Form DEPLW0604-B
Created: November 21, 2013
Revised: February 21, 2018

Maine Department of Environmental Protection

NET PEN AQUACULTURE
Supplemental Application Form

This form must be attached to the General Application for a Waste Discharge License / MEPDES Permit (Form DEPLW0105-B2003)

1. Facility Information

Name of Facility: Site FB01 (Long Porcupine Island) DMR Site Designation:

Town/City: Gouldsboro Water Body: Frenchman Bay

Facility Owner Name: American Aquafarms Inc.

Mailing Address: 68 Commercial Street, Portland, ME 04101

Daytime Telephone Number: (207) 405-7451 e-mail: thomas.j.brennan@americanaquafarms.com

Operator if different:

Mailing Address:

Daytime Phone Number: e-mail:

2. Net Pen, Fish Rearing and Oceanographic Conditions

a. Species of fish being reared Atlantic salmon (Common name) Salmo salar (Latin name)

Type of Net Pen	Net Pen Description	Maximum Number of Each Pen Type in Use	Net Pen Dimensions (meters)	Maximum Number of Fish per Pen	Maximum Biomass of Fish per Pen (kg)	Maximum Density of Fish per Pen (kg/M^3)
1	Ecopen; see Att. NPA-1	15	47m diameter, 36m deep	742,000	1,200,000	40
2						
3						
Total for Facility		15		4.06 million	9,200 MT	

b) What is the minimum clearance from net pen to the sea floor? 10.7 meters.

c) What is the average current velocity? See Att. NPA-2 cm/sec.

3. Feeding Conditions

a) What is the composition of fish feed, including additives? See Att. NPA-3

b) What is the maximum feeding rate? 1,418,341 kg/month.

c) What is the total quantity of feed used per year? 13,500,000 kg.

d) Describe the method(s) and/or system(s) used to dispense feed and to detect the loss of uneaten feed.

See Att. NPA-3

4. Chemical Use (disinfectants, biocides, anti-fouling agents and similar compounds)

Name of Compound	Purpose	Quantity Used per Application	Frequency of Use
See Att. NPA-4			

5. Medication Use

Name of Compound	Dosage	Duration of Use	Frequency of Use	Route of Administration	Prophylactic Use (Yes/No)
See Att. NPA-4					

6. Baseline Monitoring

Has this site been used for finfish aquaculture within the last 5 years? YES ☐ NO ☒

If NO, submit new baseline monitoring data. See Attachment NPA-5.

7. Spill Prevention and Countermeasure Plan

Is a current Spill Control and Countermeasure Plan available onsite for review? YES ☐ NO ☒

If YES, provide the date of last revision: N/A - Mar Fortune SPCC Attachment NPA-6.

If NO, the facility must provide the Department with an opportunity to review a current plan prior to operating the facility.

8. Additional Submissions Required

a) Submit a site map of the leasehold area which contains the following information: See NPA Supplemental Figure 1

- ☐ lease boundaries
- ☐ location of mooring systems
- ☐ location of support platforms
- ☐ direction of prevailing currents
- ☐ local landmarks
- ☐ representative water depths (as MLW)
- ☐ configuration and location of net pens
- ☐ proposed net pen and reference site sampling locations and GPS coordinates

b) Submit evidence of an existing leasehold or completed application for a leasehold from the Maine Department of Marine Resources. American Aquafarms submitted a draft Standard Lease Application to the Maine Department of Marine Resources in March 2021.

c) Submit evidence of the facility's permit from the U.S. Army Corps of Engineers.

American Aquafarms' draft Standard Lease Application also serves as application for a permit from the U.S. Army Corps of Engineers.

ATTACHMENT NPA-1
NET PEN DESCRIPTION

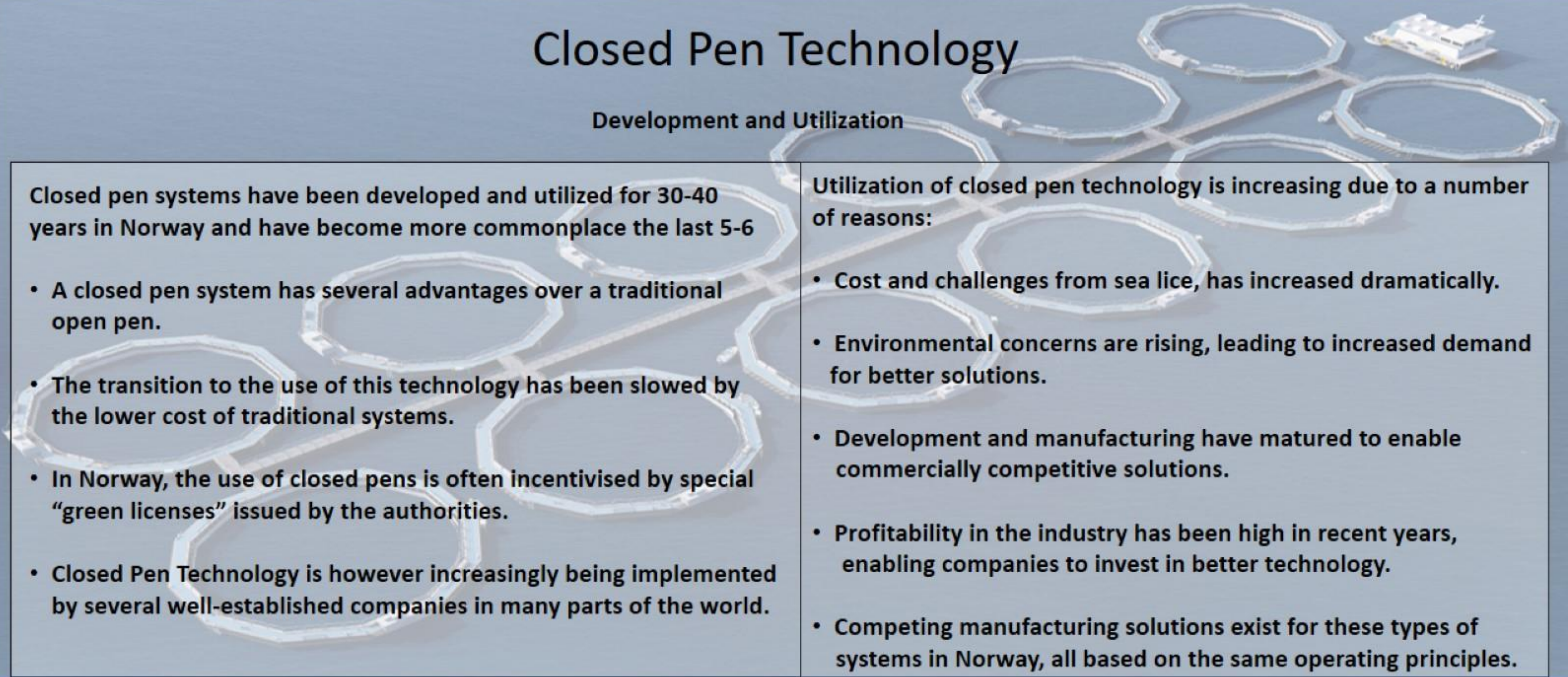
DESCRIPTION OF CLOSED-PEN SYSTEM

The Eco-pen from American Aquafarms is a floating, closed containment aquaculture production system. The floating collar is made from rigid aluminium and holds a fabric sack made from an ultra-strong impermeable polymer membrane. Inside the fabric sack is an ordinary net pen for extra security and easy handling of the fish. This creates two barriers of separation to surroundings. Water is collected at depth (estimated at 30 meters) through six separate pipes and pumped into the pen creating a circular current. The water temperature inside the Eco-pen can be optimized by collecting water from varying depths according to hydrographic conditions. The intake of water is depth adjustable and will be optimized to collect water according to desired temperature and other variables. Different and interchangeable filters can be temporarily fitted to the intake pipes to control intake water for the prevention of algae or jelly fish and other, if needed. The main outlet of the pen is located at 30 m and is fixed. By lifting the water column inside the pen between 4-15 cm (adjustable) above surrounding sea-level, the pumping action creates overpressure inside the pen. This ensures a stable and rigid placement of the polymer sack in the water column, that makes it able to withstand external pressures from weather and currents.

Oxygen is continuously added to the inflowing water by diffusors creating optimal and stable environment for fish health. The result is zero lice, high survival, lower use of feed and high growth. Waste from excess feed and fish excretions, will travel with the main water flow towards the bottom of the pen. The solids settle in a sedimentation trap/funnel that is 6 m below the primary outlet. Accumulated waste is continuously pumped from the bottom of the pen to the top of the collar by a specialized pump system, before being transported to a central waste treatment station on a waste barge where it is pressed and dried before being transported to shore for further processing. Dead fish are automatically collected from the bottom of the inside net and pumped to an ensilage system. Electricity and oxygen supply is secured through a three-level backup system. For further illustration, see the attached illustrations.

Closed Pen Technology

Development and Utilization



Closed pen systems have been developed and utilized for 30-40 years in Norway and have become more commonplace the last 5-6

- A closed pen system has several advantages over a traditional open pen.
- The transition to the use of this technology has been slowed by the lower cost of traditional systems.
- In Norway, the use of closed pens is often incentivised by special "green licenses" issued by the authorities.
- Closed Pen Technology is however increasingly being implemented by several well-established companies in many parts of the world.

Utilization of closed pen technology is increasing due to a number of reasons:

- Cost and challenges from sea lice, has increased dramatically.
- Environmental concerns are rising, leading to increased demand for better solutions.
- Development and manufacturing have matured to enable commercially competitive solutions.
- Profitability in the industry has been high in recent years, enabling companies to invest in better technology.
- Competing manufacturing solutions exist for these types of systems in Norway, all based on the same operating principles.

Closed Pen Technology is increasingly used as the preferred method of production.

American aquafarms

NEXT GENERATION FISH FARMS

- Escapes
- Medicines and Pesticides
- Microplastics
- Predators
- Sea lice and parasites
- Untreated fish waste

