample will include the following: the individual performing the sample collection, date and time of sample collection, and laboratory analysis requested. This information will be recorded on a laboratory COC form (**Appendix B**). All original COC forms will accompany samples for laboratory analysis and a copy of each will be retained by the Project Manager or

her designee for at least three years following project completion in the electronic records for the project.

Raw water level datalogger files will be transferred from each logger to a laptop computer, tablet, or other media storage device during each field visit. These raw files will be transferred to a permanent storage location upon return to the office and maintained in their original version, in addition to any corrected files incorporating atmospheric corrections or other data processing techniques. A copy of each will be retained by the Project Manager or her designee for at least three years following project completion in the electronic records for the project.

Analytical Laboratory Data

All electronic data deliverables, laboratory reports, case narratives, QC reports, email correspondence, or other information generated by an analytical laboratory will be retained by the Project Manager or her designee for at least three years following project completion in the electronic records for the project. This will include preliminary or draft data, in addition to any revisions.

Section C: Assessment and Oversight

This section addresses the activities for assessing the effectiveness of the implementation of the quality assurance and quality control activities. The purpose of the assessment is to ensure that QAPP is implemented as described.

C1: Assessments and Response Actions

The Project Manager or her designee will provide oversight for field data collection effort to ensure that protocols described in this QAPP are being followed.

The Project Manager or her designee will review each batch of reported data to ensure that appropriate protocols are being followed and data are within the accepted range for each parameter or analyte. Any "outlier" data discovered will be identified and potential sources of error will be assessed to determine possible causes and avoid or minimize those going forward.

The Project Manager or her designee may also request data audits or reviews to assess conformance and compliance to the QAPP.

C2: Reports to Management

The Project Manager or her designee will receive at least quarterly updates related to the Annualized Phosphorus, Nitrogen, and Sediment Loads and Presence/Absence of Species of Concern (Leopard Frog Call Surveys) Watershed Condition Measures during periods of active monitoring. These updates will include information on project progress and results of data quality assessments, as well as recommendations for resolving any issues associated with data quality.

The Project Manager or her designee will receive updates for all other Watershed Condition Measures on at least an annual basis during periods of active monitoring.

Section D: Data Validation and Usability

This section addresses the QA activities that occur after the data collection of the project has been completed. Implementation of these elements ensures that the data conform to the specified criteria and achieve the project objectives.

D1: Data Review, Verification, and Validation

Water Quality. Laboratories will use the control limits set forth in Section B6 to accept analytical data generated under conditions that meet the control limits for instrument precision and accuracy and reject or qualify analytical data generated under conditions that do not meet the control limits for instrument precision and accuracy.

The Project Manager or her designee will review analytical reports to verify and validate the completeness and reasonableness of laboratory results using the record of samples submitted for analysis on the completed COC forms and the anticipated environmental range of values for the selected parameter and sample matrix.

Data will be considered complete when analytical values are reported for all analyses requested for all samples submitted to the laboratory. Data will be considered reasonable when all values measured for the sample and all values generated by the same analytical procedure fall within the expected range of values for that sample or that parameter.

Data that do not meet completeness or reasonableness criteria will be evaluated for rejection (if unusable) or qualification (if usable).

Other Watershed Condition Data. Other data will be reviewed by the Project Manager or her designee to verify that the values or observations reported are consistent with the expected range for the Pilot Project Area and that the data are complete and otherwise reasonable.

Data that do not meet completeness or reasonableness criteria will be evaluated for rejection (if unusable) or qualification (if usable).

D2: Verification and Validation of Methods

Water Quality. The Project Manager or her designee will be responsible for verifying that all water quality samples collected are completely and accurately represented on COC forms prior to transfer to the laboratory for analysis. If there are data gaps or inaccuracies on COC forms, the forms will be corrected immediately and the revised information will be communicated to the laboratory.

Laboratory technicians are responsible for validating instrument calibration by measuring the calibration standards for the selected parameter after accepting a

new calibration and recalibrating if the measurements do not meet established control limits for accuracy and precision.

Analytical data provided by the laboratories will also be reviewed and validated by the Project Manager or her designee to provide information on whether data are acceptable for use in developing project conclusions and deliverables. Results of the verification and validation processes will be addressed in project deliverables. The Project Manager or her designee will be responsible for final review of deliverables for completeness and adherence to method and quality control requirements.

Other Watershed Condition Data. Other data will be reviewed by the Project Manager or her designee to verify that the values or observations reported are consistent with the expected range for the Pilot Project Area and that the data are complete and otherwise reasonable.

Data that do not meet completeness or reasonableness criteria will be evaluated for rejection (if unusable) or qualification (if usable).

D3: Reconciliation with User Requirements

Deliverables will include results of quality assurance activities and/or quality control outcomes. Results will be accompanied by an assessment of the impact of quality control anomalies or departures from assumptions, if any, on the ultimate usability of the data. Data qualifiers will also be described so that users are aware of these limitations.

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Appendix A – Field Sampling SOPs

GUIDELINES FOR MEASUREMENT OF SPECIFIC CONDUCTANCE

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine calibration and operation of a variety of specific conductance meters. This SOG document also addresses estimation of total dissolved solids (TDS) and salinity by direct measurement of specific conductance (specific methods and capabilities for these parameters are outlined in the manufacturer's individual instrument manuals). This SOG is designed to be consistent with EPA Method 120.1 and Standard Method 2510 B which address specific conductance measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

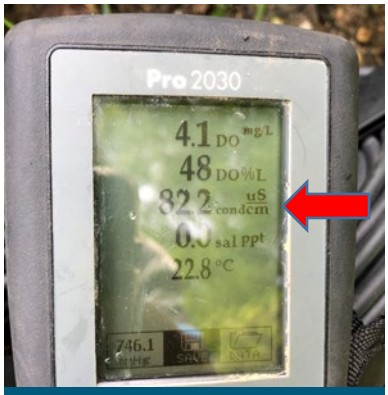
2.0 RESPONSIBILITIES

- The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.
- The analyst is responsible for verifying that the specific conductance meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Specific conductance meter
- Specific conductance meter manufacturer's instruction manual
- Deionized water
- Conductivity standard at concentration that approximates anticipated range of sample concentrations
- Lint-free tissues
- Calibration sheets or logbook
- Laboratory or field data sheets or logbooks



Handheld YSI 2030 meter, red arrow showing where the specific conductance is measured

4.0 METHODS

4.1 Sample Handling, Preservation, and General Measurement Procedures

- Specific conductance measurements should be taken in situ or soon after sample collection since temperature changes, precipitation reactions, and absorption of carbon from the air can affect the specific conductance. If specific conductance measurements cannot be taken immediately (within 24 hours), samples should be filtered through a 0.45 µm filter, stored at 4°C and analyzed within 28 days.
- Report results as specific conductance, µmhos/cm or µS/cm at 25°C.

- Secondary standards may be purchased as a solution from commercial vendors. These standards should not be used after their expiration dates as provided by the manufacturer. An expiration date of one year from date of purchase should be used if the expiration date is worn or missing.

4.2. Calibration and Measurement Procedures

- The specific conductance meter must be calibrated (or the calibration checked) before any analyses are performed.
- Set up the instrument according to the manufacturer's instructions.
- Rinse the probe with deionized water prior to use. If necessary, dry with a lint-free tissue or cloth.
- Follow the manufacturer's recommendations for appropriate calibration receptacle and depth of immersion.
- Record the stabilized specific conductance reading of the standard and the temperature. Enter the calibration mode (according to manufacturer's instructions) and change the value on the primary display to match the value of the calibration standard. The meter may be adjusted to ±20% from the default setting. If the measurement differs by more than ±20%, the probe should be cleaned, serviced, or replaced as needed.
- An additional check may be performed, if required by the project plan, by placing the probe into an additional standard. This standard should be from a different source than the standard used for the initial calibration. This standard should read within 5% of the true value.

Use of YSI handheld device and probe

- Verify the calibration at least once a month or whenever the instrument has been moved from freshwater to saltwater environments or vice versa. Recalibrate or service the instrument, as needed, if the check value is not within 15% of the true (calibration standard) value.

4.3. Troubleshooting Information

If there are any performance problems with any of the specific conductance meters which result in inability to achieve the acceptance criteria presented in Section 5.0 or the project-specific acceptance criteria, consult the appropriate section of the meter instruction manual for troubleshooting procedures. If the problem persists, consult the manufacturer's customer service department immediately for further guidance.

4.4. Maintenance

- Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.
- The probe must be stored and maintained according to the manufacturer's instructions.

5.0 QUALITY CONTROL

- The meter must be calibrated (or the calibration checked) before sampling, and will not be used for sample determinations of specific conductance unless the initial check standard value is within 5% of the true value.

- Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within 10%.

6.0 DOCUMENTATION

- Meter calibration, temperature check, and maintenance information will be recorded in a calibration log. Specific conductance data may be recorded on the appropriate laboratory or field data sheets or logbooks.
- Calibration documentation should be maintained in a thorough and consistent manner. At a minimum, the following information should be recorded:
  - Date and time of calibration
  - Person performing the measurement
  - Instrument identification number/model
  - Expiration dates and batch numbers for all standards
  - Reading for standard before and after meter adjustment
  - Readings for all continuing calibration checks
  - Temperature of standards (corrected for any difference with reference thermometer)
  - Comments
- Documentation for recorded data must include a minimum of the following:
  - Date and time of analysis
  - Person performing the measurement
  - Sample identification/station location
  - Temperature (corrected for any difference with reference thermometer) and conductance of sample (including units and duplicate measurements).
  - Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform specific conductance measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that specific conductance measurements be taken in the field by, or in the presence of, personnel that are qualified under the certification program.

GUIDELINES FOR MEASUREMENT OF DISSOLVED OXYGEN

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine measurement of dissolved oxygen using a polarographic sensor-equipped dissolved oxygen meter with a digital read-out (e.g., YSI Pro2030 Dissolved Oxygen, Conductivity, Salinity Instrument). Measurements are made in accordance with methods that address dissolved oxygen measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

2.0 RESPONSIBILITIES

- The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.
- The analyst is responsible for verifying that the dissolved oxygen measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

Handheld YSI 2030 meter, Red arrow showing were the dissolved oxygen is measured

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Dissolved oxygen meter with digital read-out device
- Manufacturer's instruction manual for the instrument
- Manufacturer’s recommended operating solution and replacement membranes or caps
- Laboratory or field data sheets or logbooks

4.0 METHODS

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate dissolved oxygen measurements, samples should be analyzed in situ. Measurements in flowing waters should be made in relatively turbulent free areas. Measurements in standing waters may require gentle probe agitation to create water movement around the probe (check instrument manual to confirm).

4.2. Calibration and Measurement Procedures

To accurately calibrate some dissolved oxygen meters, it may be necessary to know the altitude of the region in which you are located and the approximate salinity of the water you will be analyzing. Fresh water has a salinity of approximately zero. Seawater has an approximate salinity of 35 practical salinity units

(psu). If uncertain, measure salinity first with an appropriate device. The instructions below are applicable to the YSI Model 55; for other instruments, consult the instruction manual.

- Ensure that the sponge inside the instrument's calibration chamber is wet then insert the probe into the chamber. Turn the instrument on and wait for readings to stabilize (as long as 15 minutes, depending on the model).
- To calibrate, enter the calibration menu by pressing and releasing both the up and down arrow keys at the same time. Enter the altitude (in hundreds of feet) at the prompt by using the arrow keys to increase or decrease the altitude (example: 12 = 1,200 feet). Press enter when correct altitude is shown.
- The meter should display CAL in the lower left of the display with the calibration value in the lower right of the display and the current D.O. reading (before calibration) should be on the main display. Once the D.O. reading is stable, press ENTER. Enter the salinity at the prompt by using the arrow keys. Press ENTER when finished and the instrument will return to normal operation.
- Calibration should be performed at a temperature within ± 10°C of the sample temperature. Recalibrate every 15 samples and whenever the unit is turned on.
- If calibration is out of range, erratic readings occur, bubbles appear, or if the membrane becomes damaged, wrinkled, or fouled refill the membrane solution and/or replace the membrane, per the manufacturer's manual.

Use of YSI handheld device and probe

- Avoid contact with environments containing substances that may attack the probe materials (e.g. acids, caustics, and strong solvents).

4.3. Troubleshooting Information

If there are any performance problems with the dissolved oxygen-measuring device, consult the appropriate section of the instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4. Maintenance

Instrument maintenance for meter-type dissolved oxygen measuring devices should be performed according to the procedures and frequencies required by the manufacturer. Rinsing the probe with distilled or deionized water and preventing exposure of the membrane to drying is typically all that is required on a day-to-day basis.

5.0 QUALITY CONTROL

Duplicate measurements of a single sample should be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within ± 0.2 mg/L.

6.0 DOCUMENTATION

All dissolved oxygen meter calibration, checks, and maintenance information will be recorded in a calibration logbook. Dissolved oxygen data may be recorded on the appropriate field data sheets or field books.

- Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:
    - Date and time of calibration
    - Person performing the measurement
    - Instrument identification number/model
    - Readings for all continuing calibration checks
    - Comments
- Documentation for recorded data must include a minimum of the following:
    - Date and time of analysis
    - Person performing the measurement
    - Sample identification/station location
    - Dissolved oxygen, both in mg/L and percent saturation and temperature of sample (including units and duplicate measurements)
    - Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform dissolved oxygen measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that dissolved oxygen measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

GUIDELINES FOR MEASUREMENT OF PH

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine calibration and operation of a variety of pH field pens. Although these meters may measure additional parameters (e.g., temperature, specific conductance, etc.), this SOG addresses pH measurement only (other capabilities are outlined in the appropriate SOG and manufacturer's individual instrument manuals). This SOG is designed specifically for the measurement of pH in accordance with EPA Method 150.1 and Standard Method 4500-H B which address electrometric pH measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

2.0 RESPONSIBILITIES

- The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.
- The analyst is responsible for verifying that the pH meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

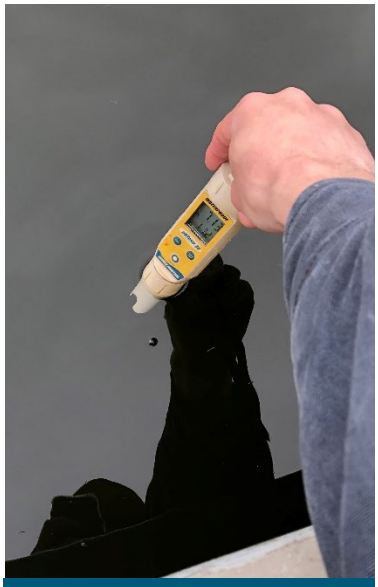
The following materials are necessary for this procedure:

- pH meter
- pH meter manufacturer's instruction manual
- Deionized or distilled water
- 4.0, 7.0, and 10.0 buffer solutions
- Lint-free tissues
- Mild detergent
- Manufacturer’s recommended storage solution
- Manufacturer’s recommended cleaning solution
- Field data sheet or logbook
- Calibration sheet or logbook

4.0 METHODS

4.1 Sample Handling, Preservation, and General Measurement Procedures

- To achieve accurate pH measurements, samples should be analyzed immediately in the field, or as soon as possible after collection. Sample should be measured *in situ* or collected in plastic or glass containers.
- As temperature can affect the pH measurements obtained, both the pH and the temperature of the sample must be recorded, unless the meter is capable of automatic temperature correction (ATC).
- Primary standard buffer salts available from NIST can be purchased and are necessary for situations where extreme accuracy is required. Secondary standard buffers may be purchased as a solution from commercial vendors and are recommended for routine use. Buffers should not be used after their expiration dates as provided by the manufacturer. An expiration date of one year should be used if the manufacturer does not supply an expiration date or if the buffers are prepared from pH powder pillows, etc.
- Keep the probe elevated off the bottom to avoid disturbing sediments. Allow readings to fully stabilize before recording the pH measurement. This may take several minutes, especially if the pH is drastically different from the last reading or the bulb has been allowed to dry out between readings.
- Rinse the electrode with deionized or distilled water between samples and wipe gently, if needed, with a lint-free tissue. If a more thorough cleaning is required, use a mild detergent (e.g., dish soap) or the manufacturer's recommended cleaning solution.
- Store the probe in the manufacturer's recommended storage solution or, if this is not available, tap water. Do not use distilled or deionized water for storage purposes.



Handheld pH meter

4.2. Calibration and Measurement Procedures

- The pH meter should be checked weekly before any analyses are performed. Otherwise, the meter should be checked or calibrated at the frequency specified in the project plan.
- Calibration should include a minimum of one point but ideally, a two point calibration that brackets the expected pH of the samples to be measured is desirable. Calibration measurements should be recorded in the calibration logbook.
- Choose either 7.0 and 10.0 (high range) or 4.0 and 7.0 (low range) buffers, whichever will bracket the expected sample range. Pour each buffer into a clean glass beaker. The volume should be sufficient to fully submerse the pH bulb and thermistor. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Measure and record the temperatures of the buffers using a calibrated thermometer or automatic temperature compensation (ATC).
- Follow the manufacturer's calibration instructions.
- Once calibration is complete, discard the buffer and rinse the beaker (and stirring bar, if used) thoroughly with distilled or deionized water.

- An additional check may be performed, if required by the project plan, by placing the electrode into an additional buffer solution. This buffer should be from a different source than the buffers used for the initial calibration. This buffer should read within ±0.2 pH units of the buffer's true pH value.
- Recalibrate the instrument if any of the following apply:
  - the check value varies more than 0.2 pH units from the true value
  - the expected pH of the sampled water body is outside the current calibration range
  - readings are erratic or do not stabilize
  - the instrument has just been cleaned or otherwise disturbed for maintenance

4.3. Troubleshooting Information

If there are any instrument performance problems that result in the inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for troubleshooting procedures. If the problem persists, consult the manufacturer's customer service department immediately for further guidance.

4.4. Maintenance

- Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.
- The electrode should be stored and maintained according to the manufacturer's instructions.

5.0 QUALITY CONTROL

- Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within ±0.2 pH units.

6.0 DOCUMENTATION

- All pH meter calibration, temperature check, and maintenance information will be recorded in a calibration logbook.
- pH data may be recorded on the appropriate laboratory or field data sheets or logbooks.
- Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:
  - Date and time of calibration
  - Person performing the measurement
  - Instrument identification number/model
  - Expiration dates and batch numbers for all buffer solutions
  - Reading for pH 7.0 buffer before and after meter adjustment
  - Reading for pH 4.0 or 10.0 buffer before and after meter adjustment
  - Readings for all continuing calibration checks
  - Temperature of buffers (corrected for any difference with reference thermometer), including units
  - Comments

- Documentation for recorded data must include a minimum of the following:
    - Date and time of analysis
    - Person performing the measurement
    - Sample identification/station location
    - Temperature and pH of sample (including units and duplicate measurements)
    - Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform pH measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

GUIDELINES FOR MEASURING STREAMFLOW

1.0 INTRODUCTION

These guidelines provide instructions for the field measurement of flow rate in bodies of running water.

Descriptions of two field techniques are provided.

The first, called the time of travel method, is simple and does not require expensive or specialized equipment. This is most appropriate for rapid streamflow assessments where order of magnitude accuracy is acceptable or water depth is too low for the accurate measurement using a velocity meter.

The second method requires the use of a current meter, which is the preferred method where discharge measurements are being used to develop at-a-station rating curves and water depth is sufficient for measurement.

Additionally, these guidelines provide This method of calculating streamflow involves determining the cross-sectional area of the stream and measuring the average time it takes for a neutrally buoyant object to travel a known distance.

2.0 REQUIRED MATERIALS

The following materials are necessary for the measuring streamflow:

- Measuring stick to measure stream depth (folding stick is recommended)
- Flexible tape measure (longer than the width of the stream)
- Field data sheet, logbook, or tablet with electronic data sheet

If using a velocity meter, the following additional materials are also required:

- Swoffer Model 2100 current velocity meter (or similar)
- Calibrated wading rod

If using the time of travel method, the following additional materials are also required.

- A neutrally buoyant float
- Stopwatch (built-in app on most smartphones)
- Net (to catch the float)

3.0 METHODS

3.1 Choosing a Cross Section

Measuring stream depth using a folding yard stick.

- Select an appropriate stream cross section. The location selected should be straight (no bends), and free of obstructions. Unobstructed runs are ideal.
- Identify the left and right banks of the stream. When working in streams, left and right are relative to the mean flow direction. Therefore, the left bank will be to one's left when facing downstream but to one's right when facing upstream.
- To assure consistency of measurements and allow for easier comparison of data across time, flow should be measured at the same cross section of the stream during all visits. Include descriptions of site landmarks in field notes, and/or take photos of measurement locations. If site conditions allow, install permanent cross section markers, such as stakes or rebar.
- If a staff gauge is present near the stream measurement location, record the staff gauge depth during each visit.

3.2. Divide the Channel into Subsections

- Establish a transect by stretching the measuring tape across the stream, perpendicular to the channel axis. Secure each end of the tape to the stream banks so that the tape is taut.
- Take a minimum of four photographs, including one each facing upstream, the left bank, downstream, and the right bank.
- Starting with the left edge of water, measure width and stream depth at no less than three locations (stations) within the steam channel. This is the minimum number of stations and most streams will require more than three measurements to accurately calculate discharge.
- The area between each vertical station represents a channel subsection.

3.3. Measuring Velocity

3.3.1 Time-of-Travel Method

- To measure travel time, time how long it takes for a neutrally buoyant object (a float) to travel a known distance. Suitable objects should float, but sit very low in the water to minimize influence from wind, and can be untethered or tethered (methods adapted from EPA, 2012a described below).

- Suitable floats include:
    - citrus fruits or pieces of citrus peel
    - cheese puffs
    - small sponge rubber balls
    - small sticks or bits of vegetation
- Always face upstream when taking velocity measurements. Stand far enough downstream that stream velocity is not affected in the location being measured.
- Surface velocity is generally greater than depth-averaged velocity, so a correction factor (0.8 for rocky-bottom streams, 0.9 for muddy-bottom streams) is applied to float travel times (see Section 3.3, EPA 2012b)
- Untethered floats should be biodegradable, or a second person equipped with a net should be stationed downstream of the sampling reach to retrieve the float(s).
- Hold the measuring stick above the water surface, perpendicular to the cross section. Release the untethered float somewhat upstream of the end of the measuring stick to allow the float to reach full flow velocity. Using a stopwatch, time how long it takes for the float to travel a known distance (3 ft is recommended for most streams but longer distances may be appropriate where velocity is high). Repeat this process three times to obtain an average time to travel at one station before proceeding to the next station.

3.3.2 Depth-Averaged Current Meter Method

- Set the current meter to average measurements over at least a three second period. Longer periods may be used if appropriate to conditions.
- Always face upstream when taking velocity measurements. Stand far enough downstream that stream velocity is not affected in the location being measured.
- Carefully place the wading rod in the flow until the base is firmly on the stream bottom.
- Orient the current meter perpendicular to the cross section transect.
- Ensure that the wading rod is straight up and down (not angled).
- Hold the wading rod steady while adjusting the calibrated height of current meter to match the measured depth. This will allow collection of measurements that are reflective of depth-averaged velocity.
- Once at least three seconds have passed, view the reading from the current meter. Allow at least three readings to occur before recording. This will prevent erroneous data due to averaging of measurements from the set up process.

3.4. Calculating Flow

- The following equation is used to calculate flow using the time-of-travel method):

$$Q = (ACL)/T$$

  - Q = stream discharge
  - A = cross sectional area
  - L = distance traveled by the float
  - C = correction factor (0.8 for rough streambeds, 0.9 for smooth streambeds)
  - T = average time of travel (seconds)

The following equation is used to calculate flow using the depth-averaged current meter method:

$$Q = AV$$

  - Q = stream discharge
  - A = cross sectional area
  - V = velocity at 60% depth

4.0 DOCUMENTATION

Record streamflow data on field sheets, field notebooks, or electronic tablets. Any unanticipated site-specific information, which requires deviation from the above guidelines should also be recorded. In addition to recording the required discharge data, field notes for streamflow measurement should include a minimum of the following:

- Name or initials of person conducting the measurement
- Discharge measurement method used
- Site ID or name
- Date and time of streamflow measurement
- Environmental conditions (wind, temperature, etc.)
- Other relevant observations about site conditions
- Photographic evidence of streamflow and site conditions is also useful for verification of relative stream stage and flow from different visits, as well as any environmental factors that may have influenced data collection.

5.0 REFERENCES

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GUIDELINES FOR THE MEASUREMENT OF TURBIDITY

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine measurement of turbidity using a nephelometric turbidity meter with a digital read-out device (e.g., LaMotte 2020we Turbidimeter). Measurements are made in accordance with EPA Method 180.1 that addresses nephelometeric turbidity measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

2.0 RESPONSIBILITIES

- The analyst is responsible for verifying that the turbidity measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.
- The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Turbidity meter with digital read-out device
- Manufacturer's instruction manual for the instrument
- Turbidity tubes/cuvettes
- Mild detergent
- Lint-free cloth
- Distilled water
- Nephelometric Turbidity Unit (NTU) calibration standards
- Laboratory or field data sheets or logbooks

Example of a sample cuvette

4.0 METHODS

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate turbidity measurements, samples should be analyzed immediately upon collection (preferably within 15 minutes). Samples should be collected in glass or plastic containers.

4.2. Calibration and Measurement Procedures

- Select a turbidity standard in the range of the samples to be tested (typically 0.00 NTU, 1.00 NTU or 10.0 NTU) or as recommended by the manufacturer. Fill a turbidity tube or cuvette with the standard, cap, and wipe the tube with the clean lint-free cloth.

- Place the sample into the turbidity meter such that the indexing arrow or line on the turbidity tube is aligned with the indexing arrow or notch on the meter face. Close the lid and press the “READ” button. If the displayed value is not the same as the value of the standard (within 10%), continue with the calibration procedure.
- Follow the calibration procedures outlined by the manufacturer’s manual.
- Verify the calibration every 15 samples and at the end of the day. Recalibrate the instrument if the check value varies more than 10% from the true value.
- Rinse cuvettes with deionized or distilled water and wiped gently with a lint-free tissue between sample analysis.
- Recalibrate the instrument with the appropriate NTU standard if the standard is not of the same order of magnitude as the samples being tested.
- The meter must be re-calibrated following any maintenance activities and prior to the next use.
- Record the turbidity reading to the nearest 0.01 NTU for measurements less than 11 NTU and to the nearest 0.1 for measurements greater than 11 NTU but less than 110 NTU. For values greater than 110 NTU record to the nearest 1 NTU.

Turbidity meter

4.3. Troubleshooting Information

If there are any performance problems with any of the meter-type turbidity measuring devices, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4. Maintenance

Instrument maintenance for meter-type turbidity measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

The turbidity measuring tubes will, at a minimum, be checked against NTU calibration standards at the frequency stated in Section 4.2. This verification procedure will be performed in accordance with the manufacturer’s manual.

Duplicate measurements of a single sample will be performed at the frequency required in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within + 5% for readings below 10 NTU and + 10% for readings above 100 NTU.

6.0 DOCUMENTATION

All turbidity meter calibration, checks, and maintenance information will be recorded on the daily calibration sheet or logbook. Turbidity data may be recorded on the appropriate laboratory or field data sheets or logbooks.

Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all standard solutions
- Reading for each standard before and after meter adjustment
- Readings for all continuing calibration checks
- Comments

Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Person performing the measurement
- Sample identification/station location
- Turbidity of sample (including units and any duplicate measurements)
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform turbidity measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

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GUIDELINES FOR USE OF WATER LEVEL DATALOGGERS IN SURFACE WATER

1.0 INTRODUCTION

These guidelines provide basic instructions for programming, deploying and downloading data from electronic dataloggers which are used for long-term water level monitoring and monitoring of other water quality parameters (e.g., temperature, conductivity). The methods outlined below are intended to (1) standardize datalogger monitoring of water levels; (2) ensure that data collected represent field conditions as accurately as possible; (3) provide guidance for the secure transfer and storage of data; and (4) assure proper field measurements and documentation.

2.0 REQUIRED MATERIALS

The following materials are necessary:

- Datalogger or Barologger
- Rebar, metal fence post or similar
- Sledgehammer or post driver
- Computer, laptop or tablet
- Communications device
- Datalogger software
- PVC housing and hose clamps
- Braided nylon twine or wire
- Water level monitoring device (electronic water level meter, plunker, etc.)
- Tools

3.0 PROGRAMMING THE DATALOGGER

The following general procedure is followed to edit the datalogger information and program the datalogger to collect readings. Always refer to the manufacturer's instructions for datalogger programming, deployment and data downloading and correction instructions.

If the datalogger is unvented, a second datalogger or Barologger will also need to be deployed to collect barometric pressure readings to support the barometric pressure correction of the data downloaded from the unvented datalogger.

- Connect the datalogger or Barologger to the computer, laptop or tablet using the supplied communications device.
- Select the appropriate COM or USB port for the communications device.
- Open the logger programming or launching page to allow for entering the logger details and programming the device for frequency of readings.
- Check the datalogger battery life and available memory to verify that it is acceptable for the intended use. This information can also be used to determine the frequency of data downloads that will be necessary.
- Rename the datalogger using the Site location.