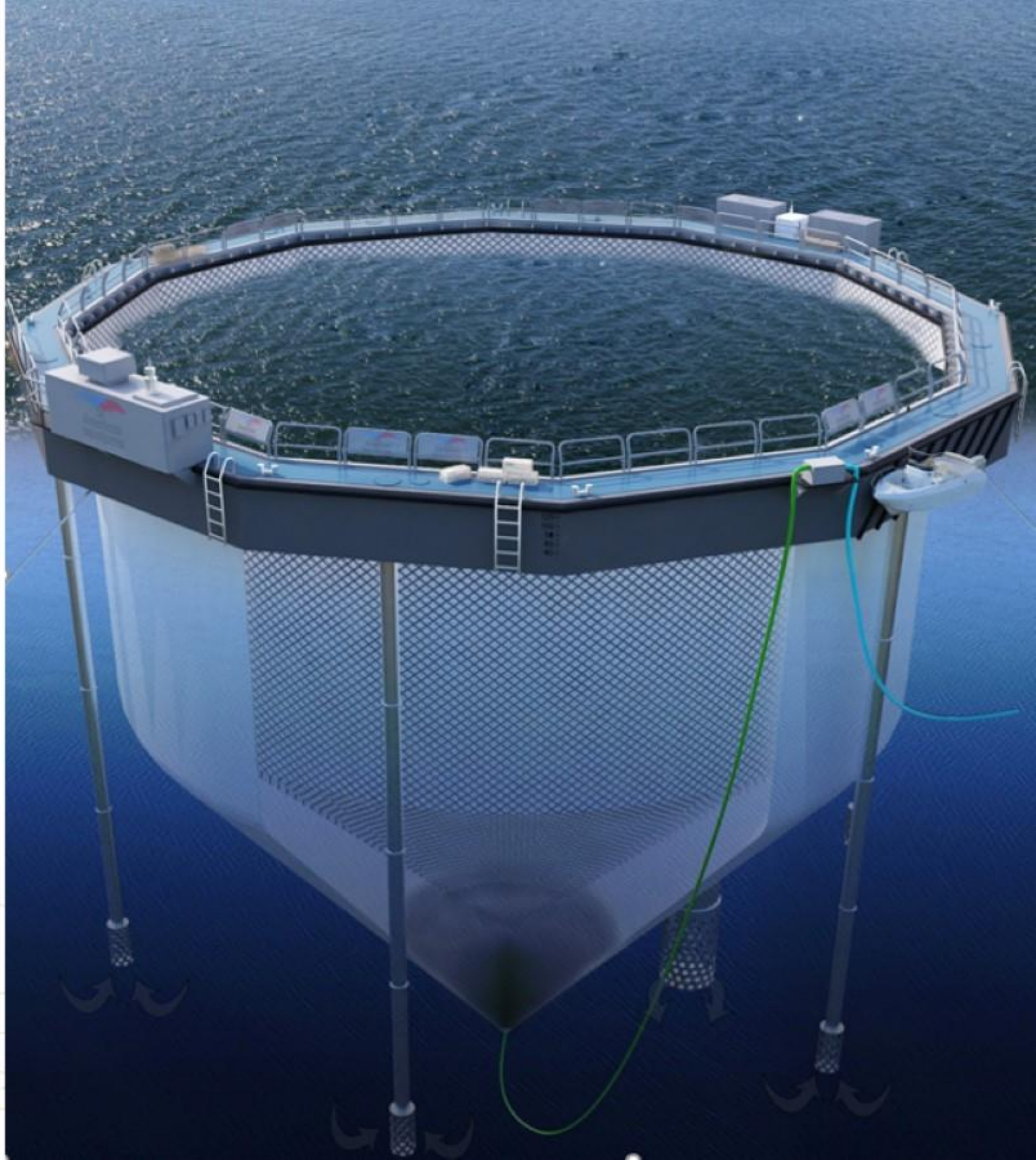
CLOSED PEN SYSTEMS

American
aquafarms

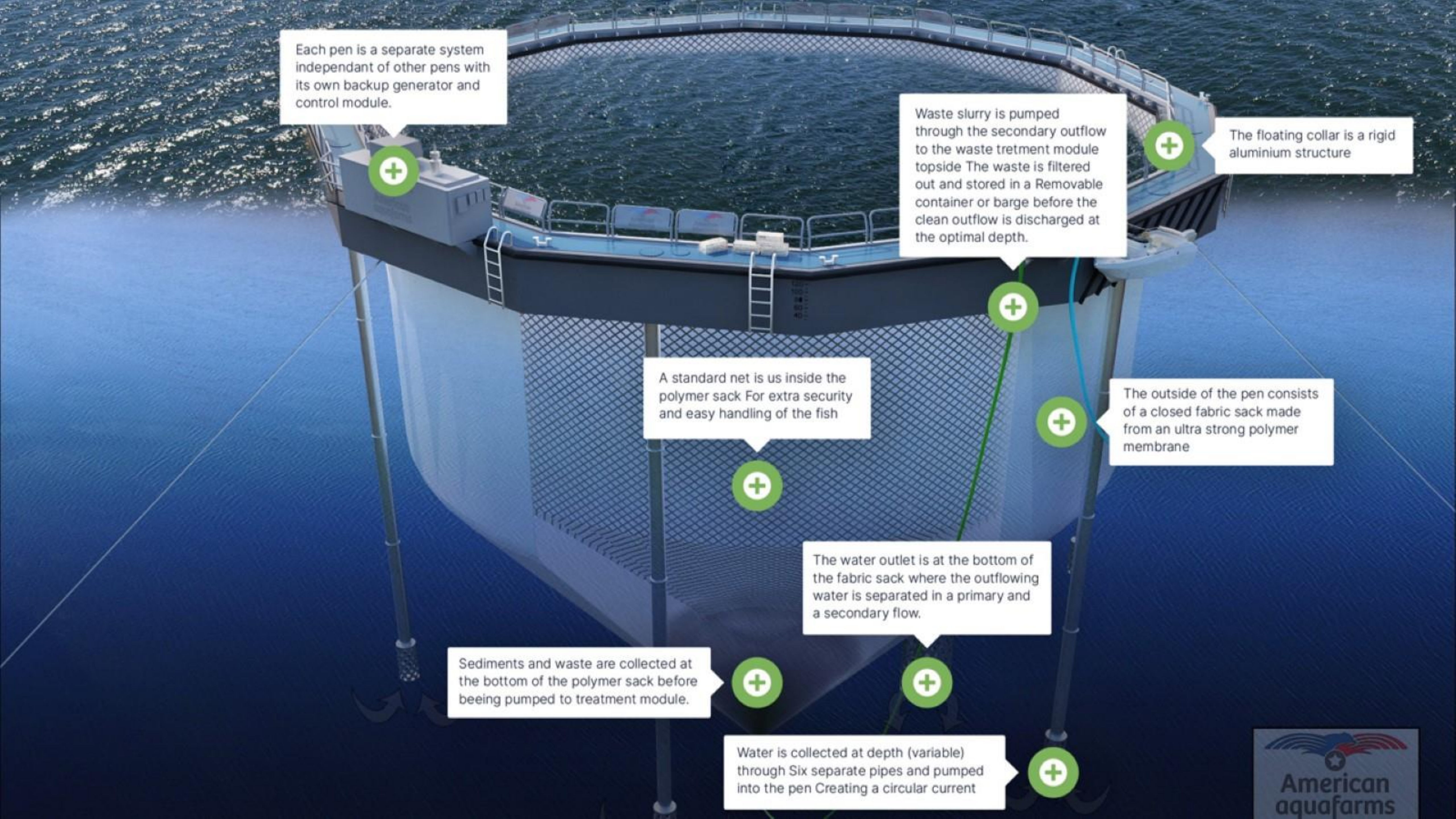
Closed pen technology removes most of conventional industry issues, and is a modern day solution to age-old problems.

CLOSED PEN TECHNOLOGY

- A proven and robust technology that has significant less risks than traditional open net pen operations
- Controls waste from reaching the ocean
- Eliminate fish escapes
- Reduces the need for medicine/chemicals
- Eliminates sea lice
- Prevents predators



American aquafarms



Each pen is a separate system independant of other pens with its own backup generator and control module.

Waste slurry is pumped through the secondary outflow to the waste tretment module topside The waste is filtered out and stored in a Removable container or barge before the clean outflow is discharged at the optimal depth.

The floating collar is a rigid aluminium structure

A standard net is us inside the polymer sack For extra security and easy handling of the fish

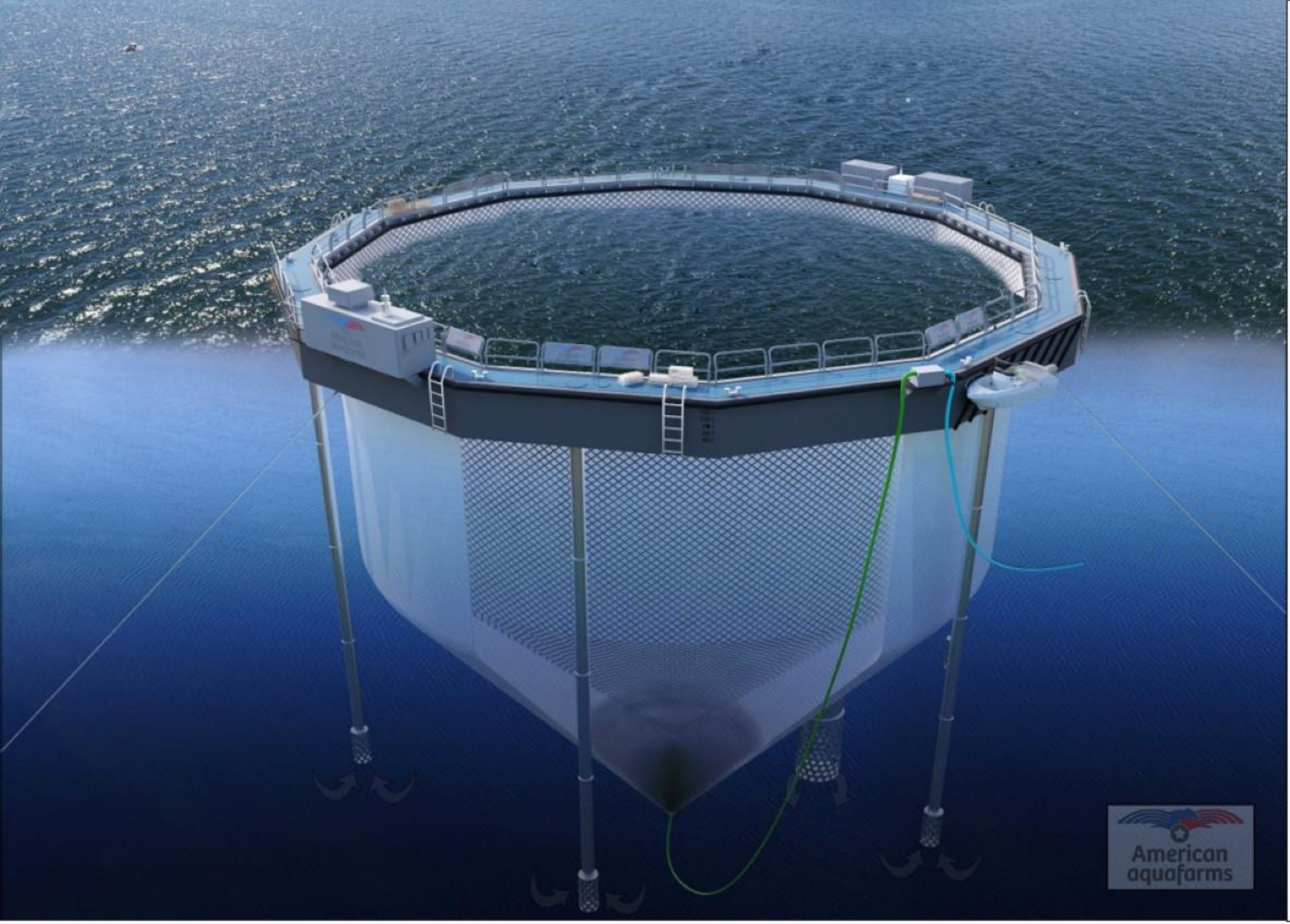
The outside of the pen consists of a closed fabric sack made from an ultra strong polymer membrane

The water outlet is at the bottom of the fabric sack where the outflowing water is separated in a primary and a secondary flow.

Sediments and waste are collected at the bottom of the polymer sack before beeing pumped to treatment module.

Water is collected at depth (variable) through Six separate pipes and pumped into the pen Creating a circular current

American aquafarms



Key measurements

Height above waterline, collar: 1m

Height above waterline, container: 4 m

Outside diameter: 47 m

Depth below waterline: 36 m

Outflow diameter: 2,8 m

Polymer membrane thickness: 3 mm

Sediment trap height: 6 m

Secondary outflow: 12 cm

Waste treatment module

Inside net: traditional

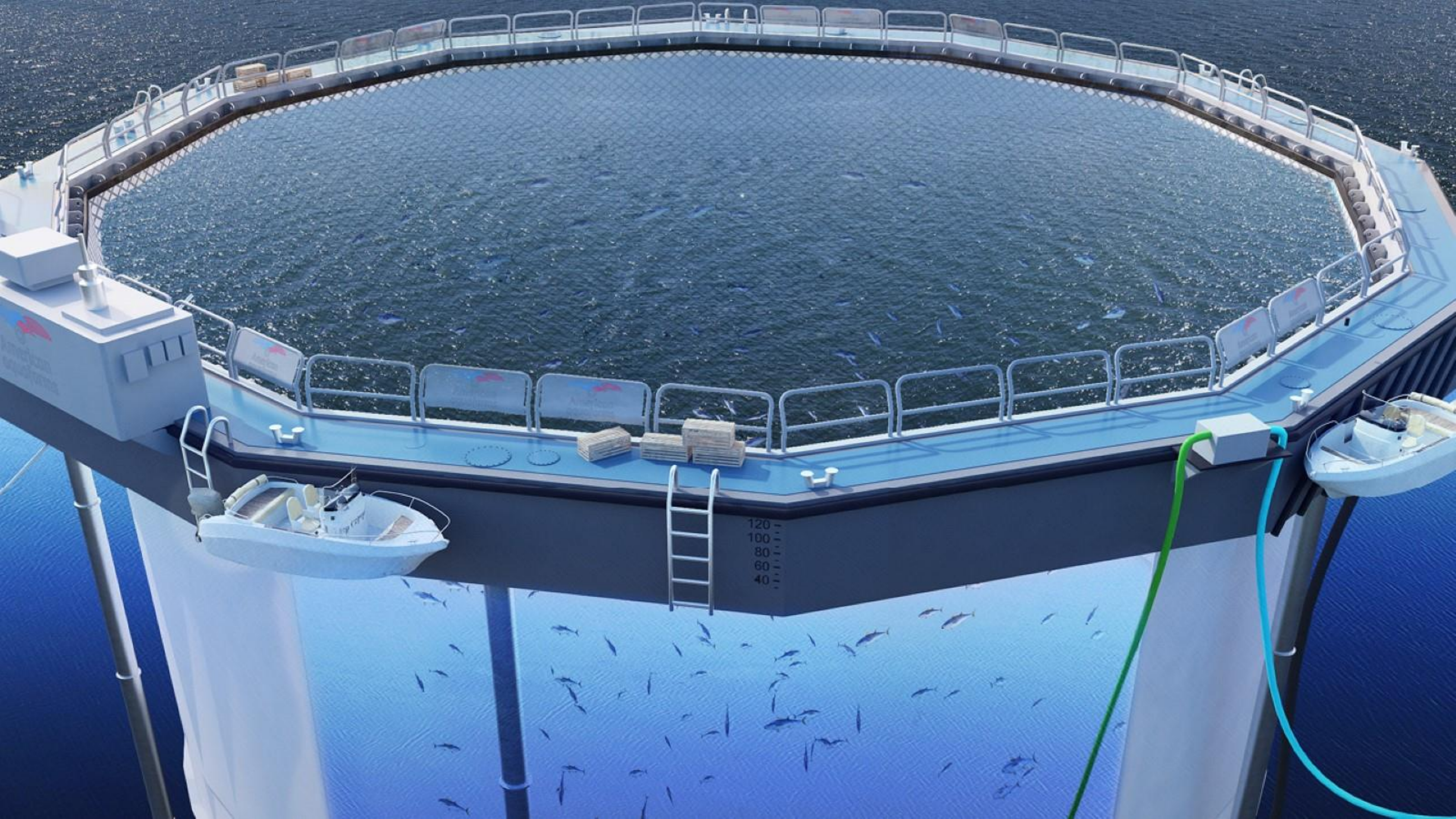
Collar: Rigid welded aluminium

Flow rate: $1 m^3/s$ pr pump total $6 m^3/s$

Overpressure: 10 cm height inside pen

Intake Pipe diameter: 1 m, Length: 20-40 m

Volume: 30 000 m^3


American Aquafarms
120
100
80
60
40

RANSOM

112 Corporate Drive
Portsmouth, NH 03801
603.436.1490

MEMO

Date: April 8, 2021

To: American Aquafarms
68 Commercial Street
Portland, Maine

From: Thomas B. Neilson, L.G.

Subject: Current Assessment
Proposed Closed-Pen Aquaculture Site
Long Porcupine (FB01)

Project No.: 201.05012

Hydrography

An acoustic doppler current profiler (ADCP) was deployed at the site to measure currents over the course of a full tidal cycle during the baseline survey with the objective of measuring the current speed and direction at the location of the proposed closed-pen system. The location of the ADCP and approximate direction of ebb and flood currents are shown in Figure 1, attached here. Current direction shown on Figure 1 is an approximation based on the results presented below and generally represents maximum ebb and flood directions in the near-surface water.

Deployment Methodology and Data Processing

To measure current speed and direction, an RD Instruments (Teledyne Marine) 300khz Workhorse Sentinel ADCP mounted in an Open Seas Instrumentation SUBS A2 ADCP buoy was deployed in an upward-looking configuration. The ADCP was deployed with a 175 lb. mooring and acoustic release at a depth of approximately -53.09 meters MLLW. The instrument was located approximately 2.70 meters above the sea floor, and was configured to collect current magnitude, direction, depth, and temperature in 1-meter bins throughout the water column at 6-minute intervals. The first bin was located 3.22 meters from the instrument, which corresponds to a depth of -47.17 meters MLLW. The ADCP was located at 44.420950° N, -68.160250° W (WGS84) and deployed on September 16, 2020, began recording at 11:54, and was recovered on September 17, 2020 with a final measurement at 10:42. The total deployment duration was 23 hours 6 minutes.

The ADCP was configured immediately prior to deployment according to the manufacturer's configuration process, which included calibrating the pressure transducer, compass, and acoustic transducers. Raw data from the ADCP were post-processed to account for a magnetic variation of 15.85° W. The sea surface elevation was accounted for by identifying all bins that were further from the instrument than the depth of the water, as determined by the on-board pressure transducer at the time of the current reading. Measurements within these out-of-range bins were set to null in the dataset.

The nearest tide station to the deployment site is located in Bar Harbor (NOAA Station 8413320) and is approximately 3 nautical miles southeast of the lease area. Tidal observations reported by NOAA were used to further process the ADCP data. The time of shallowest water at the ADCP location was found to coincide exactly with the time of observed low tide at the tide station, so no temporal correction was applied to account for delay in tide height. The instrument elevation relative to MLLW was calculated by subtracting the minimum depth observed at the ADCP from the minimum observed tide height at the station in meters MLLW, which was equal to -50.37 meters MLLW. This offset was used to reference all current measurements to MLLW. Tidal period was also classified as "ebb" or "flood" for the full tidal cycle (low to low) that was recorded during the deployment to aid in interpretation of current observations.

After post-processing, data were sub-sampled and averaged to provide a mean current velocity at approximate depths of -1 to -5 meters relative to the sea surface, -35 to -39 meters MLLW (approximate bottom of the pens), and from -43 to -47 meters MLLW[1] for each current reading. Depth-averaged data are plotted in Figure 2, showing the directions of the all ebb and flood measurements as well as the maximum ebb and flood measurements for each depth-averaged interval.

Mean Current Velocity Results

Current velocity data are presented in several ways. Depth-averaged data are plotted in Figure 2, showing the directions of the ebb and flood measurements as well as the maximum ebb and flood measurements for each depth-averaged interval. Figure 3 shows depth-averaged current velocity and current direction over time, as well as tide height observations from the Bar Harbor tide station.

The results for maximum and mean ebb and flood current velocity from each depth-averaged interval are presented in the tables below:

---

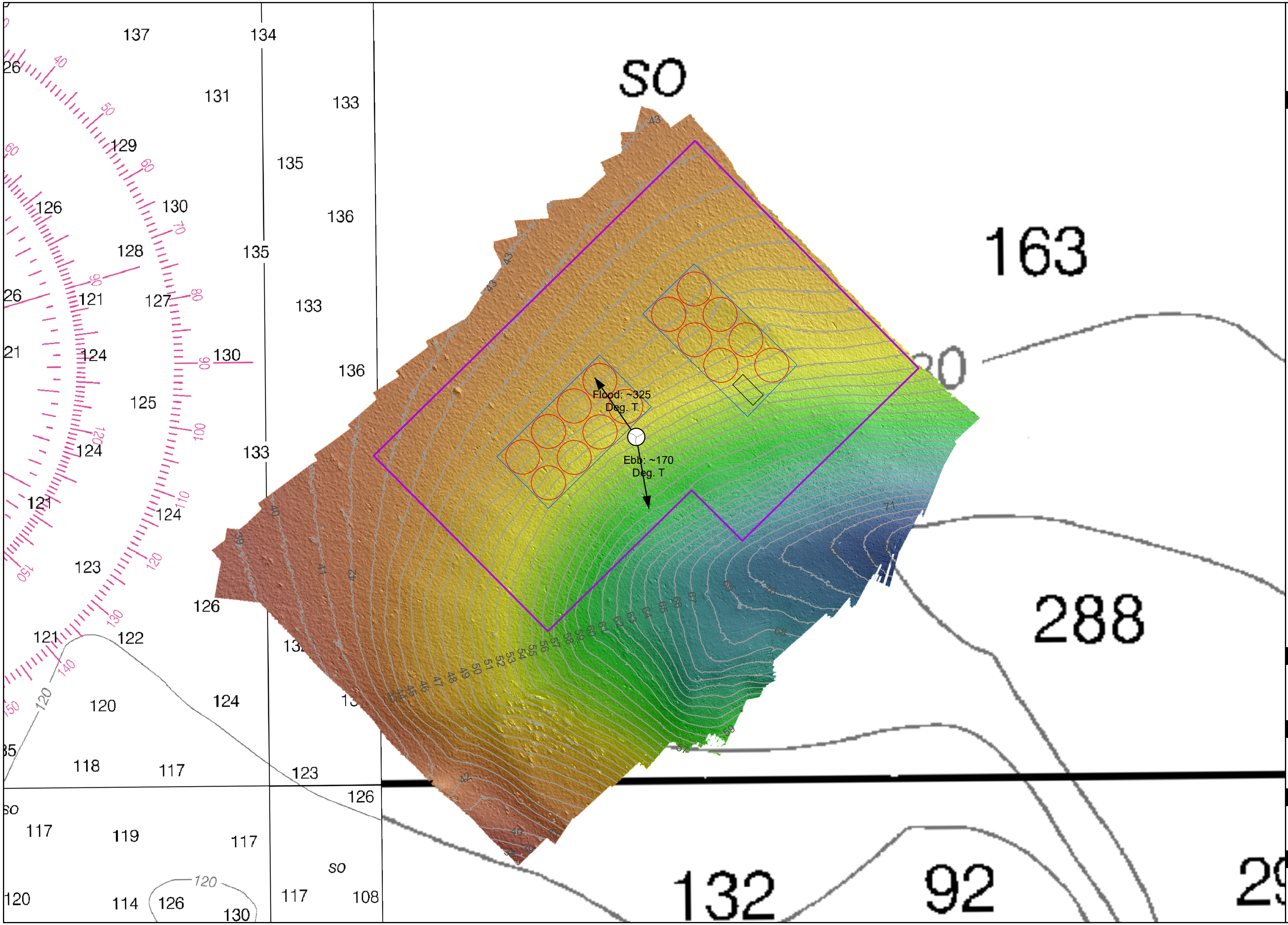
[1] -43 to –47 meters MLLW is the closest measurement to the ocean floor that is technically feasible.

American Aquafarms.

Measurement	**Maximum Ebb Magnitude (cm/s)**	**Maximum Ebb Direction (degrees true)**	**Maximum Flood Magnitude (cm/s)**	**Maximum Flood Direction (degrees true)**
Mean, Surface (-1 to -5 m below surface)	42.77	118	38.23	101
Mean, Pen Depth (-35 to -39 m MLLW)	20.2	174	16.76	298
Mean, Bottom (-43 to -47 m MLLW)	28.22	148	15.7	322

Measurement	**Mean Ebb Magnitude (cm/s)**	**Mean Ebb Direction (degrees true)**	**Mean Flood Magnitude (cm/s)**	**Mean Flood Direction (degrees true)**
Mean, Surface (-1 to -5 m below surface)	26.72	146	30.34	122
Mean, Pen Depth (-35 to -39 m MLLW)	8.39	153	10.40	283
Mean, Bottom (-43 to -47 m MLLW)	14.12	159	10.86	267

Tidal currents observed at the site generally indicate semi-rotary type currents rather than strictly bi-directional or reversing current. Current near the surface appears to have been heavily influenced by wind during the deployment, which was gusting to 35 knots from the west. Surface currents were only measured flowing from northeast to southeast, even during flood, which is considered to not be representative of true surface current, which is expected to flow in similar directions to current at depth. Current at the bottom of the pens and near the bottom were similar, directionally, with primary flood flow to the northwest and ebb flow to the south-southwest. Maximum current velocity was measured near the surface (42.77 cm/s or 0.83 knots), and generally decreased with depth with the lowest maximum tidal velocity observed near the sea floor on the flood tide (15.70 cm/s or 0.31 knots). Ebb currents were higher than flood currents at all depths.



RANSOM
Consulting
Engineers
and Scientists

Legend & Notes

- Lease Boundary
- Mooring Grid Boundary
- Pen Outline
- Barge Outline
- ADCP Location
- Bathy. Contour (m MLLW)

Bathymetry (m MLLW)
High : -37
Low : -75

General Notes:

1. Basemap from NOAA RNC server.
2. Some features shown are approximate in location and scale.
3. This map was prepared for American Aquafarms. All other uses are not permitted unless written permission is obtained from Ransom Consulting, LLC.

Scale & Orientation

0 200 400
1 inch = 400 feet

Prepared For

American Aquafarms
68 Commercial Street
Portland, Maine

Site Address

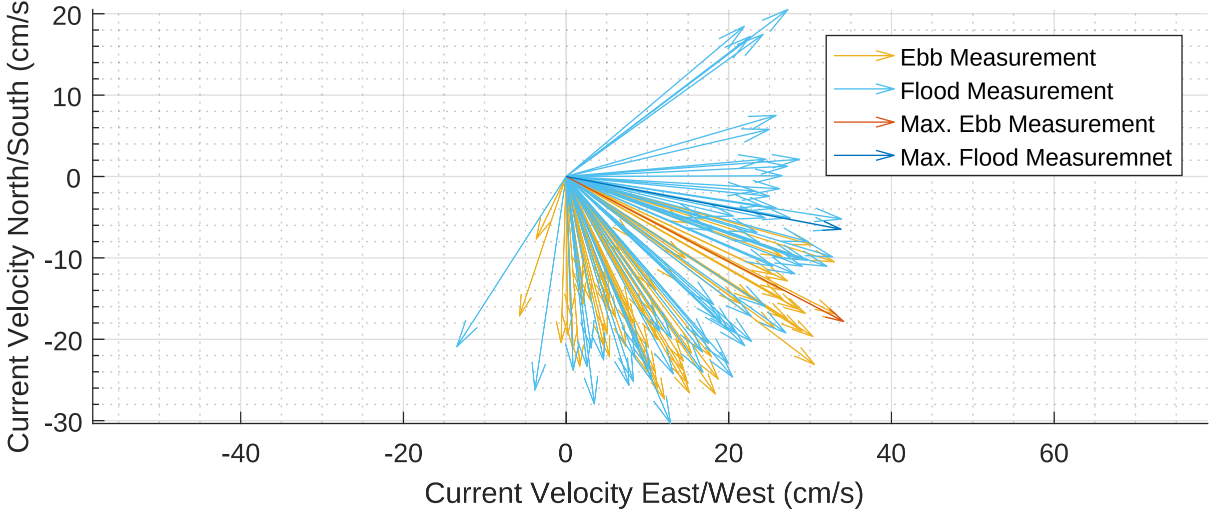
Long Porcupine (FB01)
Frenchman Bay
Gouldsboro, Maine

201.05012 | Apr 2021

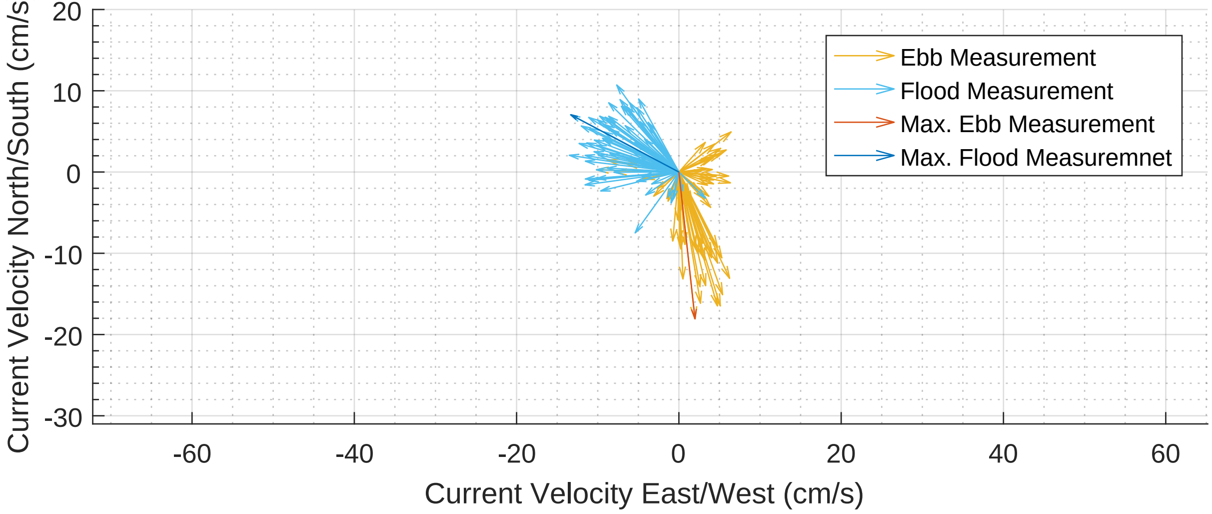
NPA Attachment 2
Figure 1
ADCP and Currents

NPA Attachment 2: Figure 2

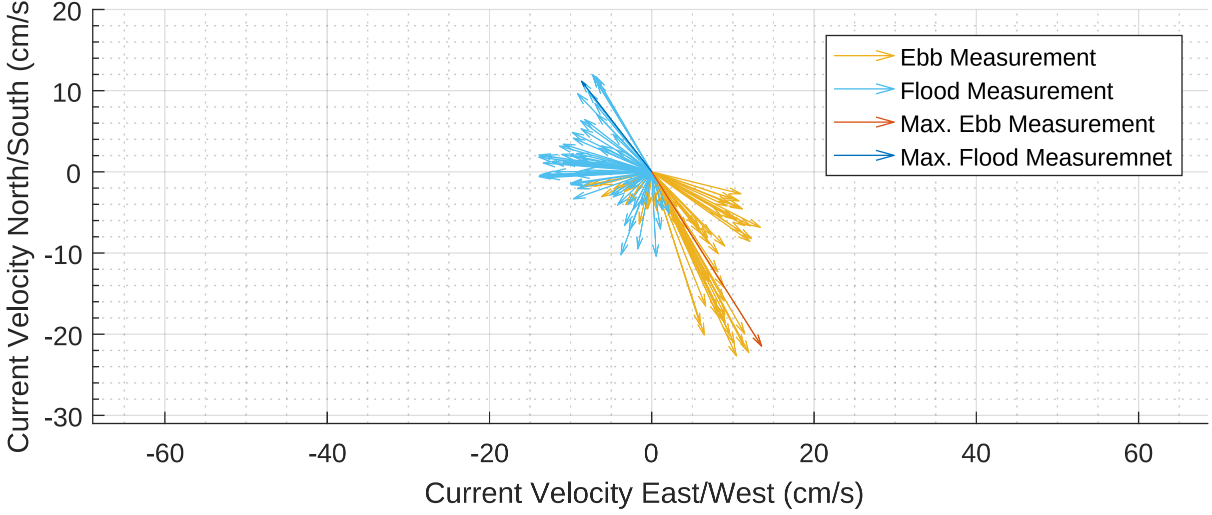
Current Measurements from -1 to -5 meters below surface