- Program the datalogger to collect readings for the selected parameters at the specified interval.
    - The additional datalogger or Barologger should be programmed to collect readings at the same interval.
- Disconnect the datalogger from the communications device using the software program to disengage or remove from the communications device.

4.0 DEPLOYING THE DATALOGGER

The following general procedure should be used to deploy the datalogger for collection of readings.

- An appropriate location for the installation of the datalogger should be selected within a deep portion of the water body to allow for fluctuations in the water level.
    - An additional datalogger or Barologger should be mounted in the air in an inconspicuous location such as a tree or attached to a building.
- In surface water, the datalogger should be deployed within a PVC housing, if possible, to help to dampen any fluctuations (e.g., wave action or turbulence) in the surface water surface.
- The PVC housing should be secured using hose clamps to a mounting post (typically either rebar, metal fence post or similar) that can be driven into the substrate beneath the water body.
- Prior to deployment, collect a depth to water measurement from the top of the mounting post (rebar, metal fence post or similar) and an approximate depth to the bottom of the PVC housing.
- Deploy the datalogger by placing inside the PVC housing, attaching either braided nylon twine or wire to the datalogger, and threading through the top of the PVC cap to allow for retrieval of the datalogger without disturbing the position of the PVC housing.

5.0 DOWNLOADING DATA

The following general procedure should be followed when downloading the data.

- Soon after deployment (typically within two weeks, or as soon as possible given project constraints), a verification download should be performed to verify that the datalogger was programmed correctly and is collecting data at the appropriate interval.
- Subsequent downloads can be scheduled as necessary taking into consideration the frequency of readings and the available memory and battery life of the datalogger.
- Prior to removing the datalogger to download the data, collect a depth to water measurement from the top of the mounting post or other acceptable measuring point.
- Remove the datalogger and connect to the communications device.
- Connect the communications device to the computer, laptop or tablet and open the software.
- Navigate to the data download page and proceed with downloading the collected data.

  - Certain datalogger software packages will require that the datalogging process be stopped and the datalogger will have to be reprogrammed prior to redeployment.

- If the datalogger is unvented, correct the downloaded data using the program supplied by the Vendor using the data collected from the additional datalogger or Barologger.

6.0 STREAMFLOW MEASUREMENTS

In order to convert the hydraulic head (pressure) measurements from the datalogger to streamflow, a series of streamflow measurements needs to be collected at varying stream stages or flow stages to support the development of a rating curve.

REFER TO THE GUIDELINES FOR MEASURING STREAMFLOW SOP FOR DETAILS ON THE COLLECTION OF STREAMFLOW MEASUREMENTS AND DEVELOPMENT OF A RATING CURVE.

7.0 DOCUMENTATION

Maintain field notes for the datalogger deployment and data download events including the collected field measurements that will be used to QC the data collected by the datalogger and convert the collected measurements to elevation, if applicable.

Photodocumentation of the datalogger deployment and setup should also be maintained as well as any observations during the field visits.

8.0 REFERENCES

Select references to Vendors that supply dataloggers are provided below.

Solinst, Levelogger Series Software User Guide, https://www.solinst.com/products/dataloggers-and-telemetry/3001-levelogger-series/operating-instructions/user-guide/3001-user-guide.php

In-Situ, Rugged Troll 100 and 200 and Rugged Troll BaroTroll Instruments, https://in-situ.com/pub/media/support/documents/Rugged_TROLL_Manual.pdf

Onset, HOBO U20L Water Level Logger (U20L-0x) User's Manual, https://www.onsetcomp.com/support/manuals/u20l_17153/

Appendix B – Laboratory SOPs

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
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SOP No.: 321.4500PE

Title: Phosphorus, All Forms

Scope and Application: This method covers the determination of all forms of phosphorus, including o-phosphate, organic phosphate, and total phosphorus. This method is applicable to all drinking water, surface water, ground water, industrial and domestic waste, and soil samples and their leachates. The range for this procedure is 0.010-1.0 mg/L. Samples above 1.0 mg/L can be analyzed by sample dilution prior to digestion. The limit of detection is 0.010 mg/L.

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates, and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms. These forms of phosphate arise from a variety of sources. Small amounts of certain condensed phosphates are added to some water supplies during treatment. Larger quantities of the same compounds may be added when the water is used for laundering or other cleaning, because these materials are major constituents of many commercial cleaning preparations. Phosphates are used extensively in the treatment of boiler waters. Orthophosphates applied to agricultural or residential cultivated land as fertilizers are carried into surface waters with storm runoff and to a lesser extent with melting snow. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota.
Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater, agricultural drainage, or certain industrial wastes to that water may stimulate the growth of photosynthetic aquatic micro- and macro-organisms in nuisance quantities.

I. Summary of Method

A. Ammonium molybdate and potassium antimony tartrate react in an acid medium to form a heteropoly acid (phosphomolybdic acid) which is reduced to an intensely blue-colored complex by ascorbic acid. The intensity of the blue color is directly proportional to the concentration of orthophosphate in the sample.

B. Since the above reaction only occurs with the o-phosphate form of phosphorus, pretreatment of condensed and organically bound is required for their

determination. Condensed phosphates must undergo acid hydrolysis at boiling-water temperature. Organically bound phosphates must undergo a digestion using acid, heat, and an oxidizing agent. These treatments transform the bound phosphates to o-phosphate, where they can be quantified.

C. For dissolved phosphate, filter the sample through a 0.45um filter before analysis. If only particulate phosphate is to be determined, analyze for dissolved phosphate and for total phosphate, and subtract dissolved from total.

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

1. Total Orthophosphate (P, ortho) or Total Reactive Phosphorus- inorganic phosphorus [$(PO_4)^{-3}$] in the sample as measured by the direct colorimetric analysis procedure.
2. Total Hydrolyzable Phosphorus (P, hydro) – phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus pre-determined orthophosphates. This hydrolyzable phosphorus includes polyphosphorus. [$(P_2O_7)^{-4}$, $(P_3O_{10})^{-5}$, etc.] plus some organic phosphorus.
3. Total Organic Phosphorus (P, org) – phosphorus (inorganic plus oxidizable organic) in the sample measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate.

E . Dissolved Phosphorus (P-D)- all of the phosphorus present in the filtrate of a Sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure.

1. Dissolved Orthophosphate (P-D, ortho) or Dissolved Reactive Phosphorus- as measured by the direct colorimetric analysis procedure.
2. Dissolved Hydrolyzable Phosphorus (P-D, hydro)- as measured by the sulfuric acid hydrolysis procedure and minus pre-determined dissolved orthophosphates.
3. Dissolved Organic Phosphorus (P-D, org)-as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate.

F. The following forms, when sufficient amounts of phosphorus are present in the sample to warrant such consideration, may be calculated:

1. Insoluble Phosphorus (P-I) = (P)-(P-D).
2. Insoluble orthophosphate (P-I, ortho)=(P,ortho)-(P-D,ortho).
3. Insoluble Hydrolyzable Phosphorus (P-I,hydro)=(P,hydro)-(P-D,hydro).
4. Insoluble Organic Phosphorus (P-I, org)=(P,org) – (P-D,org).

G. See Appendix B for flow chart.

II. Interferences

A. Glassware must be scrupulously cleaned, as carry over from phosphates is common. Refer to Appendix A in this SOP for the Phosphorus Glassware Cleaning Instructions.

B. Arsenate present in the sample to be tested is a positive interference, as it reacts with the molybdate reagent to produce a blue color similar to the blue formed by o-phosphate. Concentrations as low as 0.1 mg/L are enough to cause interference. By adding sodium bisulfite to the sample, arsenate is reduced to arsenious acid.

C. High concentrations of iron in a sample will give erroneously low results, as it will take away some of the reducing agent. Sodium bisulfite treatment will eliminate this interference.

D. Hexavalent chromium and nitrite interfere to give low results. Concentrations of these ions need to be at least 10.0 mg/L to interfere signifcantly.

E. Particulates may interfere with spectrophotometric analysis by absorbing light. Samples may be filtered after hydrolysis or digestion but not once the color reagent is added.

F. In the determination of orthophosphate, color can interfere. A color blank may be performed.

III. Sample Collection, Preservation and Storage

A. Samples can be collected in plastic or glass. They must be stored at 4°C until time of analysis.

B. Orthophosphate samples must be unpreserved and analyzed within 48 hours from time of collection.

C. Total Phosphorus samples must be H_2SO_4 preserved to pH<2 and analyzed within 28 days of collection.

IV. Equipment and Supplies

A. Spectophotometer, 880nm.

B. Environmental Express 54-position Hot Block with custom glass digestion vials.

C. Pro Clean Environmental Containers glass 60mL vials, P/N GV60/PCLB-72.

D. Graduated cylinder, 50mL.

E. Volumetric flasks, 50mL.

F. Class "A" volumetric pipets; assorted.

G. Hengar granules, boiling stones

V. Reagents and Standards

A. Sulfuric Acid, 5N. Dilute 139mL conc H_2SO_4 to 1000mL with DI water.

B. Potassium antimony tartrate solution. Dissolve 1.3715 g $K(SbO)C_4H_4O_6$ 1/2 H_2O in 400 mL DI water in a 500ml volumetric flask and dilute to volume. Store in a plastic, stoppered bottle.

C. Ammonium molybdate solution. Dissolve 20 g $(NH_4)_6 Mo_7O_{24}*4H_2O$ in 500ml DI water. Store in plastic, stoppered bottle.

D. Ascorbic acid solution, 0.01M. Dissolve 3.52g ascorbic acid, reagent grade, in 200mL DI water. Make fresh daily.

E. Combined color reagent. Mix 100mL 5N H_2SO_4, 10mL potassium antimony tartrate, 30mL ammonium molybdate, and 60mL ascorbic acid solution in a clean 200mL erlynmeyer flask. If more reagent is needed, increase volumes of each solution proportionally. **Add reagents in the order given, and mix after the addition of each reagent.** Combined reagent is only stable for 4 hours.

F. Sulfuric acid, 11N. Slowly add 300mL conc. H_2SO_4 to 600mL DI water. Cool in a water bath, and dilute to 1 liter. Alternatively, make from 18N (1:1) H_2SO_4 by diluting 600mL to 1 liter of DI water.

G. Ammonium persulfate $(NH_4)_2\ S_2O_8$ solid, reagent grade. Potassium persulfate may be used as an oxidizing reagent.

H. Sodium bisulfite solution. Dissolve 5.2 g of $NaHSO_3$ in l00mL of l N H_2SO_4.

I. Sodium hydroxide solution, 2N. Dissolve 80 g NaOH into 1 liter DI water.

J. Phenolphthalein indicator solution, alcohol-based. Dissolve 5g of phenolphthalein in approximately 500mL 95% ethyl or isopropyl alcohol. Dilute to 1 L with DI water.

K. Stock phosphorus solution. Dissolve 0.4393g of oven-dried and desiccated KH_2PO_4 in 1L DI water. 1.0mL = 100ug P. Record preparation of standards in standards logbook.

L. Standard phosphorus solution. Dilute 100mL of stock phosphorus solution to 1L with DI water. 1.0mL= 10ug P. Record preparation of standards in standards logbook.

VI. Definitions

A. Laboratory Control Sample (LCS)- A purchased standard of a known concentration that is from a different source than the calibration standards. The LCS is used to determine laboratory performance.

B. Preparation Blank (Prep Blank)- An aliquot of reagent water that is treated exactly as a sample. The prep blank is used to determine if method analytes are present in the lab environment, reagents or glassware.

C. Calibration standard- A solution prepared from the dilution of stock standard solutions. The calibration solutions are used to calibrate instrument response with respect to analyte concentration.

D. Matrix Spike (MS)- An aliquot of environmental sample to which a known quantity of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

VII. Procedure

A. Determination of total phosphorus (TP-WM), all forms of phosphate.

1. Check pH of the sample and record in Phos batching program.

2. Into a digestion vessel, measure 50mL sample or an aliquot diluted to 50mL, hengar granules, and add a drop of phenolphthalein solution. If it turns pink, add 11N H_2SO_4 drop-wise until color disappears.
3. Add 0.4g solid ammonium or potassium persulfate.
4. Add lmL 11N H_2SO_4. Use disposable pipets to reduce chances of contamination.
5. Set the Hot Block to approximately 140°C, enough to get the samples to 100°C. Boil gently under a ventilation hood until a final volume of less than 10mL is achieved. Do not allow sample to go to dryness.
6. For samples suspected to contain iron interference, add 5mL of sodium bisulfite and mix. continue heating for 20 min. Do not allow sample to go to dryness.
7. Cool, dilute to 30mL in the digestion vessels (filter sample, if needed), add 1 drop phenolphthalein solution, and neutralize to a faint pink color using 2N NaOH solution. *Do not filter while sample is neutral, as phosphate-containing precipitates form that will dissolve under acid conditions.*
8. Bring sample up to 50 mL volume with DI water in digestion vessel and mix well. Transfer a 25 mL aliquot to 60 mL glass vessel. If a larger dilution is necessary, transfer a small volume to glass vial and bring to 25 mL final volume with DI water.
9. Determine phosphorus as outlined in the determination of orthophosphate, C.2. and C.3. below.

B. Determination of total phosphates (code "TPFATE") hydrolyzable and condensed phosphates.

1. Into a digestion vessel, measure 50mL sample or an aliquot diluted to 50mL and add a drop of phenolphthalein solution. If it turns pink, add 11N H_2SO_4 drop-wise until color disappears. Record pH of original sample in Phos Batching sheet.
2. Add 1 mL of H_2SO_4 to sample. Use a disposable pipet to reduce risk of contamination.
3. Set the Hot Block to approxiamately 140°C, enough to get the samples to 100°C. Boil gently with hengar granules under a ventilation hood until a final volume of less than 10mL is achieved. Do not allow sample to go to dryness.
4. Treat samples containing interferences, as above.
5. Cool samples, filter if needed, then add 1 drop phenolphthalein solution, and neutralize to a faint pink color using 6N NaOH solution. *Do not filter while sample is neutral, as phosphate-containing precipitates from that will dissolve under acid conditions.*
6. Bring sample up to 50 mL volume with DI water in digestion vessel and mix well. Transfer a 25 mL aliquot to 60 mL glass vessel. If a larger dilution is necessary, transfer a small volume to glass vial and bring to 25 mL final volume with DI water.