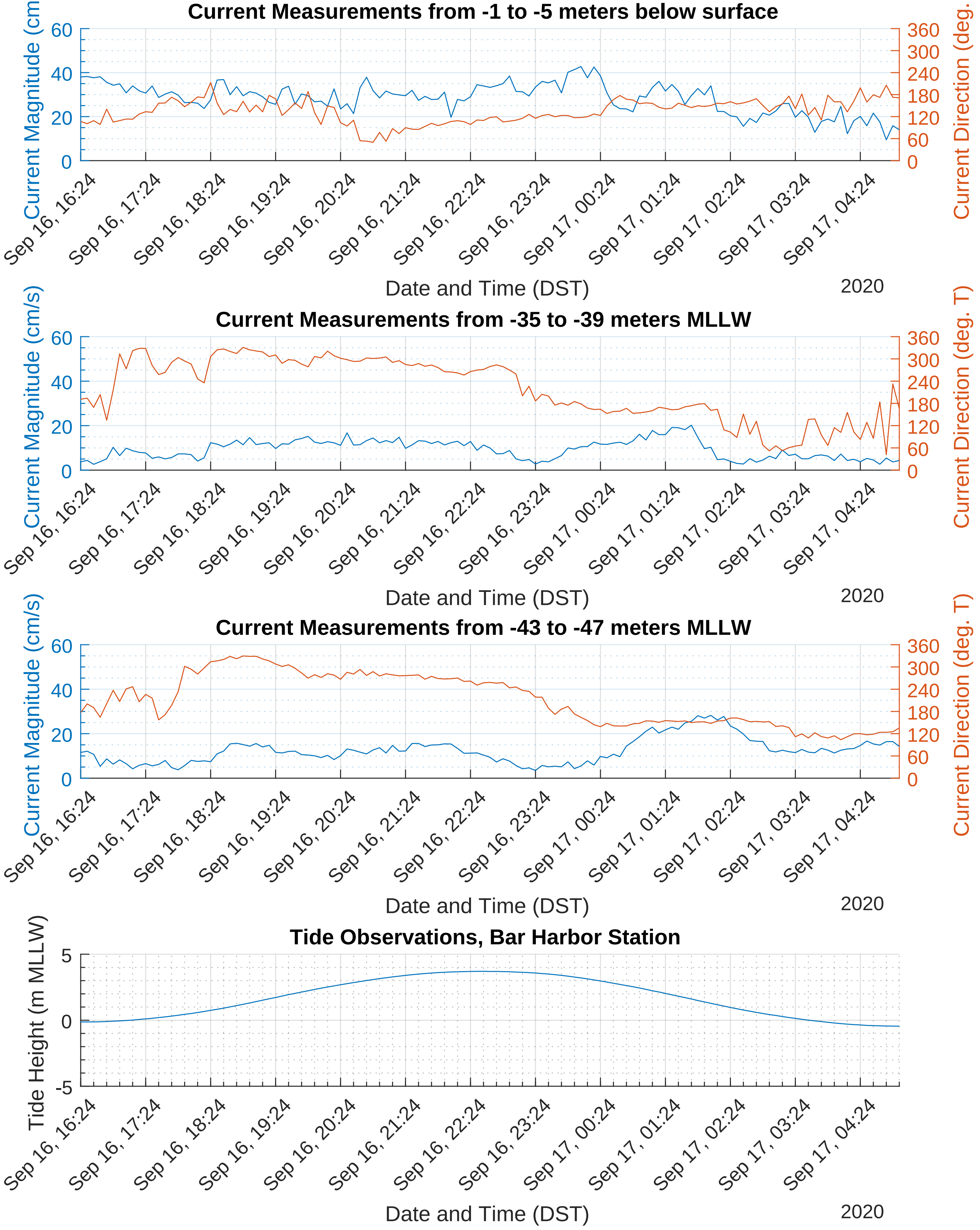
Current Measurements from -35 to -39 meters MLLW



Current Measurements from -43 to -47 meters MLLW



NPA Attachment 2: Figure 3



ATTACHMENT NPA-3
FEEDING CONDITIONS MEMO

OVERVIEW

This memo discusses feeding conditions for American Aquafarms' proposed Atlantic salmon production at site FB01 (Long Porcupine Island). American Aquafarms plans to use semi-closed containment system technology. This production method differs from the traditional way of farming finfish in open net pens in that the fish is completely isolated from the upper water stratum. This is achieved by an enclosing polymer sack that surrounds the entire rearing net that holds the fish. Water is supplied by pumps collecting water from deeper water stratum through elongated pipes extending down to desired depths of 25-50 meters. The water depth from which the water is collected can be chosen, as the pipe length can be altered. As the water moves from the bay into the pipes, it passes through a coarse filter, preventing fish and other marine organisms from entering the pipe.

Feed is distributed from a centralized processing barge to the 15 cages in the system. As the fish consume feed and excrete fecal matter, this waste is collected in a waste collection funnel in the bottom of the pen. The flow velocity in the lower part of the pen allows for separation of solids from the water flow, which later exits the cage. As the waste is collected in a sedimentation funnel, it is removed and transported to the process barge for treatment. Uneaten feed pellets will be collected in the sedimentation funnel rather than being discharged into surrounding water.

FEED SCHEDULE

Fish will be fed 2-3 times per hour per pen on average. The daily dose will depend on stocking density and environmental conditions such as temperature and light. Daily feed use per site will vary depending on the size of the fish and time of year. The feed schedule is based on extensive research and experience with farming of Atlantic salmon in Norway to achieve optimal fish health and growth and to limit waste. By implementing a steady rate of feed delivery in a closed pen system the fish will utilize up to 97% of the feed. This reduces excess feeding to approximately 3%. Comparative numbers of excess feed in traditional net pens are15-25% The excess feed is collected and removed from the pens by being pumped to the feed/process barge together with the waste/by-product.

FEED TECHNIQUES AND TYPE OF FEED USED

The feed to be used in the project will be based on standard commercial feed formula. American Aquafarms are currently in discussions with potential feed suppliers. Standard commercial salmon feed diets are composed of nitrogen (5.5 – 6.3 %), phosphorous (0.6 – 1 %) and organic carbon (51 – 65 %)[1]. These nutrients are derived from fishmeal, fish oil, vegetable proteins and oils, and surplus protein resources from the human food consumption industry. The feed ingredients are made from a range of natural raw materials sourced from agricultural crops, fisheries and byproducts from the human-food consumption industry. Ingredients from by-products from human-food processing chain (20%) would otherwise have been wasted if not used in the fish feed industry. This represents an efficient use of natural resources and supports the development of a circular economy. The feed will contain no antibiotics, no palm oil, no ingredients made from genetically modified organisms, and no LAPs. Our feed suppliers

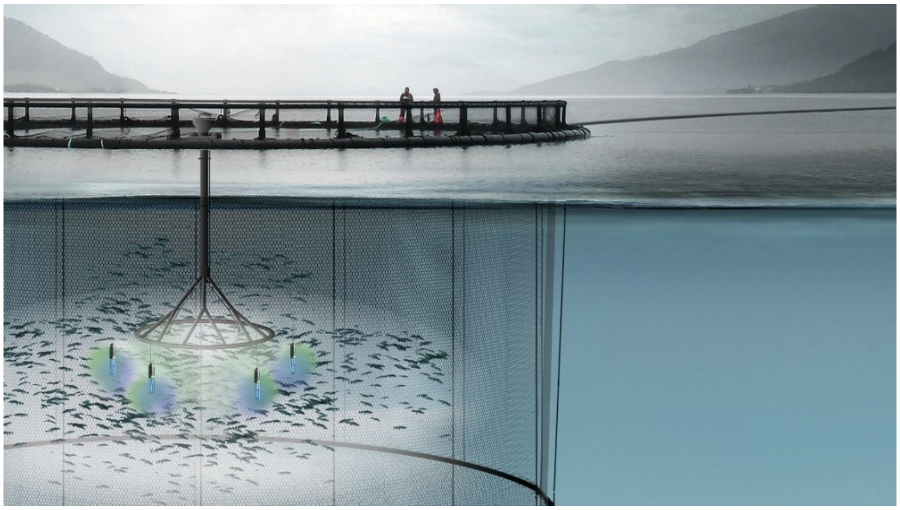
---

[1] IMTA – Integrated Aquaculture Report (Norwegian Directorate of Fisheries)

meet high standards on social and environmental responsibility and are members of sustainable sourcing projects such as the Roundtable on Responsible Soy (RTRS) and the IFFO-RS MarinTrust Standard.

The feed is distributed from the processing barge through pipes to each individual pen. The feed is waterborne, which means that feed is mixed with water and pumped to the pen where it is distributed to the fish. The process of feeding is automated, and closely monitored from the control center as well as on-site by the farmers.

Waterborne feeding is considerably gentler on both the pellets and the feeding pipe, and noise and micro-plastic discharge are reduced to an absolute minimum. As pellet density is not an issue for waterborne feeding, the feeding capacity is more than doubled.



As uneaten feed pellets and feces are pumped up from the pen, waste can be inspected through a looking glass before it is transported further through pipes to the central processing barge for treatment.

FEED MONITORING SCHEDULE

The feed dose will be regulated according to feeding behavior of the fish (on camera monitor) and level of uneaten feed pellets measured in the waste collection pipe. The feeding operation will be monitored both from the farm site by staff through hand feeding and from the remote operated control room (24/7) at the land facility. Feeding is one of the most important aspects of achieving optimal fish health and growth for the fish. Multiple cameras and sensors will continuously monitor the fish. The monitoring and logging of data will be controlled by a state-of-the-art control room located at the land base at Prospect Harbor. The control room will monitor, catalogue, and optimize all relevant variables including feed, temperature, oxygen, PH, fish health, mortality and stocking densities as well as net and polymer sack integrity. Control room operators will work in collaboration with on-site crew to optimize operations with regards to rearing the fish. Communication between site and on-shore control room is planned via 4g network and/or dedicated radio link. Control room is also responsible for continuous monitoring and contingency or emergency response.

RAMP UP DESCRIPTION

Production ramp up will commence at FB01 (off Long Porcupine Island) and will begin 9-12 months after receiving the leases and will follow a gradual ramp up to 8 pens during its first 32 weeks, before approaching full operational scale with deployment of all 15 pens. Harvesting is expected to commence approximately 17 months after initial deployment of fish in the sea.

Discharge will be proportional to production intensity. A production ramp up plan is described in the table below for the site.

The ramp-up plan describes the production from when the first pen is ready to use (hereafter referenced to as week/month 1).

FB01 (Long Porcupine Island) – The table below presents the monthly feed use at the site over the first year of start-up and during a month of maximum production. Feed schedule will vary depending on seasonal factors, and age and size of the fish at specific temperatures and light conditions. Table is based on projections with a January start.

Month	**1**	**2**	**3**	**4**	**5**	**6**	
Pounds	28,220	42,387	94,151	168,672	323,095	527,533	
Month	**7**	**8**	**9**	**10**	**11**	**12**	**Max**
Pounds	843,930	887,109	1,016,445	1,260,445	1,384,336	1,439,335	3,126,908

The maximum feeding number is expected to have a duration of up to 1.5 months each year, while total average monthly feed use is estimated to be 2,480,175 pounds divided among 15 pens, once stage 3 is finalized and the desired production level is established.

The amount of feed is the primary factor determining the volume of discharge. Discharge values are calculated based on our projected feeding requirements for achieving 15,000 metric tons of annual production.

Net Pen Aquaculture Supplemental Application Form Attachment NPA-4

Chemical use on site

This document is a summary of the therapeutants and compounds used for American Aquafarm's projected Atlantic salmon rearing operation in Frenchman Bay, Maine.

Disinfectants/sanitizers

Compounds intended to be used

The compound listed below will be discharged in the secondary outlet together with the waste processing reject water.

Virkon® Aquatic. A powerful cleaning and disinfecting solution with efficacy against fish viruses, bacteria, fungi, and molds. Virkon® Aquatic is EPA registered for the disinfection of environmental surfaces associated with aquaculture**.** Active ingredient: Potassium monopersulfate (21.4%). Used in accordance with label as a general cleaner and in footbaths. Working solution strengths normally range from 0.5% - 2.0%. Approx. annual use: 66-88 lbs/year (30-40 kg/year).

Therapeutants

Compounds intended to be used:

The compound listed below will be discharged in the secondary outlet together with the waste processing reject water.

Finquel® or Tricane-S. (Tricaine methanesulfonate). Used periodically during fish health inspection. Typical dose rates of 100-330 mg/L. Approximate annual use: 22 lbs/year (10 kg/year).

Compounds Rarely Used Only in Emergency Situations:

American Aquafarms has to this date no intended use of any therapeutic compounds. However, if any fish is to be treated by any of the compounds listed below it will be performed in an isolated compartment at the well boat. Dewatering units for ingoing and outgoing water, entering, and exiting the well boat, will ensure that the treatment water is kept contained on the well boat. This method of treatment allows American Aquafarms to avoid discharge of therapeutants into bay water if any compounds was to be used for contingency response. All products listed will be used

according to label use or a licensed veterinarian's prescription. There will be no prophylactic use of antibiotics.

Parasite-S, Formalin-F, and Formacide-B. (Formalin). Active ingredient 37% formaldehyde.Used according to the label if needed to alleviate fish health issues due to parasitic copepods, external protozoa and monogenetic trematodes. Typical dose rates from 25ppm to 1,000 ppm. Expected annual use: 0 gallons/year (0 lbs/year).

Halamid® Aqua. (Chloramine-T). Active ingredients N-chloro, p-toluenesulfonamide and sodiumsalt trihydrate. Used periodically according to the label if needed to alleviate fish health issues due to bacterial gill disease. Typical dose range 12-20 ppm. Expected annual use: 0 lbs/year (0 kg/year).

Praziquantel. Considered as 100% active. Can be used if fish are suffering from trematode/cestode (i.e. flatworm) infections. Typical dose ranges from 5-200 ppm depending on length of standing bath treatment. Used as needed/intermittent or emergency use only according to label use or as prescribed by a licensed veterinarian. Expected annual use: 0lbs/year (0 kg/year).

Terramycin® 200. (oxytetracycline dehydrate, 44% active): Can be used as an in-feed treatment(maximum of 0.08 g active oxytetracycline/kg fish/day) if fish are suffering from certain bacterial infections. Used as needed/intermittent or emergency use only, according to label use or as prescribed by a licensed veterinarian. Expected annual use: 0 lbs/year (0 kg/year).

Aquaflor®. (florfenicol; 50% active). Can be used as an in-feed treatment (maximum of 15mg/kg fish/day) if fish are suffering from certain bacterial infections. Used as needed/intermittent or emergency use only, according to label use or as prescribed by a licensed veterinarian. Expected annual use: 0 lbs/year (0 kg/year).

Romet® 30/Romet® TC. (sulfadimethoxine/ormetoprim, 30% active or 20% active, respectively). Can be used as an in-feed treatment (maximum of 50 mg/kg fish/day) if fish are suffering from certain bacterial infections. Used as needed/intermittent or emergency use only, according to label use or as prescribed by a licensed veterinarian. Expected annual use: 0 lbs/year (0 kg/year).

Chemical use for ensilage

Chemistry used for ensilage will be used in an enclosed system and will not have contact with bay water at any time. The amount of ensilage chemistry used on site will vary depending on mortality. Thus, the chemical usage is based on a best effort estimate, but could however change after the operation has started.

SoftAcid Aqua MAB+. Main active ingredient formic acid. Product composition: formic acid (85 %), lignosulfonic acid (19.75 %), propyl gallate and citric acid (0.35 %) and BHA (0.16 %). To be used for preserving residual raw material in the fresh fish remains. Used in accordance with label or as described by supplier. Expected annual use 5,000 – 10,000 lbs/year (2,300 – 4,550 kg/year)

Chemicals used for flocculation of waste

A flocculant will be used for coagulating/thickening the solid waste as it is introduced to the waste processing system. As the flocculant is added it binds to the solids and increases the adherence between the particles. Thus, as the flocculant is added it is bound to the waste and the water is being separated from the waste, the flocculant will not be emitted but collected with the dewatered waste. Since the flocculant will be bound to the waste, only a minor portion will escape into the discharge water. The flocculant is composed of the following compounds listed below.

Acetic acid (60 %). Used for dissolving the calcium carbonate in fresh water. Expected annual use 17,650 – 22,050 lbs/year (8-10 MT/year.)

Calcium carbonate. (85-100%). Used for pH correction of waste flow prior to flocculation with sodium alginate. Expected annual use 3,750 – 4,630 lbs/year (1,700 – 2,100 kg/year)

Sodium Alginate. Biopolymer to be used as a waste thickener. Consists of 1,4-β-d-mannuronic (M) and α-l-guluronic (G) acids. Used in accordance with label or as described by supplier. Expected annual use 1874 - 2315 lbs/year (850 – 1,050 kg/year).

WATER QUALITY MONITORING REPORT
FRENCHMAN BAY SITE NO. 1
HANCOCK COUNTY, MAINE

Prepared on behalf of:

American Aquafarms
68 Commercial Street
Portland, Maine

To fulfill the requirements of:

Maine Department of Marine Resources
Division of Sea Run Fisheries and Habitat
21 State House Station
Augusta, Maine

And:

Maine Department of Environmental Protection
Division of Water Quality Management
17 State House Station
Augusta, Maine

Prepared by:

Ransom Consulting, LLC
400 Commercial Street, Suite 404
Portland, Maine 04101
(207) 772-2891

Project 201.05012
March 3, 2021

TABLE OF CONTENTS

FIGURES

TABLES

APPENDICES

1.0 INTRODUCTION

This report summarizes the water quality monitoring data collected at the proposed aquafarm lease site identified as Frenchman Bay Site No. 1, referred to as FB01, located in Frenchman Bay, Hancock County, Maine. The water quality monitoring data described herein was collected by Ransom Consulting, LLC (Ransom) on behalf of American Aquafarms in support of permit applications as described below. Water quality monitoring and sample collection and analysis activities were performed in accordance with Ransom's *"Sampling and Analysis Plan (SAP), Water Quality Monitoring – Frenchman Bay and Blue Hill Bay, Maine"*, dated November 12, 2020 and reviewed by Maine Department of Environmental Protection (MEDEP) on August 17, 2020 (see Appendix A).

1.1 Purpose

The water quality monitoring program discussed herein was developed to assess the existing, or "baseline", water quality conditions in support of permit applications for a proposed closed-pen aquaculture facility. The purpose of this Water Quality Monitoring Report is to provide the information required by the Maine Department of Marine Resources (MEDMR) Application for Standard Aquaculture Lease Net Pen Aquaculture Discharge, which includes data required by the MEDEP as part of a Maine Pollutant Discharge Elimination System (MEPDES) Individual Discharge permit application as well as the United States Army Corps of Engineers standard permit requirements. The information and data provided in this report were collected as part of a larger monitoring program involving several potential lease sites. However, the information and data presented in the report is specific to the lease site identified as FB01 except where data from other sites are relevant for quality assurance/quality control (QA/QC) assessment.

1.2 Scope of Work

The water quality monitoring activities included *in-situ* vertical water column characterization, water transparency observations, and collection and laboratory analysis of discrete-depth water samples in the area of the proposed aquafarm lease. Monthly water quality monitoring activities were performed from July through October 2020 to provide a representative characterization of ambient water quality conditions. Specific methodologies utilized for the water quality monitoring activities are described below.

2.0 METHODOLOGY

The following methodologies and Standard Operating Procedures (SOPs) were utilized to collect, analyze, and document the water quality monitoring data. The water quality monitoring methodologies described below were developed in consultation with MEDEP staff and proposed in Ransom's SAP dated November 12, 2020. Technical review of the SAP was provided by Ms. Angela Brewer, Biologist with the MEDEP.

2.1 Site Location

Potential lease site FB01 was chosen through an iterative process that accounted for water depth, relative protection from swell and wind during major storms, and expected currents. The proposed lease area was updated several times during the monitoring work based on preliminary data collection and feedback from stakeholder groups; however, the general study area is not significantly different from the original study area. Once the proposed lease site was chosen, the site coordinates were programed into the onboard GPS unit to facilitate navigation to the site for each water quality monitoring event.

The FB01 lease area encompasses an area of 60.37 acres and is shown in Figure 1. The proposed lease corners are summarized in the table below.

Corner Location	Latitude	Longitude	Easting (Maine State Plane East, feet)	Northing (Maine State Plane East, feet)
West	44.420750	-68.164800	1,071,821.1	275,048.0
North	44.424617	-68.159183	1,073,282.7	276,463.7
East	44.421767	-68.155350	1,074,288.4	275,429.0
Mid-South	44.419650	-68.158433	1,073,486.2	274,653.9
Mid-North	44.420283	-68.159300	1,073,258.7	274,883.7
South	44.418550	-68.161817	1,072,603.7	274,249.3

2.2 Monitoring Schedule, Frequency, and Conditions

Ransom completed monthly water quality monitoring events at site FB01 from July to October 2020. As required by MEDMR's application requirements, water column profiles were collected at multiple points in the tidal cycle and on at least one occasion within two hours of sunrise. Discrete-depth water samples were also collected for laboratory analysis in a variety of sea state conditions. Details of the individual monitoring events including time of data/sample collection, weather conditions, sunrise, and tide conditions, are summarized in Table 1.

2.3 *In-situ* Vertical Water Column Characterization,

In-situ water column characterization was conducted in accordance with the MEDEP's SOP for Water Quality Sonde Profile Data Collection and Processing, Rev. 01, dated April 7, 2017. During each monthly monitoring event, Ransom conducted *in-situ* vertical water column characterization at three different profile locations within or adjacent to the proposed lease area using a YSI EXO1 water quality multimeter. Water quality parameters were recorded at 3-second intervals from the water surface to a maximum depth of approximately 200 feet or the bottom of the water column, whichever was shallower. Water column profile data were recorded for the following eight parameters:

1. Depth

2. Temperature

3. Specific Conductivity / Total Dissolved Solids (TDS) / Salinity

4. Dissolved Oxygen (DO)[1], both in percent local saturation and milligrams per liter (mg/l)

5. pH

6. Turbidity

7. Density

8. Oxidation Reduction Potential (ORP)

2.4 Water Transparency Observations

Water transparency was measured using a Secchi disk in accordance with MEDEP's SOP for Transparency Data Collection and Processing, Rev. 00, dated June 2016. During each monitoring event, a Secchi disk was lowered into the water column until the disk was no longer visible. The depth at which the disk disappeared and reappeared was recorded. This procedure was repeated three times during each sampling event, and an average of the depth of disappearance and reappearance is shown on Table 1.

2.5 Discrete-depth Water Sampling and Laboratory Analysis

During each monthly event, discrete-depth water quality samples were collected from the proposed lease site at three depths identified as "Near Surface", "Mid Column", and "Near Bottom". These sampling depths are defined as follows:

- **Near Surface** – 0.2 meters below the water surface.
- **Mid Column** – approximately ½ the total depth where the vessel was positioned, at the time of sampling.
- **Near Bottom** – 2 meters above the bottom, or sampler line fully paid out (up to approximately 200 ft).

Discrete-depth water samples were collected using a horizontal Van Dorn-style sampler in accordance with MEDEP SOP for Water Grab Sampling Collection and Processing, Rev. 00, dated June 2016. Following collection, the samples were transferred into laboratory prepared containers and transported under chain-of-custody protocol to a Maine accredited laboratory for analysis. Water quality samples were submitted for laboratory analysis of the following seven parameters:

1. Nitrate + nitrite

2. Ammonia

---

[1] Dissolved oxygen was measured using an optical sensor that was calibrated prior to each sampling event. Sensor calibration was compared with results from Winkler titration and found to be within 5% (92% saturation using optical sensor, 88% saturation via Winkler).

3. Total kjeldahl nitrogen
4. Total nitrogen mixed forms
5. Orthophosphate
6. Total phosphorous
7. Total suspended solids

Samples submitted for analysis of orthophosphate, ammonia, and nitrate/nitrite were filtered in the field using a 0.45-micron filter. Duplicate samples were collected for quality assurance purposes from the FB01 lease area and other potential lease areas as part of the larger water quality monitoring program.

Discrete-depth water samples were initially submitted to Alpha Analytical Laboratory in Westboro, Massachusetts (Alpha) for the analyses listed above. Alpha had difficulty achieving the necessary reporting limits for ammonia, nitrate/nitrite, and total kjeldahl nitrogen. Following discussion with MEDEP Biologist Angela Brewer, samples collected during the August sampling event were split with ALS Environmental laboratories of Rochester, New York for analysis of ammonia, nitrate/nitrite and total kjeldahl nitrogen. Based on a comparison of the split sample results received, samples collected during the September and October sampling events were submitted to ALS Environmental for analysis of ammonia, nitrate/nitrite, total kjeldahl nitrogen, and total suspended solids. Samples collected for total phosphorous and orthophosphate during the September and October sampling events continued to be analyzed by Alpha.

3.0 WATER QUALITY MONITORING RESULTS

Results of the water quality monitoring activities are summarized in the following sections. Tabulated data from the *in-situ* vertical water column characterization and laboratory analytical results from the discrete-depth water samples have been provided to the MEDEP in Electronic Data Deliverable (EDD) format. Graphs showing *in-situ* water characteristics by depth are provided in Appendix B. Discrete-depth water sample analytical results are summarized in Table 2; corresponding laboratory analytical reports are included in Appendix C. Sample locations are shown on Figure 2.

3.1 *In-situ* Vertical Water Column Characteristics

Graphs showing water quality parameters versus depth for each profile location (profiles 1 through 3) and for each monthly monitoring event are included in Appendix B. All parameters were recorded directly or calculated within the YSI sonde except for density. Density was calculated as the sigma-t density using the SIGMA function included in the Ocean toolbox maintained by the Scripps Institution of Oceanography. This function calculates the density of seawater at a given temperature and salinity at atmospheric pressure and subtracts 1000 kg/m^3 from the result. Seawater density in this report is reported as total density by adding 1000 kg/m^3 back to the result. Dissolved oxygen is reported in both milligrams per liter (mg/l) and percent saturation.

The data collected during the July monitoring event suggest relatively strong stratification in the water column with a thermocline, halocline, and pycnocline apparent at a depth of approximately 13 to 14 meters below the water surface. Below a depth of 13 to 14 meters, temperatures stabilize to approximately 9.6-10.4°C and salinity stabilizes at approximately 31.1 PSU. Density, which is largely a function of salinity and temperature, showed a similar pattern with depth, stabilizing at approximately 1,023.8-1,024.3 kg/m^3 below a depth approximately 15 meters below the water surface. The dissolved oxygen data from the July event also show a gradual concentration gradient in the top 15 meters, stabilizing to a constant concentration of 8.35 mg/l or 93% saturation below approximately 15 meters.

In the August data, stratification is still evident, however, the boundary between the mixed layer and the layer below is less abrupt, indicating more mixing between layers than in July. The depth of the thermocline and chemocline is similar to what was observed in July, approximate 13 to 14 meters; however, temperature and dissolved oxygen do not stabilize as quickly below this depth in August. An anomaly in the halocline is observed in profile 1 at approximately 13 to 14 meters, however, profiles 2 and 3 do not show a similar anomaly. Profile 1 was the furthest west of the three profiles collected during August, indicating that at the time sampling occurred more mixing was taking place in the eastern portion of the study area, closer to one of the major channels between inner and outer Frenchman Bay and more exposed to swell or other weather events from the south. Dissolved oxygen is well mixed and supersaturated to a depth of approximately 6 meters, at which point dissolved oxygen declines fairly rapidly to a depth of approximately 20 meters before beginning to stabilize. The minimum dissolved oxygen concentration and saturation values, 7.25 mg/l or 80% saturation, were observed at the maximum depth of approximately 59 meters.

Little to no stratification is apparent in the September and October vertical profiling data, although dissolved oxygen data do display some stratification. Temperatures remain essentially constant at about 12.5°C in September and 12.0°C in October from the water surface to the maximum depth. Similarly, salinity values are stable throughout the water column, measuring about 32.8 PSU in September and 30.5 PSU in October. Salinity in October was lower than any other month monitored, which may be due to significant precipitation in the week prior to sampling (2.46 inches). Dissolved oxygen values show a slight gradient in the top 20 meters before stabilizing at depths below 20 meters at approximately 7.65 mg/l or 88% saturation in September and 8.25 mg/l or 91% saturation in October.

Overall, stratification of the water column was strongest during the July sampling event, while dissolved oxygen concentration and saturation was lower during the August sampling event. After August, dissolved oxygen increased in both September and October. The dissolved oxygen content meets the MEDEP standard for Class SB waters (i.e., not less than 85% of saturation) across the four months of monitoring, except for data collected from depths below approximately 30 meters during the August monitoring event.

3.2 Water Transparency

Average water transparency observations for each monitoring event are included in Table 1. Secchi depths ranged from approximately 3.7 to 5.7 meters below water surface, becoming progressively less clear from the July to October monitoring events.

3.3 Discrete-depth Sample Analytical Results

Analytical results for each discrete depth interval (near surface, mid-column, and near bottom) for each monitoring event are shown on Table 2. Table 2 also includes mean value for each analytical parameter at each depth interval. Corresponding laboratory analytical reports are included in Appendix C.