7. Determine phosphorus as outlined in the determination of orthophosphate, C.2. and C.3. below.

C. Determination of orthophosphate (OP-WM).

1. Decant 25mL of unpreserved sample or an aliquot diluted to 25mL into a glass vial, add 1 drop of phenolphthalein solution. If a pink color appears, add 5N H_2SO_4 drop-wise until color disappears.
2. Add 4 mL combined color reagent and mix thoroughly.
3. After 10 (but before 30) minutes, measure the absorbance of each sample at 880nm, using DI water as the reference solution. The absorbance is correlated to a point on a linear regression which correlates to a concentration of phosphorus.

D. For other forms of Phosphorus, see Section 1.C – 1.F.

VIII. Calculations

A. Refer to the Standards Logbook for the most current calibration curve. To generate a new calibration curve, use phosphorus standard solution. Make the following dilutions: blank (DI water), 0.01mg/L, 0.025 mg/L, 0.05 mg/L, 0.10 mg/L, 0.20 mg/L, 0.50 mg/L and 1.0 mg/L. Add phenolphthalein and 5N H_2SO_4 to each, as you would a sample. Add 8 mL combined color reagent, and read at 880 nm after 15 minutes. Record absorbances, and plot them on the x-axis against the corresponding concentrations. Follow with a linear regression, and use the following calculation:

$Y = (MX + B) * DF$, where: Y = concentration (mg/L)
M = slope of the linear regression
X = absorbance at 620nm
B = y-intercept of linear regression
DF = dilution factor, if any

Note that M and B values will differ with each calibration.

B. Calculating RPD:

The RPD is the relative percent difference, and is a measure of the consistency of the sample replicate for the sample set. First, calculate the results for both the sample and the sample replicate. Then apply the two results to the following calculation:

$$RPD, \% = \frac{(R1 - R2)}{(R1+R2)/2} \times 100$$

where: R1 = value achieved for sample, mg/L
R2 = value achieved for sample replicate, mg/L

C. Calculating the sample spike recovery:

The sample spike is a test if the sample matrix is suitable for accurate testing. To calculate the % recovery, first calculate both the sample and the spiked sample. Then apply the two results to the following calculation:

$$\% \text{ Recovery, } \% = \frac{(SR - R)}{STV} \times 100$$

where: SR = value of sample with added spike, mg/L
R = value of sample, mg/L
STV = true value of spike added to sample, mg/L

D. Calculating the LCS:
The LCS is the laboratory control standard and is a test of the accuracy of the calibration and the test overall. To calculate the % recovery of the LCS, calculate the LCS sample, and then apply it to the following calculation:

$$\% \text{ Recovery, } \% = \frac{(RL)}{(LTV)} \times 100$$

where: RL = achieved value for LCS sample, mg/L
LTV = true value of LCS, mg/L

E. Calculating poly-phosphate, as P is done during data transfer, automatically, when needed.

IX. Quality Control

A. All reagents and standards are to be labelled with the following information: date prepared, date expires, preparer's initials, analyte, contents, and concentration of contents. Record preparation of all reagents in the reagent prep logbook.

B. A sample prep blank of dilution water must be analyzed with each batch of twenty or fewer samples. Prep blanks must be determined to be BRL (below reportable level).

C. Two purchased, second source, laboratory control standards (LCS) are run with each batch of twenty or fewer samples. One is a "complex" solution, which goes through the digestion process, while the other is a "simple" solution, which is not digested. The acceptable recovery of a LCS is 85-115%.

D. An in-house standard (CC) made from the standard solution used to calculate the curve is run after every ten samples. The true value of the CC is 0.25mg/L. The acceptable recovery of an in-house standard is 90-110%.

E. A DI water blank, (CB), is analyzed after the CC every ten samples. The CB must be BRL (below reportable level).

F. A sample spike and a sample duplicate are run with every batch of twenty or fewer samples. The acceptable recovery for a sample spike is 75-125%. The acceptable relative percent difference (RPD) for a sample duplicate is less than or equal to 20%.
G. A new calibration curve must be analyzed annually or whenever a new stock solution is made. The correlation coefficient of the curve must be >0.9975.
H. Samples labeled as trip blanks, field blanks, or equipment blanks are ineligible samples for spiking and duplicating.

X. Safety

A. The toxicity and 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 to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.
B. Always wear safety glasses for eye protection as well as lab coats.
C. When diluting H_2SO_4, always add acid to water.
D. Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

XI. Pollution Prevention

A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.
B. Reagents and chemicals should be purchased and/or prepared in volumes consistant with laboratory use to minimize the volume of disposal.

XII. Waste Management

A. It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

XIII. Method Performance

A. This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.
B. See section IX Quality Control in this SOP for acceptable limits.

XIV. Corrective Action for Out-of-Control or Unacceptable Data

A. Should the calibration curve have a correlation coefficient of <0.9975, remake and reanalyze curve before processing samples.
B. Should the preparation blank, LCS or inhouse standard fail acceptance criteria, redigest and reanalyze batch.
C. Should the matrix spike or sample duplicate analysis fail acceptance criteria, a non-conformance report must be generated or the sample QC must be reanalyzed.

XV. Batching Program for Phosphorus

A. Double click on Phoenix MDB (2K MDE) to get to main menu.
B. Click on QA/QC Batching and double click on Prep Batching.
C. Go to the Batch square and type in "Phos-T".
D. Go to New Batch and click, this brings up the last batch made.
E. Click again and this brings up a new list of samples to pick for each batch to be made.
F. Click on numbers under Batches to build for the number needed.
G. Click on Build Batch.
H. Finish making the batch by filling in QC sample, weight/volume, initials and finish date.
I. All samples MUST be batched. They cannot be added to the Sequence Program if they are not in a batch.
J. All batches must have Initial sample weight/volume entered by the analyst before the batch can be added to the Sequence Program.
K. Samples cannot be transferred if % Solid is still pending.
L. Samples cannot be transferred if the batch LCS fails.
M. Changing the QC Sample:
1. A batch cannot be loaded in the Sequence Program until it has been assigned a QC sample. However, a QC sample can be changed.
2. To change a QC sample, select the batch in Prep Batching and Clear the finish date.
3. Change the QC sample to the desired QC sample. The LCS and Blank will automatically change in the Sequence Program.
4. The original spike and dup will remain. Do not select them for transfer and the results will go nowhere.
5. The spike and dup for the new QC sample will have to be added manually to the end of the sequence.

N. Updating the Calibration:
1. When recalibrating the instrument, change the dates in the set-up to reflect the effective ending date of prior calibration.

2. Enter a new record. This can be done by copying an existing record and pasting it on a new line.
3. Update the appropriate date, Slope, y-intercept, LCS true value and source, spike true value, etc.

O. Selecting or Deselecting Samples for Transfer:

1. Before transferring samples, the Batch Blank and Batch LCS must be selected. This is necessary so when a sample is selected, the program knows which QC is associated with it.
2. If a Blank or LCS is deselected, all samples in the batch will be deselected.

P. Rerunning QC Samples:

1. QC can be re-analyzed by re-adding the samples and appropriate QC record to the end of the sequence.
2. Deselect the original run(s) and select the new one, and the corresponding samples.

XVI. Data Transfer Program for Phosphorus

A. Click on Phoenix MDB (2K MDE) to go to the main menu.
B. Click on Data Transfer.
C. Double Click on Phosphate Sequence and Data Transfer.
D. Select "P" on Run Type drop down box.
E. Click on New Sequence or Add Batch for however many to be done.
F. After batches are completed, add analysts initials to bottom and click on Transfer to Labworks.
G. Click on Present Log to print a copy and exit page.

XVII. References

A. Standard Methods for the Examination of Wastewater, 22nd ed., 2011.
Methods used: 4500-P B2 for hydrolysis; 4500P B5 for digestion; 4500-P E for orthophosphate and color formation.
B. Phosphorus, All Forms, Environmental Express Hot Block Standard Procedure.
Method used: Colorimetric, Ascorbic Acid, Single Reagent.
C. USEPA Method 365.3, Phosphorus, All Forms, 1978.

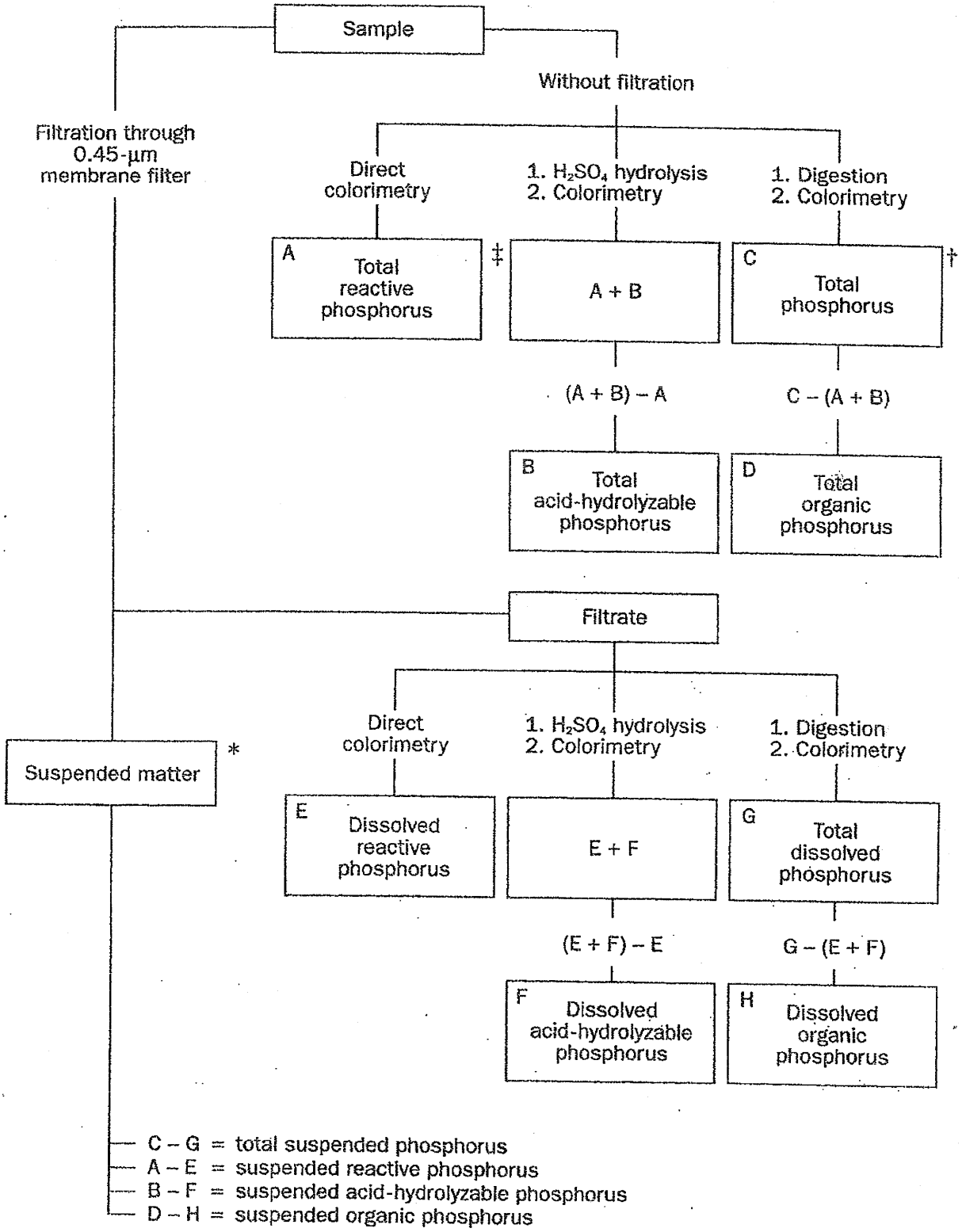
Appendix A- Phosphorus Glassware Cleaning Instructions

1) Make new 10% HCl solution at least once a week.
   a) Dump old solution in acid waste.
   b) Rinse bottles and caps thoroughly with deionized water.
      i) Bottles are usually in hood #8.
   c) Fill bottles to 900 mL line with deionized water.
   d) In a hood, fill bottles to 1000 mL line (the bump near the top), with concentrated HCl.
   e) Cap bottles and CAREFULLY invert several times to mix.

2) PUT 10% HCl bottles on stir/heat block in Hood #8 to heat.
   a) Turn on power switch for block.
   b) Turn heater switch to high. (Downward is low, upward is high, middle is off.)
   c) Set the heat knob to 150°C. Bottles should heat up within the hour.
   d) Monitor bottles to make sure they don't boil.

3) Handwash the 60 mL vessels (NOTE: Some vessels have a 50mL volumetric line for digestions, while others have a 25mL volumetric line for color developement. They are washed the same way!)
   a) Pour out the water that the vessel has been soaking in, while in the white floats.
   b) Fill with hot water, add a squirt of Phosphate free detergent and scrub with brush.
      i) Brush is the clean, white bristled brush hanging to the right of the sink.
      ii) The Phosphate free detergent is in a squeeze Nalgene bottle near brush.
      iii) Remove any sharpie marks on the outside of the vessel while scrubbing.
   c) Rinse washed vessels numerous times with DI water.
   d) Place the scrubbed / rinsed vessels upright in the plastic storage trays.
      i) Trays can usually be found piled up in the phosphorus area.
         (1) Blank trays are for "25 mL" vessels
         (2) Numbered trays are for "50 mL" vessels.
   e) In a hood, carefully fill each vessel to the brim with HOT 10% HCl solution, which was heated in Step #2.
      i) Use a hotglove borrowed from the ammonia setup if necessary; the acid solution should be hot enough to be slightly uncomfortable, but not boiling hot.
   f) Let the vessels soak in HCl solution for a few minutes
      i) The time necessary to pour another tray should be sufficient.
   g) Using the funnel that was stored with the bottles, pour the hot HCl solution back into its bottle. Cap it and store in hood #8 when finished.
   h) Take the tray of acid washed vessels to the sink for rinsing.
      i) Rinse three times with deionized water and pour out.
      ii) Fill vessels to the brim with deionized water
   i) Store clean deionized water filled tray of vessels either on the phosphorus countertop or in the shelves in the rear storage room.
      i) Cover vessels with clean paper towel to protect them.
   j) Dump DI water in vessels and rinse once more with DI before use.