During the July and August sampling events, Alpha was not able to achieve the necessary reporting limits for ammonia, nitrate/nitrite, and/or total kjeldahl nitrogen, which resulted in the reporting of estimated (or "J-flagged") concentrations. In consideration of these limitations, these analytes were analyzed by ALS in August, September, and October. ALS was able to achieve the necessary reporting limits. Therefore, data provided by Alpha was eliminated from the calculation of the average value for ammonia, nitrate/nitrite, and total kjeldahl nitrogen shown on Table 2. In the event that an analyte was not detected above the method detection limit (U<[value]), a value of half the method detection limit was used to calculate the average concentration for that analyte.

Total nitrogen concentrations ranged from <0.1 mg/l (below the detection limit) to 0.26 mg/l, with an overall average of approximately 0.19 mg/l. In August, the total nitrogen concentrations were observed to be lowest in the surface sample, highest in the mid-column, and lower in the bottom sample. In September and October, the total nitrogen concentrations appear to be relatively consistent throughout the water column. Of the three months for which total nitrogen values were calculated, September yielded the highest concentration of total nitrogen throughout the water column.

Total phosphorous concentrations ranged from 0.017 mg/l to 0.031 mg/l, with an overall average of approximately 0.025 mg/l. In July, concentrations increased with depth in the water column. In August and September, the concentration of phosphorus was lowest near the surface, highest mid-column, and slightly lower near the bottom of the water column. In October, phosphorus concentrations were fairly stable throughout the water column although the highest value was measured at the bottom.

Data for total suspended solids were reviewed for the September and October monitoring events. (Note that data provided by Alpha for the July and August monitoring events were rejected as discussed in Section 4.0 below.) In September, total suspended solids were higher near the surface and the bottom of the water column and lower in the middle of the water column. In October, total suspended solids were lower near the surface and the bottom of the water column and higher in the middle of the water column.

3.4 Possible Effects of Precipitation

Frenchman Bay, where the proposed lease is located, is a relatively saline estuary with semi-restricted access between the Bay and the Gulf of Maine. Because of this, there is potential for precipitation events to influence the chemical and/or physical characteristics of the Bay. In order to assess this potential, precipitation data from the Bar Harbor/Trenton airport (Station WBAN:14616) for the 7 days prior to each monitoring event were reviewed (Table 3). Precipitation amounts varied from 0.01 inches (September) to 2.46 inches (October) in the week prior to sampling.

Precipitation does not appear to have a significant relationship between total suspended solids data or Secchi depth measurements, although this could either be due to limited data or because precipitation and total suspended solids are in fact not correlated. Precipitation prior to sampling does appear to correlate with salinity, however, which is generally lower after higher precipitation. Similarly, this assessment was conducted with a relatively limited dataset and may not fully represent the true relationship between precipitation and water characteristics.

4.0 DATA QUALITY & USABILITY

The objective of the water quality monitoring program was to generate the quality and quantity of data necessary to document the ambient water quality conditions in the area of the proposed aquafarm lease site. *In-situ* readings and discrete-depth samples obtained during the monitoring program were collected in accordance with MEDEP SOPs.

No anomalies were noted in the *in-situ* water quality data collected. Except for salinity values for profile 1 during the August sampling event, similar values and trends were observed in each of the three vertical water column profiles conducted during each of the monthly sampling events. As discussed in Section 3.1, the salinity anomaly observed in profile 1 in August is believed to represent a real anomaly and is not considered a data quality or usability issue. The measured pH values in August were notably higher than other sampling events, at both FB01 and other sites monitored in the vicinity of FB01 earlier on the same day. Subsequent assessment of the pH probe using standard solutions indicated that the electrode on the pH probe had begun to fail shortly before the August 2020 sampling event at FB01. Due to this, pH data collected in August at FB01 are not considered reliable data. Except for the instance noted above, the consistency in water parameter values and trends observed during the replicate vertical profiling activities suggests the vertical profiling data are accurate and usable for planning and decision-making purposes.

Ransom has reviewed the laboratory data packages provided for the discrete-depth water samples collected during the monitoring program. Certain data contained within the Alpha laboratory packages for the July and August sampling events were reported as “estimated” concentrations due to detections below the laboratory reporting limit. Due to the inability of Alpha to meet the required reporting limits, data reported by Alpha was excluded from the calculation of average concentrations for ammonia, nitrate/nitrite, and total kjeldahl nitrogen.

In reviewing the data reported for total suspended solids, it is apparent that Alpha consistently reported significantly higher concentrations of total suspended solids than ALS. It is also noted that Alpha’s reporting limit for total suspended solids was 5 times the reporting limit of ALS. No data quality issues were reported by either laboratory for analysis of total suspended solids. Following a review of the data and based on experience with other marine ambient water quality monitoring efforts, it is Ransom’s opinion that the total suspended solids results reported by ALS are accurate, and the total suspended solids results reported by Alpha may be suspect. Because of this, Ransom rejected the Alpha data, and therefore the ALS results for total suspended solids were used to generate the average concentrations shown in Table 2.

As part of the larger water quality monitoring program and in accordance with Ransom’s SAP dated November 12, 2020, a total of six duplicate samples were collected. In comparing duplicate sample results, it should be noted that in many cases, the reported concentrations are only slightly above the laboratory reporting limit, which can be reasonably expected to result in a fair amount of variability in results. In certain instances, minor absolute differences in the results of the parent sample versus the duplicate sample result in elevated relative percent differences when the magnitude of difference between the sample results is in fact quite small. Because of this, relative percent difference values for results near the detection limit are not considered to be a reliable estimate of the true reproducibility of the sample result. Therefore, relative percent differences[2] were calculated for samples where both sample and duplicate values were greater than five times the laboratory reporting limit. Based on Ransom’s review of the laboratory data reports data reproducibility between duplicate and parent samples is not considered to represent a data quality or usability issue. The six duplicate samples collected as part of the larger water

---

[2] Relative percent difference (RPD) is calculated as follows: $RPD = \frac{(Sample\ Result - Duplicate\ Result)}{Mean\ of\ the\ Two\ Results}\ x\ 100$

quality monitoring project, including the duplicate sample collected at potential lease area FB01, show relatively good correlation with the corresponding parent samples. Duplicate sample results are shown in conjunction with their corresponding parent sample results on Table 4.

Table 1: Water Quality Monitoring Event Conditions
Water Quality Monitoring Report
Frenchman Bay, Site No. FB01
Hancock County, Maine

Date	Water Column Profile Time	Discrete Depth Sample Time	Weather Conditions Arrival	Weather Conditions Departure	Water Transparency (Secchi Depth)	Low Tide Time	High Tide Time	Sunrise Time
July 22, 2020	P1: 07:50 to 08:03	Near Surface (FB01-G1-1): 10:40	Cloud Cover = 75% Wind Direction = N Wind Speed = 2 kts Beaufort Scale = F0	Cloud Cover = 75%, Wind Direction = N, Wind Speed = 3kts, Beaufort Scale = F1	5.7 m	6:47	12:46	5:08
	P2: 08:30 to 08:39	Mid Column (FB01-G1-100): 10:15						
	P3:08:52 to 09:03	Near Bottom (FB01-G1-193): 09:45						
August 20, 2020	P1: 12:21 to 12:29	Near Surface (FB01-G1-SUR): 11:55	Cloud Cover = 1%, Wind Direction = WNW, Wind Speed = 15 kts, Beaufort Scale = F4	Cloud Cover = 2%, Wind Direction = WNW, Wind Speed = 2-4 kts, Beaufort Scale = F1/F2	4.1 m	6:22	12:20	5:41
	P2: 12:40 to 12:51	Mid Column (FB01-G1-MID): 11:35						
	P3: 13:00 to 13:30	Near Bottom (FB01-G1-BOT): 11:10						
September 24, 2020	P1: 07:55 to 08:05	Near Surface (FB01-SUR): 09:30	Cloud Cover = 0%, Wind Direction = NW, Wind Speed = 10 kts, Beaufort Scale = F3/F4	Cloud Cover = 10%, Wind Direction = NW, Wind Speed = 15 kts, Beaufort Scale = F4	4.0 m	11:20	5:02	6:21
	P2: 08:13 to 08:25	Mid Column (FB01-MID): 09:15						
	P3: 08:30 to 08:39	Near Bottom (FB01-BOT): 08:50						
October 21, 2020	P1: 12:24 to 12:32	Near Surface (FB01-SUR): 11:50	Cloud Cover = 100%, Wind Direction = ESE, Wind Speed = 5 kts, Beaufort Scale = F2	Cloud Cover = 100%, Wind Direction = ESE, Wind Speed = 3 kts, Beaufort Scale = F0	3.7 m	8:56	14:55	6:54
	P2: 12:37 to 12:44	Mid Column (FB01-MID): 11:35						
	P3: 12:49 to 12:57	Near Bottom (FB01-BOT): 11:30						

Secchi Depth is calculated as the average of the disappearance and reappearance depths for three sequential attempts.

Table 2: Discrete-depth Water Sample Analytical Results
Water Quality Monitoring Report
Frenchman Bay, Site No. FB01
Hancock County, Maine

Site	Sample Depth (m)	Laboratory	Average of Total Nitrogen (MG/L)	Average of Total Phosphorus as P (MG/L)	Average of Ammonia as N (MG/L)	Average of Nitrate + Nitrite as N (MG/L)	Average of Total Kjeldahl Nitrogen (MG/L)	Average of Orthophosphate as P (MG/L)	Average of Total Suspended Solids (MG/L)
FB01 - SUR	**Mean Value:**		**0.15**	**0.021**	**0.009**	**0.042**	**0.12**	**0.014**	**4.7**
July 22	0.2	Alpha	NC	0.020	J 0.060	U<0.10	0.384	0.007	14
Aug 20	0.2	Alpha	NC	0.017	U<0.075	J 0.033	J 0.172	0.006	28
		ALS	U<0.1	NA	0.011	U<0.050	U<0.10	NA	NA
Sept 24	0.2	Alpha	NC	0.018	NA	NA	NA	0.023	6.2
		ALS	0.24	NA	0.010	0.075	0.17	NA	5.8
Oct 21	0.2	Alpha	NC	0.028	NA	NA	NA	0.020	14
		ALS	0.17	NA	U<0.010	U<0.050	0.13	NA	3.6
FB01 - MID	**Mean Value:**		**0.22**	**0.027**	**0.015**	**0.075**	**0.15**	**0.021**	**4.8**
July 22	30.4	Alpha	NC	0.024	0.118	J 0.085	J 0.276	0.019	20
Aug 20	27.4	Alpha	NC	0.029	0.078	0.11	J 0.294	0.019	18
		ALS	0.24	NA	0.020	0.057	0.18	NA	NA
Sept 24	23.8	Alpha	NC	0.027	NA	NA	NA	0.026	U<5.0
		ALS	0.23	NA	0.019	0.103	0.13	NA	3.8
Oct 21	25.9	Alpha	NC	0.028	NA	NA	NA	0.021	5.0
		ALS	0.20	NA	U<0.010	0.064	0.14	NA	5.8
FB01 - BOT	**Mean Value:**		**0.20**	**0.028**	**0.022**	**0.080**	**0.11**	**0.022**	**4.9**
July 22	58.8	Alpha	NC	0.028	J .070	J .073	J 0.268	0.019	17
Aug 20	44.3	Alpha	NC	0.028	0.080	J 0.090	J 0.261	0.022	20
		ALS	0.17	NA	0.026	0.069	0.11	NA	NA
Sept 24	51.8	Alpha	NC	0.026	NA	NA	NA	0.025	5.3
		ALS	0.26	NA	0.036	0.093	0.17	NA	5.1
Oct 21	49.1	Alpha	NC	0.031	NA	NA	NA	0.022	15
		ALS	0.18	NA	U<0.010	0.077	U<0.10	NA	4.7

NOTES:
mg/l = milligrams per liter
NC = Not calculated due to "estimated" values for ammonia, nitrate/nitrite, and/or total kjeldahl nitrogen.
NA = Not analyzed.
U<(Value) = not detected above the given value.
J = Estimated concentration detected above the method detection limit but below the laboratory reporting limit.
FB01-DUP is a duplicate sample of FB01-SUR on October 21, 2020.
Estimated concentrations and data of questionable usability (shown with gray shading) not included in calculations of average concentrations.
In the case of non-detects, a value of half the method detection limit was used in the calculation of average concentration.

Table 3: Precipitation Data
Water Quality Monitoring Report
Frenchman Bay, Site No. FB01
Hancock County, Maine

Monitoring Date	Precipitation (inches)							
	7 Days Prior	6 Days Prior	5 Days Prior	4 Days Prior	3 Days Prior	2 Days Prior	1 Day Prior	Total Precipitation in Week Prior to Monitoring
July 22, 2020	0.00	0.00	0.32	0.00	0.00	0.00	0.00	0.32
August 20, 2020	0.00	0.00	0.00	0.00	0.07	0.78	0.00	0.85
September 24, 2020	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.01
October 21, 2020	1.19	0.00	0.11	1.15	0.00	0.00	0.01	2.46

Source: National Centers for Environmental Information, National Oceanic & Atmospheric Administration. Local Climatological Data (LCD) for Bar Harbor Airport, ME US. Station ID: WBAN:14616. Data available at https://www.ncdc.noaa.gov/cdo-web/datatools/lcd.

Table 4: Discrete-depth Duplicate Sample Comparison
Water Quality Monitoring Report
Frenchman Bay, Site No. FB01
Hancock County, Maine

Proposed Lease Site			**Blue Hill Bay 04**			**Blue Hill Bay 06**			**Blue Hill Bay 04**			**Frenchman Bay 01**			**Frenchman Bay 02**			**Prospect Harbor 01**		
Sample Date			**23-Jul-20**			**19-Aug-20**			**25-Sep-20**			**21-Oct-20**			**21-Oct-20**			**22-Oct-20**		
Sample I.D.	**Reporting Limit**	**Laboratory**	**BHB04-SUR**	**DUP**	**Relative % Difference**	**BHB06-SUR**	**BHB06-DUP**	**Relative % Difference**	**BHB04-SUR**	**DUP**	**Relative % Difference**	**FB01-SUR**	**FB01-DUP**	**Relative % Difference**	**FB02-SUR**	**FB02-DUP**	**Relative % Difference**	**PH01-SUR**	**PH01-DUP**	**Relative % Difference**
Total Nitrogen	0.1	ALS	NA	NA	NC	0.14	0.15	NC	0.18	U<0.1	NC	0.17	0.16	NC	0.67	0.14	NC	U<0.1	U<0.1	NC
Total Phosphorus	0.01	Alpha	0.019	0.018	NC	J 0.009	0.015	NC	0.021	0.022	NC	0.028	0.024	NC	0.021	0.026	NC	0.016	0.019	NC
Amonnia as N	0.075	Alpha	J 0.049	J 0.031	NC	J 0.051	J 0.027	NC	NA	NA	NC	NA	NA	NC	NA	NA	NC	NA	NA	NC
	0.01	ALS	NA	NA	NC	0.012	0.016	NC	U<0.010	U<0.010	NC	U<0.010	U<0.010	NC	U<0.010	0.015	NC	0.015	U<0.010	NC
Nitrate/Nitrite as N	0.1	Alpha	J 0.027	J 0.024	NC	U<0.10	U<0.10	NC	NA	NA	NC	NA	NA	NC	NA	NA	NC	NA	NA	NC
	0.05	ALS	NA	NA	NC	U <0.050	U <0.050	NC	U<0.050	U<0.050	NC	U<0.050	U<0.050	NC	U<0.050	U<0.050	NC	U<0.050	U<0.050	NC
Total Kjeldhal Nitrogen	0.3	Alpha	J 0.144	J 0.215	NC	J 0.275	J 0.181	NC	NA	NA	NC	NA	NA	NC	NA	NA	NC	NA	NA	NC
	0.1	ALS	NA	NA	NC	0.15	0.16	NC	0.15	0.10	NC	0.13	0.13	NC	0.66	0.13	NC	0.10	U<0.10	NC
Orthophosphate	0.005	Alpha	0.005	0.005	NC	0.008	0.006	NC	0.019	0.019	NC	0.020	0.018	NC	0.013	0.013	NC	0.016	0.017	NC
Total Suspended Solids	5	Alpha	27	22	NC	51	35	**37%**	47	53	NC	14	9.5	NC	9.3	8.3	NC	13	10	NC
	1	ALS	NA	NA	NC	NA	NA	NC	5.4	2.4	NC	3.6	4.2	NC	3.6	2.9	NC	4.1	3.4	NC

Notes:
Results in milligrams per liter (mg/l)
NC = Not Calculated, due to (1) either sample or duplicate value was estimated ("J flagged"), (2) either sample or duplicate value was not detected above the laboratory reporting limit, or (3) both sample and duplicate values were less than five times the laboratory reporting limit.
NA = Not Analyzed
Relative % Difference (RPD) is calculated as follows: RPD = ((Sample Result - Duplicate Result) / Mean of the Two Results) x 100
Relative % Difference values greater than 20% are shown in bold

RANSOM
Consulting
Engineers
and Scientists

Legend & Notes

☐ Lease Boundary
☐ Mooring Grid Boundary
☐ Pen Outline
☐ Barge Outline

General Notes:

1. Basemap from USGS Seamless Topo Map Server.
2. Some features shown are approximate in location and scale.
3. This map was prepared for American Aquafarms. All other uses are not permitted unless written permission is obtained from Ransom Consulting, LLC.

Scale & Orientation

0 1,500 3,000
1 inch = 3,000 feet

Prepared For

American Aquafarms
68 Commercial Street
Portland, Maine

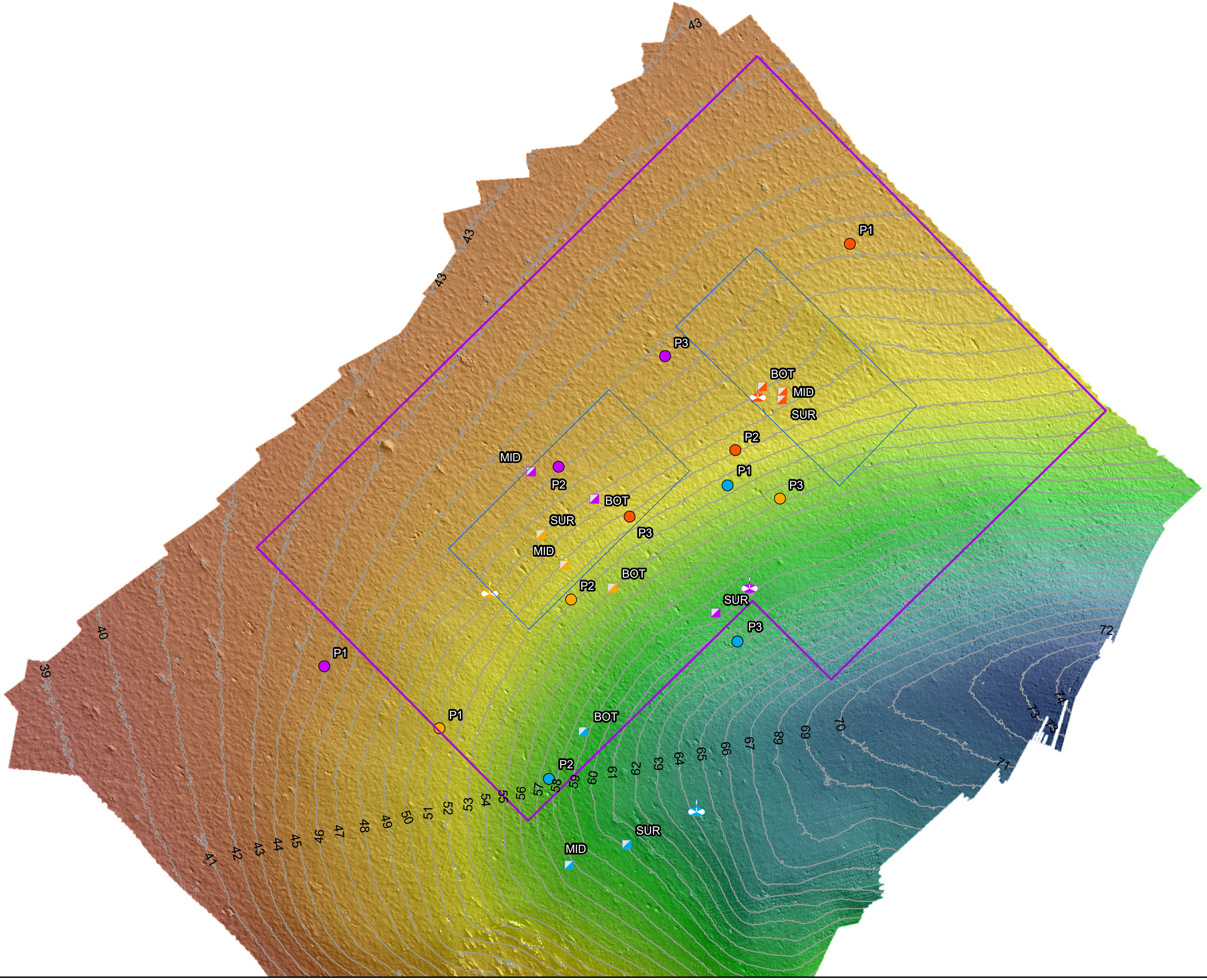
Site Address

FB01 - Long Porcupine Island
Frenchman Bay
Gouldsboro, Maine

201.05012 | Mar 2021

Figure 1
Site Location Map

C:\Users\thomas.neilson\Documents\American Aquafarms\C



RANSOM
Consulting
Engineers
and Scientists

Legend & Notes

- ▭ Lease Boundary
- ▭ Mooring Grid Boundary

Jul 2020 Sampling
- ● Profile
- ◩ Sample
- ✢ Secchi

Aug 2020 Sampling
- ● Profile
- ◩ Sample
- ✢ Secchi

Sep 2020 Sampling
- ● Profile
- ◩ Sample
- ✢ Secchi

Oct 2020 Sampling
- ● Profile
- ◩ Sample
- ✢ Secchi

— Bathy. Contour (m MLLW)

General Notes:

1. Basemap from 50 cm multibeam bathymetric survey. All depths reference to meters MLLW.
2. Some features shown are approximate in location and scale.
3. Sample locations are geographically averaged points from drift tracks.
4. This map was prepared for American Aquafarms. All other uses are not permitted unless written permission is obtained from Ransom Consulting, LLC.

Scale & Orientation

0 125 250

1 inch = 300 feet

Prepared For

American Aquafarms
68 Commercial Street
Portland, Maine

Site Address

FB01 - Long Porcupine Island
Frenchman Bay
Gouldsboro, Maine

201.05012 *Mar 2021*

Figure 2
Sample Location Map

C:\Users\thomas.neilson\Documents\American Aquafarms\

APPENDIX A

Sampling and Analysis Plan (SAP)
Water Quality Monitoring – Frenchman Bay and Blue Hill Bay, Maine

Water Quality Monitoring Report
Frenchman Bay Site FB01
Hancock County, Maine

Ransom Consulting, LLC
Project 201.05012

TITLE PAGE

Document Title:

Sampling and Analysis Plan (SAP)
Water Quality Monitoring – Frenchman Bay and Blue Hill Bay, Maine

Preparer's Name and Organizational Affiliation:

Eriksen Phenix, L.G. and Elizabeth Ransom, P.G.
Ransom Consulting, LLC (Ransom)
400 Commercial Street, Suite 404, Portland, Maine 04101
(207) 772-2891

Preparation Date:
November 12, 2020

Ransom Project Manager: Elizabeth M. Ransom
Signature/Date
Elizabeth Ransom/Ransom
Printed Name/Organization

Ransom Project QA Officer: Erik Pheng
Signature/Date
Eriksen Phenix/Ransom
Printed Name/Organization

MEDEP Project Biologist/Technical Reviewer: ______________________
Signature/Date
Angela Brewer /MEDEP
Printed Name/Organization

TABLE OF CONTENTS

TABLES

FIGURES

APPENDICES

1.0 INTRODUCTION

This SAP has been prepared in support of American Aquafarms' aquaculture project lease area selection and future permit applications for the Maine Department of Marine Resources (MDMR) and Maine Department of Environmental Protection (MEDEP). The purpose of this SAP is to outline the field and analytical protocols, sampling locations, data quality measures, schedule, and personnel to be used in conjunction with the American Aquafarm's aquaculture water quality monitoring project. The SAP is based on the methodology presented in the Marine Environmental Monitoring Program (MEMP) Quality Assurance Project Plan (QAPP) dated April 7, 2017.

2.0 PROJECT FRAMEWORK

This water quality monitoring program has been developed to assess the existing water quality conditions at three potential lease sites in Frenchman Bay and Blue Hill Bay. Potential lease sites are shown on the attached site plans Figure 2-1 and Figure 2-2.

Ransom will perform water quality monitoring monthly at each of these sites in July, August, September, and October 2020. Monitoring will be conducted in general conformance with the MEDEP Marine Environmental Monitoring Program QAPP to ensure that collected data will be of sufficient quality to be scientifically defensible.

Each month Ransom will conduct in-situ vertical water column characterization at each potential lease site from the surface to a maximum depth of 200 feet using a water quality multimeter. Water column profiles will record the following six parameters:

1. Depth;
2. Temperature;
3. Conductivity;
4. Dissolved oxygen;
5. pH; and
6. Turbidity.

In addition to the six parameters listed above, water clarity will also be measured at each potential lease site using a Secchi disk. As directed by MDMR, water column profiles will be collected at two different points in the tidal cycle and on at least one occasion within two hours of sunrise.

During each monthly event, discrete water quality samples will be collected from each proposed lease site at three depths identified as "Near Surface", "Mid Column", and "Near Bottom". These sampling depths are defined as follows:

Near Surface – 0.2 meters below the water surface.

Mid Column - ½ the total depth where the vessel is positioned, at the time of sampling.

Near Bottom - 2 meters above the bottom, or sampler line fully payed out (200 ft).

Water quality samples will be submitted for laboratory analysis of the following seven parameters:

1. Nitrate + nitrite;
2. Ammonia;
3. Total kjeldahl nitrogen;
4. Total nitrogen mixed forms;
5. Orthophosphate;
6. Total phosphorous; and
7. Total suspended solids.

Samples will be collected into laboratory prepared containers and transported under chain-of-custody protocol to a Maine accredited laboratory for analysis. One duplicate sample will be collected to adhere to a 10% sample replication rate. This parameter list and number of samples may be amended based on continuing discussions with MEDEP and MDMR.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY FLOW CHART

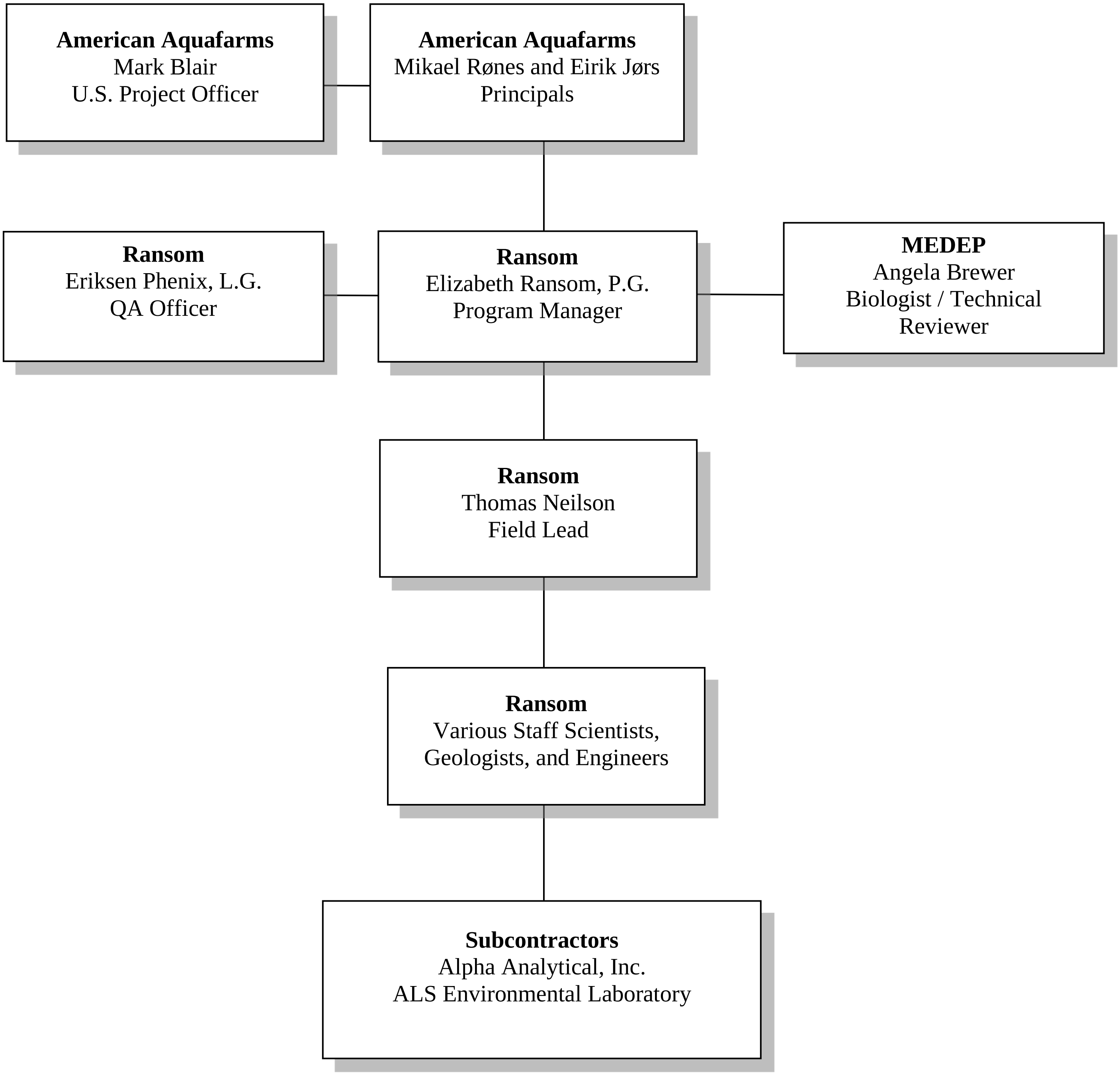
This section provides a brief description of how the ambient Water Quality Monitoring program in the areas of Frenchman Bay and Blue Hill Bay will be organized, including identification of the key project personnel, their responsibilities, and a flow chart showing the project chain of command.