Appendix B


Sample
Without filtration
Filtration through 0.45-µm membrane filter
Direct colorimetry
1. H_2SO_4 hydrolysis 2. Colorimetry
1. Digestion 2. Colorimetry
A Total reactive phosphorus
‡
A + B
C Total phosphorus
†
(A + B) – A
C – (A + B)
B Total acid-hydrolyzable phosphorus
D Total organic phosphorus
Filtrate
Direct colorimetry
1. H_2SO_4 hydrolysis 2. Colorimetry
1. Digestion 2. Colorimetry
Suspended matter
*
E Dissolved reactive phosphorus
E + F
G Total dissolved phosphorus
(E + F) – E
G – (E + F)
F Dissolved acid-hydrolyzable phosphorus
H Dissolved organic phosphorus
C – G = total suspended phosphorus
A – E = suspended reactive phosphorus
B – F = suspended acid-hydrolyzable phosphorus
D – H = suspended organic phosphorus

Revision History Log for SOP #321 TP

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
1/6/14	5	Simple & Complex LCS added, recording pH of sample, updated vessel cleaning procedure	Kathy Cressia	Eric Geyer	1/6/14
10/04/16	5.1	Section 1.0- added D, E, F, G (forms of phos), Section VII- added D, added EPA method to references, just referring to forms of Phos, added Appendix B- flow chart	Kathy Cressia	Eric Geyer	10/04/16
3/15/18	5.2	Doubled combined reagent recipe, updated method reference year for MUR	Kathy Cressia	Eric Geyer	3/15/18

Expiration Date

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Effective Date: 08/22/19
Version No.: 5.2
Initiated By: Kathy Cressia
Approved By:

SOP No.: 324.2540D

Title: Total Suspended Solids (TSS) dried at 103°-105°C

Scope: This method covers the determination of suspended solids in drinking, surface, saline waters, and domestic and industrial wastes. The practical range of the determination is 5.0-2000mg/L. Samples containing more than 2000mg/L suspended solids require sample dilution prior to analysis.

I. Summary of Method

A. A well-mixed sample is filtered through a pre-weighed glass fiber filter. The filter is retained and dried to a constant weight at 104°C. The increase in filter weight represents the total suspended solids (TSS).
B. If total dissolved solids (TDS) is being determined on the sample, the filtrate may be retained for TDS analysis.

II. Interferences

A. Exclude large floating particles or submerged agglomerates of nonhomogeneous materials from the sample if it is determined that their inclusion is not desired in the final result. Because excessive residue on the filter may form a water-trapping crust, limit sample to that yielding no more than 200mg residue.
B. For samples with high TDS, thoroughly wash filter to insure no TDS will be measured as part of the TSS.

III. Sample Collection, Preservation and Storage

A. Samples for solids analysis are unpreserved and stored at 4°C prior to analysis.
B. Samples are analyzed as soon as possible, but not after a holding time of 7 days after sample collection.

IV. Equipment and Supplies

A. Glass fiber filters, Whatman 934-AH or equivalent.
B. Gooch crucibles of same size as filters, with adapter.
C. Filter flasks, 250ml or 500ml volume.
D. Drying oven, 104°C +/-1°C.
E. Desiccator. Change desiccant every month, or when desiccant changes color.
F. Graduated cylinder, 100mL.
G. Analytical balance, capable of weighing to 0.1mg.

V. Reagents and Standards

A. Laboratory deionized water.
B. Laboratory Control Sample; purchased. ERA Solids catalog #499 or equivalent.

VI. Definitions

A. Laboratory Control Sample (LCS)- A purchased standard of a known concentration that is from a different source than the calibration standards. The LCS is used to determine laboratory performance.
B. Preparation Blank (Prep Blank)- An aliquot of reagent water that is treated exactly as a sample. The prep blank is used to determine if method analytes are present in the lab environment, reagents or glassware.
C. Sample Duplicates (Dup)- Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of the sample and duplicate indicate precision associated with laboratory procedures.

VII. Procedure

A. Preparation of gooch crucibles.
1. Clean and rinse well enough crucibles to complete a full batch of twenty samples, allowing one crucible for each sample, plus one for a LCS, one for a blank, and two crucibles for sample replicates. Be sure crucibles are marked for identification purposes.
2. Attach filter flasks to the filter set-ups. Place the rubber adapter to the top of the flask.
3. Insert a glass fiber filter disc into a clean gooch crucible, grid side down. Place the crucible to the adapter. With vacuum applied, wash the disc with 30mL DI water to insure a snug fit, and to rinse loose fibers from filter. Discard filtrate. Place crucibles in oven at 104°C for one hour.
4. Move crucibles to desiccator to cool for 1 hour. Handle crucibles using forceps. Oils from the skin can effect final results.

B. Batch Creation
1. Open Phoenix Laboratories Lims and click on QA/QC Batching,TSS/TDS/TS Batching.
2. Click on "New Batch" and use the pull-down menu to select TSS analysis. This will bring up all samples in the batching program that need to be run.
3. Create a batch of 20 samples or fewer by clicking on the box in the "Select" column.
4. Using the pull-down menus at the bottom of the window, choose a QC sample. If the batch contains more than 11 samples, choose a sample for the second dup. Click "OK"; this will bring up a TSS Batch Worksheet.
5. Using forceps to handle each gooch crucible, record the Dish ID # in the appropriate column. Then record the initial weight of each crucible in

the"Tare Weight" column of the spreadsheet electronically, using Wetlab Analytical #1A. (Please note: it is important to use forceps when handling the crucibles, as oils from skin can affect final results).

C. Preparation of samples.

1. Allow samples to come to room temperature before analysis.

D. Preparation of filter set-up.

1. Attach filter flasks to the filter set-ups. Place the rubber adapter to the top of the flask.
2. The flask will contain the sample filtrate. Mark it as waste unless TDS is to be performed in conjunction with TSS. Be sure to use clean, DI rinsed filter flasks for TDS determination.

E. Filtration of samples.

1. Place a pre-weighed gooch crucible on the adaptor, shake sample vigorously and rapidly measure appropriate sample volume into a graduated cylinder. Choose a sample volume to yield between 2.5 – 200mg of dried residue. If the volume filtered fails to meet the minimum yield, increase sample volume up to 1L.
2. Pour the sample into the filter set-up. Rinse the graduated cylinder with three successive 20mL portions of DI water, and pour washings into the filter set-up. Allow complete drainage between washings.
3. Continue suction for 3 minutes after filtration is complete.
4. If more than 10 minutes is needed to complete filtration, decrease sample volume.
5. If a representative sample aliquot is not possible by decanting, due to floating material, use a pipet submerged to the center of the sample container to extract a homogeneous representative sample aliquot. Exclude from analysis sand or other heavy materials that settle to the bottom of the sample container after vigorous shaking.
6. Place gooch crucibles with samples particulates trapped on the filter into the drying oven at 104°C. Reserve sample filtrate if TDS is to be determined.
7. Record on the TSS Batch worksheet: sample volume used, oven temperature, time and date samples were put into the oven.

F. Final Analysis of TSS.

1. After a minimum of one hour in the oven at 104°C, transfer the crucibles to a desiccator.
2. Cool in desiccator for a minimum of 1 hour and determine the first weight. Record weight electronically under the appropriate batch number.
3. Return crucibles to the 104C oven for at least an 1 hour.
4. Cool in desiccator for at least an 1 hour and determine the second weight. Record the weight electronically.
5. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% previous weight or less than 0.5mg, whichever is less.
6. Print out each completed batch number and file in the TSS logbook.

VIII. Calculations

A. The TSS value is calculated using the following equation:

$$TSS\ (mg/L) = \frac{(A - B) \times 1000}{SV}$$

Where: TSS = total suspended solids (mg/L)
A = average of final weights within constant weight criteria (g)
B = initial weight of gooch crucible (g)
SV = sample volume (L)

B. Calculating RPD:
The RPD is the relative percent difference, and is a measure of the consistency of the sample replicate for the sample set. First, calculate the results for both the sample and the sample replicate. Then apply the two results to the following calculation:

$$RPD, \% = \frac{(R1 - R2) \times 100}{(R1+R2)/2}$$

where: R1 = TSS value achieved for sample, mg/L
R2 = TSS value achieved for sample duplicate, mg/L

C. Calculating the LCS:
The LCS is the laboratory control standard and is a test of the accuracy of the calibration and the test overall. To calculate the % recovery of the LCS, calculate the LCS sample, and then apply it to the following calculation:

$$\%\ Recovery, \% = \frac{(RL) \times 100}{(LTV)}$$

where: RL = achieved value for LCS sample, mg/L
LTV = true value of LCS, mg/L

IX. Quality Control

A. A reagent blank is analyzed with every batch of 20 or fewer samples. If the blank has a positive TSS value, the sample results are acceptable only if the blank is no more than 10% of the sample result. If the blank has a negative result of less than –5.0 mg/L, the sample results are acceptable only if the absolute value of the blank is no more than 10% of the sample result.

B. A Laboratory Control Standard (LCS) will be analyzed with every batch of 20 or fewer samples. The LCS must be recovered between 85-115%.

C. Sample duplicates are analyzed every 10 of fewer samples. The RPD must be ≤20%, else repeat sample and duplicate analysis to prove matrix effect.
D. Oven temperatures are recorded daily to insure stable oven temperatures. The drying oven must maintain temperature within 103°-105°C. Oven working thermometers are calibrated annually with NIST traceable thermometers.
E. The calibration of the analytical balance is checked daily using Class "S" weights: 50.0000g, 10.0000g, 1.0000g and 0.1000g. The balance is calibrated annually barring any problems throughout the year.

X. Safety

A. The toxicity and 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 to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.
B. Always wear safety glasses for eye protection as well as lab coats and gloves.
C. Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

XI. Pollution Prevention

A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.
B. Reagents and chemicals should be purchased and/or prepared in volumes consistant with laboratory use to minimize the volume of disposal.

XII. Waste Management

A. It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

XIII. Method Performance

A. This method was validated through internal QA/QC monitoring, including annual method detection limit studies, limit of detection studies, precision and accuracy studies, proficiency test samples, blank analysis, laboratory control samples and sample duplicates.
B. See section IX Quality Control in this SOP for acceptable limits.

XIV. Corrective Action for Out-of-Control or Unacceptable Data

A. Should the preparation blank or LCS fail the acceptance criteria given in Section IX, reanalyze batch.

B. Should the sample duplicate analysis fail acceptance criteria, reanalyze the sample and duplicate should sample volume permit. If sample volume is limited, replicate another sample in that batch.

XV. References

A. Standard Methods for the Examination of Waste and Wastewater, 22nd edition, 2011, Method No. 2540D, Total Suspended Solids Dried at 103°-105°C.

Revision History Log for SOP #324.TSS

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
1/15/14	5	LCS vendor change, put drying time as "at least" 1 hr, calibration of oven thermometers, added the 0.1g class "s" wt to daily balance check	Kathy Cressia	Eric Geyer	1/15/14
3/22/18	5.1	MUR change of reference section	Kathy Cressia	Eric Geyer	3/22/18
8/22/19	5.2	Changed / added to Section VII.E.1 – to include minimum yield requirements	Kathy Cressia	Eric Geyer	8/22/19

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Effective Date: 06/09/17
Version Number: 3.3
Initiated By:
Approved By:

SOP Number: 336.353.2

Title: Total Nitrite as Nitrogen / Total Nitrate as Nitrogen

Scope: This method is applicable for the determination of concentrations of nitrites and nitrates in drinking, surface, and ground water, domestic and industrial waste, and solid samples. This method is used for wastewater samples originating from states that do not approve the IC for wastewater samples. This method is applicable for nitrite levels of 0.01 - 0.75 ppm and nitrate levels of 0.02 - 2.5.0 ppm. Samples with nitrite levels in excess of 0.750 ppm and/or nitrate levels in excess of 2.50 ppm require sample dilution prior to analysis.

I. Summary of Method

A. Nitrate in a sample is reduced to nitrite by passage through a cadmium column in an alkaline environment. The resulting nitrite is combined with a sulfanilamide color reagent, which produces a strong pink color. The intensity of the pink color is proportional to the amount of nitrite in the sample.

B. To determine the amount of Nitrate in the sample it is necessary to subtract concentration of ambient nitrite from the concentration of transformed nitrite.
NO_3 = (NO_2 after Cd) – (NO_2 before Cd)

C. All samples that require a nitrate result require analysis of nitrite as well, regardless of whether or not it is requested by the client.

II. Sample Collection, Preservation and Storage

A. Samples are collected in clean plastic or glass containers.

B. Samples are not preserved and need to be analyzed within 48 hours from time of collection. If the Nitrite analysis is completed within the 48-hr hold time, the sample may be preserved with sulfuric acid and stored for up to 28 days at 4°C prior to completion of nitrate analysis.

C. Samples that are preserved with sulfuric acid cannot be used to test for nitrite. Furthermore, if there is no nitrite result to be subtracted form the "NO_2 after Cd" result, then no determination can be made as to how much of the "NO_2 after Cd" result was present as NO_2 initially. The best the lab can do to accommodate these clients is to report the combined result of nitrate and nitrite in a sample; that is, to report the "NO_2 after Cd" result or the "NO_2NO_3" result.

III. Interferences

A. Oxidizing agents such as Residual Chlorine may interfere by oxidizing the cadmium column. Remove residual chlorine by adding Sodium Thiosulfate ($Na_2S_2O_3$) solution.

B. EDTA is added to the buffer to reduce the negative interference of high concentrations of iron, copper, and other metals.
C. Samples that contain high concentrations of oil and grease can coat the surface of the cadmium, reducing its reducing capabilities. Extracting oils from the sample using an organic solvent prior to analysis can eliminate this interference.
D. As with all colorimetric analysis, turbidity or suspended matter can be an interference. Remove by filtering sample through 0.45um pore diameter membrane filter prior to analysis.

IV. Equipment and Supplies

A. Lachat QuikChem 8500 with both Nitrite and Nitrate Manifolds.
B. Cadmium column, Lachat Part #50237.
C. Refer to Lachat SOP #302 for consummables.

V. Reagents and Standards

A. Sodium Hydroxide, 6N. Borrowed from ammonia or phosphate method. Since we do not use a lot of this reagent, it is not worth having a sequestered reagent solely for the preparation of Ammonium Chloride Buffer for Nitrate analysis. (NOTE: When preparing 6N NaOH, decant some from the liter bottle into a separate receptacle. This will reduce the risk cross-contamination between analyses).
B. Ammonium Chloride Buffer. Into a 2-Liter volumetric flask, dissolve 170g Ammonium Chloride and 2.0g EDTA in about 1 liter DI water. Bring up to volume with DI water. Adjust the pH to 8.50+/-0.05 w/ 6N NaOH using a pH meter.
When adjusting pH of the buffer, use a separate receptacle to hold the buffer as the pH is taken. To reduce risk of contamination of the buffer from the pH probe, do not immerse the probe directly into the 2 liters of buffer.
C. Sulfanilamide Color Reagent. (NOTE: This reagent is good for both NO_2/NO_3 and NO_2.) Into a 1-Liter volumetric flask, dissolve 40g Sulfanilamide and 1.0g NED (N-(1-naphthyl)ethylenediamine dihydrochloride) in around 500mL DI water containing 100mL Phosphoric acid. Bring up to volume with DI water. Store in a dark bottle at 4°C. Prepare fresh monthly.
D. Nitrite Stock Solution, 1000ppm. RICCA Catalog # 5445-16 or equivalent.
E. Nitrite Standard Solution, 10ppm. Dilute 10mL Nitrite Stock Solution to 1 liter with DI water. Prepare fresh as needed, up to 3 months.
F. Nitrate Stock Solution, 1000ppm. RICCA Catalog # 5307-16 or equivalent.
G. Nitrate Standard Solution, 10ppm. Dilute 10mL Nitrate Stock Solution to 1 liter with DI water. Prepare fresh as needed, up to 6 months.
H. Nitrite LCS Stock Solution, 1000ppm, Environmental Express Cat # IC-N-M.
I. Nitrite Working LCS, 0.25ppm, 0.205mL of Nitrite Stock LCS into 250mL DI.
J. Nitrate LCS Stock Solution, 1000ppm, Environmental Express Cat # IC-NO-M.

K. Nitrate Working LCS, 1.00ppm, 1.106mL of Nitrate Stock LCS into 250mL DI.

L. **NOTE:** The Nitrite and Nitrate Working Standards in this SOP can be combined together along with the Chloride Working Standards of SOP 346 and the ortho-Phosphate Working Standards of SOP 355 to form a set of "4-point" Working Standards.

Nitrite Working Standards A-F. Prepare fresh as needed, at least monthly.

A. Dilute 37.5mL Nitrite Standard Solution to 500mL with DI water; t.v.= 0.750ppm
B. Dilute 25mL Nitrite Standard Solution to 500mL with DI water; t.v.= 0.50ppm
C. Dilute 12.5mL Nitrite Standard Solution to 500mL with DI water; t.v.= 0.25ppm
D. Dilute 5.0mL Nitrite Standard Solution to 500mL with DI water; t.v.= 0.10ppm
E. Dilute 2.5mL Nitrite Standard Solution to 500mL with DI water; t.v.= 0.05ppm
F. Dilute 50mL of Nitrite Working Standard "D" to 500mL with DI water; t.v.= 0.01ppm

Nitrate Working Standards A-F. Prepare fresh as needed, at least monthly.

A. Dilute 87.5mL Nitrate Standard Solution to 500mL with DI water; t.v.= 1.75ppm
B. Dilute 50mL Nitrate Standard Solution to 500mL with DI water; t.v.= 1.00ppm
C. Dilute 25mL Nitrate Standard Solution to 500mL with DI water; t.v.= 0.50ppm
D. Dilute 5.0mL Nitrate Standard Solution to 500mL with DI water; t.v.= 0.10ppm
E. Dilute 2.5mL Nitrate Standard Solution to 500mL with DI water; t.v.= 0.05ppm
F. Dilute 50mL of Nitrate Working Standard "D" to 500mL with DI water; t.v.= 0.01ppm

M. Carrier is DI water.

VI. Procedure

A. Refer to SOP #302 on use of the Lachat QuickChem 8500 for general information on the operation of the instrument. This method includes information unique to the nitrite and nitrate chemistries.

B. Attach manifold for Nitrite chemistry.

C. Attach manifold for Nitrate chemistry. (NOTE: Be sure to keep the Cadmium column closed until buffer is running through the lines. Efficiency of the reducing capacity of the cadmium reduces as it is exposed to air. Once the reagents are flowing through the manifold to the waste line, it is safe to open the column by turning the dial next to the Cd column on the manifold).

D. At this time, make a visual assessment of the Cadmium column. Check for air bubbles, lines, gaps in the column or any change in the cadmium surface characteristics (Cd granules should appear dark gray). If air bubbles cannot be dislodged by ramping the pump speed and tapping on the column, then the column needs to be replaced with a new one. Keep at least two prepared columns on hand at any time. See the QC section for assessing the efficiency of the column.

E. Load background.

F. Create a worksheet for the samples to be analyzed, including all QC requirements. The two standards for the cadmium column efficiency check (see IV.E.) should be the first two samples on each run (The column efficiency needs to be >90% to pass). Check standards will be

included automatically after 10 samples. They must pass within 90-110% of true value, otherwise the system recognizes the error and stops automatically.

G. Start standards and samples.

H. If the correlation coefficient is at least 0.9975 and the RPD values are at 10% or less, the calibration passes. It may be necessary to remake standards.

I. After samples have been analyzed, any dilutions needed have been run, and all QC criteria have been met, print out calibration statistics, and staple them along with the chart records to the sample report. Highlight all reported results on the report sheet. Leave the run on the WetChem supervisor's desk for review.

J. Close the Cd column.

K. Run DI water through the manifolds for 10 minutes to wash out any chemicals that might dry, encrust the tubing and cause blockages the next time. Remove all the tubes from the DI water and dry out the tubes by allowing the pump to run for an additional 10 minutes.

VII. Definitions

A. Laboratory Control Sample (LCS)- A purchased standard of a known concentration that is from a different source than the calibration standards. The LCS is used to determine laboratory performance.

B. Preparation Blank (Prep Blank)- An aliquot of reagent water that is treated exactly as a sample. The prep blank is used to determine if method analytes are present in the lab environment, reagents or glassware.

C. Calibration standard- A solution prepared from the dilution of stock standard solutions. The calibration solutions are used to calibrate instrument response with respect to analyte concentration.

D. Matrix Spike (MS)- An aliquot of environmental sample to which a known quantity of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

VIII. Calculations

A. Calculating Nitrate:
It is necessary to subtract the result from Nitrite analysis (done separately) from the result off the Lachat (NO_2+NO_3) to get a value for Nitrate.

$NO_3 = (NO_2NO_3) - (NO_2)$

Where: NO_3 = final result for Nitrate as N, ppm
NO_2NO_3 = result from Lachat, ppm
NO_2 = result from Lachat, ppm

NOTE: Do not subtract negative NO2 values. Only positive NO2 values are subtracted.

B. Calculating RPD:
The RPD is the relative percent difference, and is a measure of the consistency of the sample replicate for the sample set. First, calculate the results for both the sample and the sample replicate. Then apply the two results to the following calculation:

$$\text{RPD, \%} = \frac{(R1 - R2)}{(R1+R2)/2} \times 100$$

where: R1 = value achieved for sample, mg/L
R2 = value achieved for sample replicate, mg/L

C. Calculating the sample spike recovery:
The sample spike is a test if the sample matrix is suitable for accurate testing. To calculate the % recovery, first calculate both the sample and the spiked sample. Then apply the two results to the following calculation:

$$\text{\% Recovery, \%} = \frac{(SR - R)}{STV} \times 100$$

where: SR = value of sample with added spike, mg/L
R = value of sample, mg/L
STV = true value of spike added to sample, mg/L

D. Calculating the LCS:
The LCS is the laboratory control standard and is a test of the accuracy of the calibration and the test overall. To calculate the % recovery of the LCS, calculate the value of the LCS sample, and then apply it to the following calculation:

$$\text{\% Recovery, \%} = \frac{(RL)}{(LTV)} \times 100$$

where: RL = value for LCS sample, mg/L
LTV = true value of LCS, mg/L

IX. Quality Control

A. Prep Blank must be analyzed every batch of 20 or fewer samples. This is a DI water aliquot treated like a sample, undergoing all treatments the samples undergo. The prep blank must yield levels of nitrite and nitrate below the reporting level. The minimum detectable limit is 0.01mg/L for both nitrite and nitrate.

B. Laboratory Control Standards must be analyzed every batch of 20 or fewer samples. This standard comes from RICCA (or equivalent second source) and has a certified value. It must be recovered 90-110%. A failing LCS for a batch of samples requires re-analysis of the batch.

C. Continuing calibration checks must be analyzed every 10 samples, as is written into the software of the Lachat QuickChem 8000. They must pass within 90-110%. The calibration check for both nitrite and nitrate is the C standard from the calibration curves.

D. A Cadmium column efficiency check must be analyzed at the beginning of the run. Run a second source, 0.5 ppm Nitrate only standard through the column. Run a second source, 0.5 ppm Nitrite only standard through the column. The column efficiency is determined by this calculation: $\frac{\text{Nitrate peak Area}}{\text{Nitrite peak Area}} \times 100 = \%\ \text{Efficiency}$

The acceptable limits for this standard is +/-10%. If the % Efficiency result is outside of this range, the column must be repacked or replaced.

E. Sample replicates must be done every batch of 20 or fewer samples. They must have a RPD of no more than 20%.

F. Sample spikes must be done every batch of 10 or fewer samples. A known amount of nitrate is added to a sample to test the sample matrix. The spike need be recovered 90-110%, however, if recoveries fall outside of this range, and the LCS and Blank spike are acceptable, it is judged to be sample related and not system related. Report % recovery.

G. MDL studies are performed annually or when a different analyst is performing the analysis. Seven replicates of a 0.05 mg/L standard are analyzed for both nitrite and nitrate.

H. A Limit of Detection (LOD) study must be analyzed annually. All sample processing steps of the analytical method shall be included in the determination of the LOD. The LOD study is achieved by analyzing a standard at no more than 2-3x the LOD. In many cases, a 1:1 dilution of the lowest calibration standard is prepared and analyzed. LOD recoveries must be 50 – 150%.

I. Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. 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 portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

J. An initial demonstration of capability (IDOC) must be performed to prove the generation of acceptable data with regard to accuracy and precision. An initial demonstration must be performed by each analyst and consists of analyzing 4 LCS solutions, usually over a series of days.

K. Standards preparations are documented in the Standards Prep Logbook.

L. Reagent preparations are documented in the Nitrate/Nitrite Reagent Prep Logbook.

M. Lachat use and maintenance is documented in the Lachat Maintenance Logbook.

X. Safety

A. The toxicity and 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 to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.

B. Always wear safety glasses for eye protection as well as lab coats.
C. Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

XI. Pollution Prevention

A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.
B. Standards should be purchased in volumes consistant with laboratory use to minimize the volume of expired standards to be disposed.

XII. Waste Management

A. It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

XIII. Method Performance

A. This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, semiannual proficiency tests, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.
B. See section IX Quality Control in this SOP for acceptable limits.

XIV. Corrective Action for Out-of-Control or Unacceptable Data

A. Should the calibration curve have a correlation coefficient of <0.9975, remake and reanalyze curve before processing samples.
B. Should the preparation blank, LCS or inhouse standard fail acceptance criteria, do not proceed. Find problem and reanalyze. All samples must be bracketed by passing Cal checks.
C. Should the matrix spike or sample duplicate analysis fail acceptance criteria, a non-conformance report must be generated or the sample QC must be reanalyzed.

XV. References

A. Nitrate by Automated Cadmium Reduction, Method 353.2, Methods for the Chemical Analysis of Water and Wastes, EPA 600 Series, Revision 2, 1993.
B. Nitrate Nitrogen by Automated Cadmium Reduction, Method 4500-NO3 F, Standard Methods for the Examination of Water and Wastewater, 22nd edition, 2000.
C. Nitrate/Nitrite Analysis, Method 10-107-04-1-C, Lachat Instruments QuikChem 8000, July 1995.
D. Nitrite Analysis, Method 10-107-05-1-O, Lachat Instruments QuikChem 8000, July 1995.

Revision History Log for SOP # 336

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
2/12/15	3.1	Scope= new curve ranges, added new sections out of Summary, Sec. V.H adjusted A Std to reflect new range, added Sections VII, X – XIV, took Cd Column efficiency calc out of Sec. VIII, added D, H, I, J to Sec. IX, updated SM Reference in Sec. XV.	Kathy Cressia	Eric Geyer	2/12/15
5/10/17	3.2	Sec VIII.A- added "NOTE" to calculation, sec IX.D- updated Cd column efficiency check and added calculation, Sec IX.F- changed MS frequency to 10%, Sec XV.A- updated method revision and year	Kathy Cressia	Eric Geyer	5/10/17
6/9/17	3.3	Section IX.D- updated calculation based on area, section V.D, F, H-K- new sources of stock solution and second source	Eric Geyer	Kathy Cressia	6/9/17

Expiration Date

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SOP Number: 304.NH3/TKN

Title: Ammonia/TKN Phenate Method

Scope: This method covers the determination of ammonia, organic nitrogen, and Kjeldahl nitrogen in drinking, surface and saline waters, domestic and industrial wastes, soils and leachates. The working range for water samples is 0.05-8.0 mg/L, and 2.0-400 mg/kg for solids. Higher concentrations may be determined by sample dilutions.

I. Summary of Method

A. The sample is buffered at a pH of 9.5 with borate buffer in order to decrease hydrolysis of cyanides and organic nitrogen compounds, and then distilled into a sulfuric acid solution. The ammonia ion in the sulfuric acid solution is then determined by colorimetric analysis. This method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction produced is measured at 630nm, and is directly proportional to the original ammonia concentration.

B. The determination of organic nitrogen (ORGN) is based on the ammonia (NH3) and TKN results of a sample. ORGN conc. = TKN conc. – NH3 conc. Using this relationship, it is also possible to determine the concentration of TKN, when the ORGN and NH3 are known.

II. Interferences

A. When present, residual chlorine must be removed by pretreatment of the sample with sodium thiosulfate before distillation. During sample development calcium and magnesium ions may precipitate if present in sufficient concentrations. EDTA is added to the sample in-line in order to prevent this problem. Distillate turbidity may interfere and can be removed by manual filtration.

III. Sample Collection, Preservation and Storage

A. Samples can be collected in plastic or glass. They must be stored at 4°C until time of analysis.

B. Samples must be H_2SO_4 preserved to pH 1.5-2 and analyzed within 28 days of collection.

IV. Equipment and Supplies

A. Kjeldahl tubes.
B. Snap Cap, 70mL with 50mL lines.
C. Graduated cylinders, 50 mL.
D. Lachat QuickChem 8000 autoanalyzer with XYZ autosampler.
E. Digestion heating block with temperature control.
F. Reflux condenser distillation unit with temperature control.

V. Reagents and Standards

(NOTE: Use only reagent grade chemicals for standards and reagents)

A. Ammonia Stock Solution (1.0 ml=1000mg NH3-N): Dissolve 3.819g reagent grade ammonium chloride (NH4CL), that has been oven dried and desiccated, in DI water and bring to volume in a 1 liter volumetric flask.
B. Intermediate Ammonia Standard (1.0 ml=20 mg NH3-N): Dilute 20.0mL ammonia stock solution in 1-liter volumetric flask. Bring up to volume with DI water.
C. Ammonia Spiking Solution (1.0 ml=100 mg NH3-N): Dilute 100mL ammonia stock solution in 1-liter volumetric flask. Bring up to volume with DI water.
D. Ammonia Working Standards:

Std. ID	mL of Inter. Std.	Dilute to	Concentration
A	80	200mL	8.0 mg/L
B	50	250mL	4.00 mg/L
C*	40	500mL	1.60 mg/L
D	5	250mL	0.40 mg/L
E	1.0	200mL	0.10 mg/L
F	0.5	200mL	0.05 mg/L
G	0	250mL	0.00 mg/L

Note: All working standards are to be diluted and taken to volume with DI water. They are to be 1:1 H2SO4 preserved. *Working standard C is the calibration check standard.

E. Sodium hydroxide solution, 6 N: While stirring in a cold water bath, dissolve 240g Sodium Hydroxide (NaOH) in 1 liter of DI water.
F. Sodium hydroxide solution, 0.2 N: In a 1-liter volumetric, dissolve 8g of Sodium Hydroxide (NaOH) in 800mLs of DI water. Dilute to volume and mix.
G. Borate buffer: Add 44mls of 0.2 N NaOH solution to 500 ml of DI water. Add sodium tetraborate (2.5g anhydrous $Na_2B_4O_7$ or 9.50g $Na_2B_4O_7$ *$10H_20$) and dilute to 1 liter.
H. Sulfuric Acid Solution, 1.0N, to about 800mL DI water, add 28mL concentrated H_2SO_4,then bring to a final volume of 1L with DI. Invert 3 times.
I. Sulfuric acid solution, 0.02N: To about 800mL DI water, add 20mL 1.0N H_2SO_4 (see N above). Dilute to 1L with DI water.
J. Phenolphthalein Indicator Solution: Dissolve 5.0g phenolphthalein in 500mL 95% ethyl or isopropyl alcohol and dilute to 1L with DI water.
K. Sodium Phenolate: In a 1 liter volumetric flask, dissolve 88ml of 88% liquefied phenol or 83g crystalline phenol in approximately 600ml of DI water. While stirring, slowly

add 32g sodium hydroxide. Allow to cool, then dilute to volume and mix. **CAUTION: WEAR GLOVES! Phenol causes severe burns and is rapidly absorbed through skin!**

L. Sodium Hypochlorite: In a 500-ml volumetric flask, dilute 250ml "Regular Strength" bleach (5.25% NaOCl) to volume with DI water. (NOTE: DO NOT use "Industrial Strength" or "Ultra Strength" Bleach as they contain higher concentrations of NaOCl.)

M. Buffer Solution: In a 1-liter volumetric flask, dissolve 50.0g disodium ethylenediamine tetraacetate (Na_2EDTA) and 5.5g sodium hydroxide in about 900mls DI water. Dilute to volume and mix.

N. Sodium Nitroprusside: In a 1-liter volumetric flask, dissolve 3.50g sodium nitroprusside (sodium nitroferricyanide) and dilute to volume with DI water. **CAUTION: WEAR GLOVES! Health Hazard -- Affects the Central Nervous System.**

O. Carrier: DI water.

P. TKN Digestion Reagent: Dissolve 134g K_2SO_4 and 7.3g $CuSO_4$ in 500mL DI water. Slowly add 134mL concentrated H_2SO_4. Bring to a final volume of 1L with DI water.

Q. TKN Sodium Hydroxide-thiosulfate reagent: Slowly dissolve 500g NaOH and 25g $Na_2S_2O_3*5H_2O$ in water and dilute to 1 liter with DI water. **Caution: Prepare this reagent in a cooling waterbath, as it is exothermic.**

VI. Definitions

A. Laboratory Control Sample (LCS)- A purchased standard of a known concentration that is from a different source than the calibration standards. The LCS is used to determine laboratory performance.

B. Preparation Blank (Prep Blank)- An aliquot of reagent water that is treated exactly as a sample. The prep blank is used to determine if method analytes are present in the lab environment, reagents or glassware.

C. Calibration standard- A solution prepared from the dilution of stock standard solutions. The calibration solutions are used to calibrate instrument response with respect to analyte concentration.

D. Matrix Spike (MS)- An aliquot of environmental sample to which a known quantity of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

VII. Procedure

A. Samples are preserved with H_2SO_4 to pH of <2 at time of sampling, and stored at 4°C until time of analysis. Samples to be determined for ammonia or TKN have a holding time of 28 days from time of collection.

B. Sample screening:

1. Verify pH and record in Ammonia Prep Batching Program.
2. If residual chlorine is present, destroy with Sodium thiosulfate. Record check in Ammonia Prep Batching Program.

C. Ammonia distillation:

1. Rinse Kjeldahl tubes with DI water. Measure 50mL of aqueous sample (or 0.01g of soil diluted to 50mL DI water) into a Kjeldahl tube containing boiling stones. Add 5mL borate buffer, 2-3 drops of phenolphthalein, and enough 6N NaOH to turn the sample light pink. If too much 6N NaOH was added, add 0.02N H_2SO_4 drop-wise until sample is light pink. Phenolphthalein is light pink at a pH of 9.5; this is the pH at which the distillation should occur. Add 1.0mL of Ammonia Spiking Solution to all matrix and blank spike tubes (T.V.= 2.0mg/L). Label the tubes with sample ID number and sample volume using a permanent marker. Record information in the Ammonia - TKN batching program.
2. Place Kjeldahl tubes containing prepared sample onto the distillation heating block, and attach reflux unit. Turn on condenser water.
3. Label clean snap cap tubes with appropriate sample ID number and sample dilution information, and add 10mL 0.02N H_2SO_4 to each tube. Place the snap cap under the condenser, and raise it so that the tip of the condenser is below the surface of the sulfuric acid, but not touching the bottom.
4. Turn on the block and set upper temperature limit to 300°C.
5. Some sample matrices require the addition of an anti-foaming reagent. If a sample begins to bubble and climb up the sides of the tube, add a few drops of antifoam. If a sample is suspected or expected to foam, antifoam may be added as part of sample pretreatment.
6. After 40mL has been distilled, remove snap caps from under the condensers, and bring up to 50mL with DI water. Refrigerate at 4°C until time of color development on Lachat.
7. If sample requires TKN or organic nitrogen determination, save the remainder of sample in the Kjeldahl tube for digestion.

D. Organic Nitrogen/TKN digestion:

1. If samples require NH3 & TKN (or ORGN only), perform ammonia distillation procedure as described in III.B. above, discard distillate, and allow the remainder of the sample in the Kjeldahl tube to cool. If only TKN is requested, place 50mL sample in a clean Kjeldahl tube containing boiling stones.
2. Move to TKN digestion heating block, carefully add 10mL TKN digestion reagent to Kjeldahl tube. Add 1.0mL of Ammonia Spike Solution to all matrix and blank spike tubes (T.V. = 2.0mg/L). Do not attach reflux unit. Turn on block and set upper limit temperature to 400°C. Boil under a ventilation hood until copious white fumes are observed. Continue to digest for another 30 minutes after copious fumes are observed and after the temperature is at least 375°C.
3. Allow samples to cool up to 2 minutes, and dilute with 50mL DI water (NOTE: If samples are allowed to cool longer than 2 minutes, the residue has a tendancy of sticking to the bottom of the tube even with vortexing). Vortex to mix. Make sure that boiling chips are free from the dried slurry in the bottom of the tubes and that the acid residue is dissolved into the water!
4. Move to distillation heating block.

5. Tilt tube away from personnel, towards the back of the hood, and carefully add 10mL sodium hydroxide-thiosulfate reagent to form an alkaline layer at tube bottom. Connect tube to distillation apparatus and swirl tube to insure complete mixing. Do not swirl the tube until the reflux unit is attached. The pH of the solution should exceed 11.0.
6. Label clean snap cap tubes with appropriate sample ID number and sample dilution information, and add 10mL 0.02N H_2SO_4 to each tube. Place the snap cap under the condenser, and raise it so that the tip of the condenser is below the surface of the sulfuric acid, but not touching the bottom.
7. Turn on condenser water. Turn on the block. Make sure the sample ID numbers on the Kjeldahl tubes match the ID numbers on the other end of the condenser.
8. Some sample matrices require the addition of an anti-foaming reagent. If a sample begins to bubble and climb up the sides of the tube, add a few drops of antifoam. If a sample is suspected or expected to foam, antifoam may be added as part of sample pretreatment.
9. After 40mL has been distilled, remove snap caps from under the condensers, and bring up to 50mL with DI water. Refrigerate at 4°C until time of color development on Lachat.

E. Development Procedure on Lachat Autoanalyzer:

1. Refer to SOP #302, Lachat operation, for a more detailed description of instrument use.
2. Turn on power to all modules except pump. Turn on heating unit, set on 65°C, and allow to warm to temperature.
2. Attach ammonia manifold. Be sure to use the 650cm heater block. Refer to manifold diagram if necessary.
3. Load background and generate a sample tray. Be sure to include all sample dilutions and quality control samples (blank, prep blank, LCS, replicates, spikes, check standards, blank spikes) for each form of nitrogen (ammonia, TKN).
4. Use helium to degas all Lachat reagents except for sodium phenolate reagent and sodium nitroprusside.
5. Place feed lines into proper reagent containers. Check that the correct waste container is in place. The sample waste stream is acid waste.
6. Hook pump tubing onto pump tube cassettes, and push into place onto pump. Start pump. Wait while all reagents flow through lines to establish a steady baseline.
9. Place calibration standards in the tray marked S1-S8 in order of descending concentration.
10. Start calibration and samples. The calibration curve correlation coefficient must be 0.9975 or better. If the calibration fails, the standards may have to be remade.
11. Print out calibration statistics and attach it to the last page of the report. Start the run with a free ammonia LCS. Once the run is complete, staple a copy of the lachat run to all associated batch sheets and results sheets. Leave the completed report on the Wet Chemistry supervisor's desk for review.

12. For data transfer, see Appendix A.

VIII. Calculations

A. Calculating RPD:
The RPD is the relative percent difference, and is a measure of the consistency of the sample replicate for the sample set. First, calculate the results for both the sample and the sample replicate. Then apply the two results to the following calculation:

$$\text{RPD, \%} = \frac{(R1 - R2)}{(R1+R2)/2} \times 100$$

where: R1 = value achieved for sample, mg/L
R2 = value achieved for sample replicate, mg/L

B. Calculating the sample spike recovery:
The sample spike is a test if the sample matrix is suitable for accurate testing. To calculate the % recovery, first calculate both the sample and the spiked sample. Then apply the two results to the following calculation:

$$\text{\% Recovery, \%} = \frac{(SR - R)}{STV} \times 100$$

where: SR = value of sample with added spike, mg/L
R = value of sample, mg/L
STV = true value of spike added to sample, mg/L

C. Calculating the LCS:
The LCS is the laboratory control standard and is a test of the accuracy of the calibration and the test overall. To calculate the % recovery of the LCS, calculate the value of the LCS sample, and then apply it to the following calculation:

$$\text{\% Recovery, \%} = \frac{(RL)}{(LTV)} \times 100$$

where: RL = value for LCS sample, mg/L
LTV = true value of LCS, mg/L

D. The Phoenix LIMS automatically calculates all recoveries and RPDs.

IX. Quality Control

A. Prep Blank must be analyzed every batch of 20 or fewer samples. This is a DI water aliquot treated like a sample, undergoing all treatments the samples undergo. The prep blank must yield ammonia concentrations below the reporting limit.

B. Laboratory Control Standards must be analyzed every batch of 20 or fewer samples. It must be recovered 90-110%. A failing LCS for a batch of samples requires re-analysis of the batch.

C. Blank spike must be analyzed every batch of 20 or fewer samples. This is a DI water aliquot fortified by the lab with a known quantity of ammonia. The blank spike must be recovered 90-110% for ammonia and 85-115% for TKN.

D. Continuing calibration checks must be analyzed every 10 samples, as is written into the software of the Lachat QuickChem 8000. They must pass within 90-110%. The calibration check for ammonia is the 1.6mg/L standard (Std C) from the calibration curve.

E. Sample replicates must be done every batch of 20 or fewer samples. They must have a RPD of no more than 20%.

F. Sample matrix spikes must be done every batch of 20 or fewer samples. A known amount of ammonia is added to a sample to test the sample matrix. The spike recovery for ammonia 90-110% and 85-115% for TKN, however, if recoveries fall outside of this range, and the LCS and Blank spike are acceptable, it is judged to be sample related and not system related. Report % recovery.

G. MDL studies are performed annually. Seven replicates of a 0.10 mg/L standard are distilled and analyzed for ammonia. Seven replicates of a 0.50 mg/L standard are distilled and analyzed for TKN. Two MDL spikes are performed quarterly to complete a full MDL over the course of a year. An MDL study must also be performed on prep blanks.

H. A Limit of Detection (LOD) study must be analyzed annually. All sample processing steps of the analytical method shall be included in the determination of the LOD. The LOD study is achieved by analyzing a standard at no more than 2-3x the LOD. In many cases, a 1:1 dilution of the lowest calibration standard is prepared and analyzed. LOD recoveries must be 50 – 150%.

I. Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. 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 portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

J. An initial demonstration of capability (IDOC) must be performed to prove the generation of acceptable data with regard to accuracy and precision. An initial demonstration must be performed by each analyst and consists of analyzing 4 LCS solutions, usually over a series of days.

K. Sample preparations are documented in the Standards Prep Logbook.

L. Reagent preparations are documented in the Reagent Prep Logbook.

M. Lachat use and maintenance is documented in the Lachat Maintenance Logbook.

X. Safety

A. The toxicity and 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 to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.
B. Always wear safety glasses for eye protection as well as lab coats.
C. Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

XI. Pollution Prevention

A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.
B. Standards should be purchased in volumes consistant with laboratory use to minimize the volume of expired standards to be disposed.

XII. Waste Management

A. It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

XIII. Method Performance

A. This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.
B. See section IX Quality Control in this SOP for acceptable limits.

XIV. Corrective Action for Out-of-Control or Unacceptable Data

A. Should the calibration curve have a correlation coefficient of <0.9975, remake and reanalyze curve before processing samples.
B. Should the preparation blank, LCS or inhouse standard fail acceptance criteria, redistill/redigest and reanalyze batch.
C. Should the matrix spike or sample duplicate analysis fail acceptance criteria, a non-conformance report must be generated or the sample QC must be reanalyzed.

XV. References

A. Method 4500-NH3 B. and G., Standard Methods for the Examination of Water and Wastewater, edition, 1997.
B. Method 4500-Norg B., Standard Methods for the Examination of Water and Wastewater, edition, 1997.
C. USEPA Method 350.1, Determination of Ammonia Nitrogen by Semi-automated Colorimetry, Revision 2, August 1993.
D. USEPA Method 351.1, Determination of Total Kjeldahl Nitrogen by Semi-automated Colorimetry, 1978.
E. Methods for QuikChem Automated Ion Analyzer, Lachat Instruments, Method 10-107-06-1-C, August 1993.

XVI. Appendices and Diagrams

A. Appendix A- Data Transfer

Appendix A: Data Transfer to LIMS

From the Phoenix MDE Icon
Left: Data Transfer
Right: IC/Lachat Data Transfer
File Icon (Browse, Local Disk C Drive, Program Files, Lachat, Data, 2015, Data, All Files)
Select Excel File by Date and Time

Enter Initials
Load and Review Data

Edit Data:
Reject samples not needed by checking them (such as Organic Nitrogen)
Look to reject over-range samples that need a diluted sample reported
Make sure that the diluted sample is there (usually at the end of the batch)

Load Data Tab (if edit changes were made)
Load and Review Data (if edit changes were made)

Process Data
After verifying history issues, select sample by checking it under OK column
History issues include: Minimum, Adjusted Maximum, Number of Samples,
All Time Maximum
Results over the All Time Maximum may have to be prepped
(distilled, digested, etc.) again.
History verification issues can usually be ignored from other laboratories, as their location codes are not historically accurate.
All passing reps and spikes should already be selected with a check

Transfer To Labworks
Print Log

Revision History Log for SOP # 304

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
2/6/15	8.1	Changed the range in scope to "0.05ppm", section V.D changed 10 ppm std to 6.0 ppm, and changed 0.04 ppm to 0.05ppm, added dechlorination to section VII.B, added sections H, I, & J to IX (LOD, LCR, IDOC), changed acceptance criteria of LCS, blank spike, & MS to 90-110%, added EPA method references to section XV.	Kathy Cressia	Eric Geyer	2/6/15
3/8/17	8.2	Changed "A" standard to 8.0mg/L, fixed typo in borate buffer reagent, added Appendix A on data transfer	William McKernan	Eric Geyer	3/8/17
4/20/17	8.3	Fixed typo in section IX Quality Control- CCC 90-110% recovery, it said 90-100%	Kathy Cressia	Eric Geyer	4/20/17
7/26/17	8.4	During the 8.2 revision, it was missed in the Scope that the range now goes to 8.0 mg/L.	Kathy Cressia	Eric Geyer	7/26/17
7/26/19	8.5	Section IX.C & F – Added spike recovery range for TKN. Section IX.G. – Added information on MDL studies.	Kathy Cressia	Eric Geyer	7/26/19

Expiration Date

Title:	**Enterococcus Multi-well QuantiTray Enzyme Substrate Method**	Effective Date: 5/3/21
		Dept. Test Method

Sharyn B. Lawler	04/30/21	Jan Guillemette	05/03/21	Quinn B Faulk	4/30/21
Prepared By:	Date:	Dept. Approval By:	Date:	QA Approval By:	Date:

1.0 SCOPE AND APPLICABILITY

BAL Laboratory uses the Multi-well procedure (QuantiTray 2000) Enterolert (IDEXX) method to detect *Enterococci* bacteria. This method allows for the use of a single medium to detect *Enterococci* bacteria in a 24-48 hour period of time. Enterolert is recommended for analysis of drinking water, source water, wastewater, and both fresh and marine recreational waters.

2.0 SUMMARY OF METHOD

The fluorogenic enzyme β-D-glucosidase test uses a hydrolysable substrate to detect enterococci. The matrix allows the enterococci to express the enzyme while preventing non-enterococci from yielding detectable fluorescence.

3.0 HEALTH AND SAFETY WARNINGS

Enterolert does not utilize toxic or mutagenic agents so it is a much safer alternative to other test media.

Exercise caution when dealing with any samples. Wear protective gloves. Be mindful of incubators and autoclave hot surfaces. Wear protective eyewear when using UV lights in associated analyses. Lab coats and lab glasses are advised.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Samples are kept on ice (<8°C, not frozen) and brought to the laboratory for analysis preferably within six (6) hours of collection. Regulatory samples must be prepared/analyzed within 8 hours of collection. Non-Regulatory samples may be analyzed up to 24 hours from time of collection.

4.2 Thoroughly mix all samples or dilutions prior to use by rapidly making about 25 complete up and down (or back and forth) movements. Optionally, use a mechanical shaker to shake samples or dilutions for 15 seconds.

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5.0 SAMPLE COLLECTION

Samples are collected using aseptic technique with sterile glass or plastic containers. Suggested sample volumes are between 250 and 500mL. A minimum of 100mL is required. Follow the guidelines described in Standard Methods for the Examination of Water and Waste Water, for sample collection, storage, and dechlorination.

6.0 INTERFERENCES, CAUTIONS AND POTENTIAL PROBLEMS

If analyzing marine water samples (salt or brackish), the samples must be diluted at least tenfold (1:10) with sterile DI water. Multiply MPN value by the dilution factor to obtain proper quantitative results.

Non-enterococcus bacteria that produce enzyme β-D-glucosidase, such as some species of the genera *Serratia, Klebsiella,* and *Aerococcus*, are suppressed; they should not produce a positive response unless there are more than 10^5 CFU/100mL.

7.0 EQUIPMENT AND SUPPLIES

7.1 Enterolert media - Store Enterolert media at 2˚C to 30˚C away from light. Refer to expiration date of the product package to determine the shelf life. Do not expose to temperatures greater than 30˚C for extended periods of time. Prolonged exposure to elevated temperatures can affect stability.

7.2 IDEXX Quanti-Tray/2000

7.3 Quanti-Tray Rubber Insert

7.4 Quanti-Tray Sealer Plus

8.0 PROCEDURE

8.1 Allow Enterolert dehydrated medium and samples to equilibrate at room temperature (20ºC to 30ºC).

8.2 Add 100 mL of the sample to the sterile test bottle. Open the packet of dehydrated Enterolert medium and add to the test bottle with the sample.

8.3 Shake and invert the bottle to thoroughly mix the contents. Do not proceed to the next step until the Enterolert media is completely rehydrated.

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8.4 Holding the tray at an angle with the wells facing down, carefully squeeze open the multi-well tray and gently pour the contents of the sterile sample bottle (sample and Enterolert medium thoroughly mixed together) into the tray trying to avoid foaming.

8.5 Place the filled Quanti-Tray into the rubber insert matching the wells of the Quanti-Tray into the corresponding recesses in the rubber insert.

8.6 Place tray and rubber insert into the tray sealer, gently push the tray into the feed chute until the machine catches and allow the sample to be sealed.

8.7 Mark the Quanti-Tray with the sample number and incubate the sealed tray at 41± 0.5˚C for 24±2 hours.

9.0 CALCULATIONS AND INTERPRETATION OF DATA

9.1 After incubation, examine each tray for fluorescence using a long wave (366nm) ultraviolet (UV) light. Place light within 5 inches of the sample in a dark environment. Count the number of large and small wells that indicate fluorescence (bright white/blue color indicates positive fluorescence).

9.2 Using the "IDEXX Quanti-Tray/2000 MPN Table" determine the Most Probable Number of *Enterococcus.* Multiply MPN value by the dilution factor (if used) to obtain proper quantitative results.

9.3 Report the corresponding MPN/100mL.

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1. With each group of samples set up 2 additional control samples for verification. To each bottle add 100 mL of sterile water, Enterolert dehydrated medium, and

Bottle 1. Positive control: *Enterococcus faecium*
Bottle 2. Negative control (gram negative): *Serratia marcescens*

10.2. Once a month perform a leakage test on the QuantiTray sealer by adding 10 drops of liquid food coloring to 100 ml of clear water and sealing tray using normal procedure. Apply slight pressure to the sealed tray and check for leaks between cells and into neighboring cells. On every new batch of bottles preform a fluorescence test (on an un-inoculated bottle) by adding sterile water to the bottle and checking under UV light for autoflourescence. This test will rule out a bad batch of bottle by outside contamination.

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11.0 DATA VALIDATION

Excel Bench sheets are used to record all observed data. After the analyst has completed the analysis and all data entry on the bench sheet, the data are ready for entry into Element. Once in Element, they are reviewed by the Lab Manager or delegate and the data is locked. Data are then sent on for Final Report processing.

12.0 DATA AND RECORDS MANAGEMENT

Bench sheets used for this analysis are found at Q:\BAL\ Btch Sheets\. They are copied into the appropriate Year and Month folder and renamed using the batch ID such as Q:\BAL\Bitch Sheets\2021\03 March 2021\030921\DC10916 Enterolert QT. More details about bench sheets can be found in SOP A006 (current version).

13.0 REFERENCES

Enterolert is included in the on-line version of *Standard Methods for the Examination of Water and Wastewater* as the Fluorogenic Substrate Enterococcus Test (9230D) for the detection and enumeration of enterococci in water samples. The Multiple well using Quanti-Tray® or Quanti-Tray®/2000 method is section 9230D *b.*

ASTM D6503-99 Standard Test Method for Enterococci in Water Using Enterolert.

14.0 POLLUTION PREVENTION and WASTE MANAGEMENT

All disposable microbiological lab ware is considered medical waste by the state of Rhode Island Department of Health; including pipettes, petri dishes, test tubes, and syringes, etc. We have contracted with United Medical Waste Management to pick up medical wastes on a regular basis and to transport it to an appropriate medical waste disposal facility. All used media, test tubes, pipettes, sample containers, etc. are placed in the appropriate Biohazard Bins supplied by UMWM. It is not necessary to sterilize the contaminated lab ware prior to disposal since it will be incinerated at the waste disposal facility. Our Medical Waste Generator number is MAD991302266.

All leftover sample volume/aliquots are disposed of per work instruction WI009 (current revision).

15.0 DEFINITIONS

15.1 Enterococci include *Enterococcus faecalis*, *Enteroococcus faecium*, *Enterococcus avium*, and their variants.

15.2 Verification – a general process used to determine whether the microbiological analytical method is performing as expected to provide reliable data.

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15.3 MPN – Most Probable Number, otherwise known as the method of Poisson zeroes, is a method of getting quantitative data on concentrations of discrete items from positive/negative (incidence) data.

16.0 PERSONNEL QUALIFICATIONS

Staff must be fully trained and have read all appropriate SOPs prior to performing this analysis. In addition, staff must complete an Initial Demonstration of Capability and have passed associated QC prior to analysis of client's samples. All analysts shall possess a minimum of a high school diploma or equivalent.

17.0 TROUBLESHOOTING

Problems with the incubator and/or the autoclave should be addressed to the Lab Manager for possible service call. BAL maintains a service contract with DESCO to maintain them in working order.

18.0 ATTACHMENTS

N/A

19.0 REVISION HISTORY

Revision#	Type (full/minor)	Date	Description
0			Was BAL-ALP-SOP08
1	F	4/30/21	Updated to excel logs and removed Attachment A

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Fax: 401-461-4486
www.esslaboratory.com

CHAIN OF CUSTODY

ESS Lab # | Page 1 of 1

Turn Time (Days) ☐ > 5 ☐ 5 ☐ 4 ☐ 3 ☐ 2 ☐ 1 ☐ Same Day

Regulatory State: **Criteria:**

Is this project for any of the following?:

☐ CT RCP ☐ MA MCP ☐ RGP ☐ Permit ☐ 401 WQ

ELECTRONIC DELIVERABLES (Final Reports are PDF)

☐ Limit Checker ☐ State Forms ☐ EQuIS
☐ Excel ☐ Hard Copy ☐ Enviro Data
☐ CLP-Like Package ☐ Other (Specify) →

CLIENT INFORMATION

Client:
Address:
Phone:
Email Distribution List:

PROJECT INFORMATION

Project Name:
Project Location:
Project Number:
Project Manager:
Bill to:
PO#:
Quote#:

Client acknowledges that sampling is compliant with all EPA / State regulatory programs

REQUESTED ANALYSES

ESS Lab ID	Collection Date	Collection Time	Sample Type	Sample Matrix	Sample ID	Total Number of Bottles

Container Type: AC-Air Cassette AG-Amber Glass B-BOD Bottle C-Cubitainer J-Jar O-Other P-Poly S-Sterile V-Vial

Container Volume: 1-100 mL 2-2.5 gal 3-250 mL 4-300 mL 5-500 mL 6-1L 7-VOA 8-2 oz 9-4 oz 10-8 oz 11-Other*

Preservation Code: 1-Non Preserved 2-HCl 3-H2SO4 4-HNO3 5-NaOH 6-Methanol 7-Na2S2O3 8-ZnAce, NaOH 9-NH4Cl 10-DI H2O 11-Other*

Sampled by :

Chain needs to be filled out neatly and completely for on time delivery.

Laboratory Use Only

Cooler Temperature (°C): _____

Comments: *** Please specify "Other" preservative and containers types in this space**

All samples submitted are subject to ESS Laboratory's payment terms and conditions.

Dissolved Filtration

☐ Lab Filter

Relinquished by (Signature)	Date	Time	Received by (Signature)	Relinquished by (Signature)	Date	Time	Received by (Signature)

Relinquished by (Signature)	Date	Time	Received by (Signature)	Relinquished by (Signature)	Date	Time	Received by (Signature)

Appendix C – Middletown Record of Flooding Spreadsheet

Maidford River Restoration Pilot Project
Road Flooding Tracking Sheet

Date	Location of Flooding (Berkeley Avenue and/or Berkeley Avenue Extension)	Was a road closure required? (Yes or No)	Road closure duration (hours)	Were emergency vehicles diverted from using Berkeley Avenue due to the road closure? (Yes, No, Don't Know)	Did the Town have to deploy equipment to clear the road of sediment or other debris post-storm? (Yes, No)	Rainfall amount leading to flooded road condition (inches)	Notes (e.g. severity of flooding, length of road impacted by flooding)

Appendix D – Program Measure Tracking Sheet

Maidford River Restoration Pilot Project
Program Measure Tracking Sheet

	Partner Organization		Monitoring Program		Event		Volunteer		Funding			
Record #	Partner Organization Name	Partner Organization Date Joined	Monitoring Program Name	Monitoring Program Date Initiated	Event Name	Event Date	Volunteer Name	Date Volunteered	Funding Source	Funding Type	Funding Amount	Award Date
1												
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Appendix E – QAPP Acknowledgment Form

Quality Assurance Project Plan Acknowledgment and Commitment to Implement For

Maidford River Restoration Pilot Project
Monitoring Program

This form shall be completed by all organizations and/or new key personnel who were not original signatories to the QAPP.

I hereby certify that I have read and understand the QAPP for this project. I agree to adhere to the requirements of this document and contact the Project Manager if I become aware of deviations from the QAPP.

_____

Name

_____

Position/Title

_____

Affiliation

_____

Signature Date

Please return this form to the Project Manager and maintain a copy for your records.