The following page presents a Project Organization Chart depicting the agencies and companies involved with this project. Table 3-1 below describes each participant's role in this project.

Table 3-1: Project Personnel Responsibilities

Name	Title	Organizational Affiliation	Responsibilities
Angela Brewer	Biologist / Technical Reviewer	MEDEP	Reviews SAP and general project modifications. Provides technical review and assistance.
Elizabeth Ransom, P.G.	Project Manager	Ransom	Provides overall technical direction and project management for Ransom. Serves as project manager in charge of overseeing field work; oversees subcontractor coordination and field activities; coordinates data collection; participates in data interpretation and preparation of deliverables.
Eriksen Phenix, L.G.	QA Officer	Ransom	Provides project quality assurance oversight and review of deliverables.
Thomas Neilson	Project Geologist/ Field Lead	Ransom	Day-to-day technical lead in charge of field work; coordinates and conducts data collection; participates in data interpretation and preparation of deliverables; communicates and coordinates with subcontractors.
Additional Staff	Project Engineer/ Scientist/ Geologist	Ransom	Conducts field activities with oversight from Project Manager; oversees subcontractor field activities; communicates and coordinates with Project Manager and Field Lead.

Figure 3-1: Project Organization Chart



4.0 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are qualitative and quantitative statements that specify the quality and quantity of data needed to support technical decisions during site assessments. DQOs are developed by considering the purpose of collecting the data and the intended use of the data. The objective of the Water Quality Monitoring program is to collect sufficient data to characterize the ambient water quality and characteristics of the proposed lease areas.

Data Quality Indicators (DQIs) for the proposed water quality monitoring parameters are identified in Table 4-1 below:

Table 4-1: Data Quality Indicators for Ambient Water Quality Measurement Parameters

Parameter	Instrument	Measurement Range	Accuracy	Resolution
Depth	YSI EXO1	0-100 m	±0.04 % of full range	0.001 m
Temperature	YSI EXO1	-5 to +50 ºC	±0.01 ºC	0.001 ºC
Conductivity	YSI EXO1	0 to 200 mS/cm	±0.5% of reading or 0.001 mS/cm	0.00001 to 0.01 mS/cm
Dissolved Oxygen	YSI EXO1	0 to 50 mg/L 0 to 500% air saturation	±1% of reading or 0.1 mg/L ±1% of reading or 1% saturation	0.01 mg/L 0.1% air Saturation
pH	YSI EXO1	0 to 14 SU	±0.01 SU within ±10 ºC of cal. temp ±0.2 SU all other temps	0.01 SU
Turbidity	YSI EXO1	0 to 4,000 FNU	0.3 FNU or ±3% of reading	0.01 to 0.1 FNU
Water Clarity	Secchi disk	0.1 m to 10 m	n/a	0.1 m

Water quality analytical method objectives for discrete depth water quality samples are identified in Table 4-2 below.

Table 4-2: Water Quality Analytical Method Objectives

Analyte	United States Environmental Protection Agency (U.S. EPA) or Standard Method #	Alpha Analytical Inc. (Alpha) Ambient Water Reporting Limit	ALS Environmental (ALS) Ambient Water Reporting Limit
Nitrate + Nitrite	E353.2	0.1 mg/L	0.002 mg/L
Ammonia as N	E350.1	0.075 mg/L	0.001 mg/L
Total Kjeldahl Nitrogen	E351.3, 351.1	0.3 mg/L	0.100 mg/L
Orthophosphate as P	SM4500P	0.005 mg/L	Not Applicable
Total Phosphorus	SM4500P	0.01 mg/L	Not Applicable
Total Suspended Solids	SM2540 D	1.0 mg/L	1.0 mg/L

DQOs and criteria including accuracy, precision, representativeness, comparability, and completeness are discussed in Section A7 of the MEMP QAPP.

5.0 SAMPLING AND ANALYTICAL METHODS

The sampling program outlined in this SAP has been developed in consultation with MEDEP Marine Unit staff with the intention of producing reliable and comparable data for future decision-making purposes. The sampling and analytical methods proposed in this SAP are based on the methods presented in the MEMP QAPP. The MEMP QAPP was written for a broad array of MEMP activities. Based on the objectives of the monitoring program discussed in this SAP, the following deviation from the MEMP QAPP is noted: chlorophyll- A, chlorophyll A – phaeophytin and phaeophytin, as listed in the MEMP QAPP, will not be analyzed. Similarly, this program will not include continuous water quality monitoring using a fixed sonde for unattended data collection, or collection of light profile data.

Water quality monitoring and discrete depth water sampling proposed in this SAP will occur once per month at each of the potential lease sites in Frenchman's Bay and Blue Hill Bay. Proposed water quality monitoring and sampling program details are summarized in Table 5-1 below.

Table 5-1: Water Quality Monitoring and Sampling Plan

Study Area	**Lease Site (Latitude, Longitude, Deviation)**	**Analytical Sampling Zones(1)**	**Monitoring/ Sampling Schedule**	**Water Quality Monitoring Parameters**	**Grab Sample Analytical Parameters**
Frenchman's Bay	Site No. 1 (44.42 mN, -68.16 mE, ±0.25 nautical miles)	Near Surface Mid Column Near Bottom	July 22, 2020; August 20, 2020; September 16-17, 2020; October 21-22, 2020	Depth Temperature Salinity Dissolved Oxygen pH Turbidity Water Clarity	Total Suspended Solids Nitrate & Nitrite as N Ammonia as N Total Kjeldahl Nitrogen Total Nitrogen Mixed Forms Orthophosphate as P Total Phosphorus
	Site No. 2 (44.43 mN, -68.21 mE, ±0.25 nautical miles)	Near Surface Mid Column Near Bottom			
Blue Hill Bay	Site No. 4 (44.28 mN, -68.21 mE, ±0.25 nautical miles)	Near Surface Mid Column Near Bottom	July 23, 2020; August 19, 2020; September 16-17, 2020; October 21-22, 2020	Depth Temperature Salinity Dissolved Oxygen pH Turbidity Water Clarity	Total Suspended Solids Nitrate & Nitrite as N Ammonia as N Total Kjeldahl Nitrogen Total Nitrogen Mixed Forms Orthophosphate as P Total Phosphorus

(1) Near Surface = 0.2 meters below water surface.
Mid Column = ½ the total depth where the vessel is positioned, at the time of sampling.
Near Bottom = 2 meters above the bottom, or sampler line fully payed out (200 ft).

Water quality monitoring and sampling activities will be performed in accordance with the standard operating procedures included in Appendix A. Standardized field data sheets, as well as direct instrument logging, will be used for data collection. Standardized field data sheets are included in Appendix A.

Table 5-2 identifies the laboratory analyses to be performed, required sample volumes, containers, preservation, filtration, and maximum holding times. Applicable field sampling Standard Operating Procedures that are being used as guidance for this project's objectives are listed in Table 5-3 and have been included in Appendix A. Laboratory standard operating procedures are listed in Table 5-4 and have been included in Appendix B.

Table 5-2: Sample Collection and Analysis Methods Requirements

Analytical Parameter	Analytical Method and Lab SOP	Sample Volume	Containers (Number, size and type)	Preservation Requirements (chemical, temperature)	Field / Lab Filtered	Maximum Holding Time
Nitrate + Nitrite	E353.2 (Alpha SOP No. 2217 and/or ALS SOP GEN-353.2)	250 mL	1 Plastic	1:1 H_2SO_4	Yes - field	28 Days
Ammonia as N	E350.1 (Alpha SOP No. 2206 and/or ALS SOP GEN-350.1)	500 mL	1 Plastic	0.8 mL H_2SO_4	Yes - field	28 Days
Total Kjeldahl Nitrogen	E351.1 (Alpha SOP No. 2207 and/or ALS SOP GEN-351.2)	500 mL	1 Plastic	2 mL H_2SO_4	No	28 Days
Orthophosphate as P	E365.3, E365.1 (Alpha SOP No. 2225)	500 mL	1 Plastic	0 - 6°C	Yes - field	48 Hours
Total Phosphorus	SM4500P, E365.1 (Alpha SOP No. 2226)	500 mL	1 Plastic	H_2SO_4	No	28 Days
Total Suspended Solids	SM 2540 D (Alpha SOP No. 2220)	1 Liter	1 Plastic	0 - 6°C	No	7 Days

Table 5-3: Project Monitoring and Sampling SOPs Reference Table

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization
1	SOP for Navigation and Sample Site Location, Rev. 00, August 31, 2020.	Ransom
2	Water Quality Sonde Profile Data Collection and Processing, Rev. 01, April 7, 2017	MEDEP
5	Transparency Data Collection and Processing, Rev. 00, June 2016	MEDEP
6	Water Grab Sampling Collection and Processing, Rev. 00, June 2016	MEDEP

Table 5-4: Laboratory SOPs Reference Table

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization
1559	Sample Receipt and Log-In, Rev 26, April 15, 2020	Alpha
1560	Sample Custody and Tracking, Rev 18, April 15, 2020	Alpha
1561	Bottle Order Preparation, Rev 8, January 22, 2018	Alpha
2220	Total Suspended Solids Dried at 103-105 ºC, Total Volatile suspended Solids Dried at 500 ºC, Revision 16, April 22, 2020	Alpha
2217	Nitrate, Nitrite, and Nitrate/Nitrite Nitrogen, Revision 11, December 5, 2019	Alpha
2206	Nitrogen Ammonia, Revision 11, April 6, 2020	Alpha
2207	Nitrogen, Total Kjeldahl, Revision 9, February 8, 2019	Alpha
2225	Orthophosphate – Colorimetric, Combined Reagent, Revision 8, December 5, 2019	Alpha

Table 5-4: Laboratory SOPs Reference Table

SOP Reference Number	Title, Revision Date and/or Number	Originating Organization
2226	Total Phosphorous, Dissolved Phosphorus, Colorimetric, Combined Reagent, Revision 11, January 3, 2018	Alpha
GEN-350.1	Ammonia by Colorimetry, Revision 6, July 15, 2013	ALS
GEN-351.2	Nitrogen, Total Kjeldahl Block Digestion and Analysis by Flow Injection, Revision 6, June 1, 2014	ALS
GEN-353.2	Nitrate/Nitrite by Colorimetry, Revision 5, November 6, 2017	ALS
GEN-2540D	Total Suspended Solids, Revision 2, August 31, 2015	ALS

5.1 Sample Handling and Custody

Discrete depth water samples will be preserved, stored, and transported according to the methods described in SOP No. 6 and the holding times identified in Table 5-2 above. Samples will be collected in clean, laboratory provided sample containers, preserved at a temperature of approximately 4 ºC, and transported to the laboratory under chain-of-custody procedures. Sample receipt conditions will be reported in the laboratory analytical reports.

6.0 QUALITY CONTROL

For vertical sonde profiling, field instruments will be calibrated for each water quality parameter prior to each sampling event. Vertical sonde profile readings will be recorded electronically using the instruments data-logging capabilities and will be downloaded from the instrument for submission to the client and the MEDEP as part of the permit application process.

For the discrete depth water samples submitted for laboratory analysis, the MEMP QAPP will require the analytical laboratory to prepare Level II analytical data according to U.S. EPA protocols and U.S. EPA laboratory data validation guidance. The laboratory will be required to provide the following information in individual analytical reports:

1. Data results sheets;
2. Method blank results;
3. Matrix interference and data flagging;
4. Duplicate results/acceptance limits;
5. Spike/duplicate results/acceptance limits;
6. Laboratory control sample results;
7. Description of analytical methods and results;
8. Other pertinent results/limits as deemed appropriate; and
9. Laboratory narrative including sample receipt conditions and documenting any issues or problems that arose during the analysis.

Chemical analysis results and the Data Quality Packages will be reviewed by Ransom as part of data validation activities. Analytical measurement quality objectives used to assess laboratory quality control testing for dissolved and total nutrients are presented in Table 6-1 below.

Table 6-1: Data Quality Indicators for Laboratory Analytical Measurement Parameters

Quality Control Type	Frequency	Data Quality Indicator
Method Blanks	≥ 10% of samples	Less than reportable limit
Laboratory Duplicates	≥ 10% of samples	≤ 10% RPD
Laboratory Control Samples	≥ 10% of samples	90-110% recovery
Matrix Spikes	≥ 10% of samples	90-110% recovery

For discrete interval water samples submitted for laboratory analysis, one field duplicate will be collected for every 10 samples collected from each of the four distinct study areas. The duplicate sample will be submitted for laboratory analysis of all analytical parameters outlined in Section 5 above.

7.0 REPORTING

All field data and laboratory analytical results generated during the water quality monitoring program will be recorded in a format consistent with MEDEP e Electronic Data Deliverable (EDD) Version 6.0 with Validation Level and Validation Qualifiers populated. The data will ultimately be used in support of a waste water permit application.

8.0 REFERENCES

1. MEDEP, Bureau of Water Quality, Marine Unit, Division of Environmental Assessment, April 7, 2017; *“Marine Environmental Monitoring Program Quality Assurance Project Plan, Revision No. 01.”*

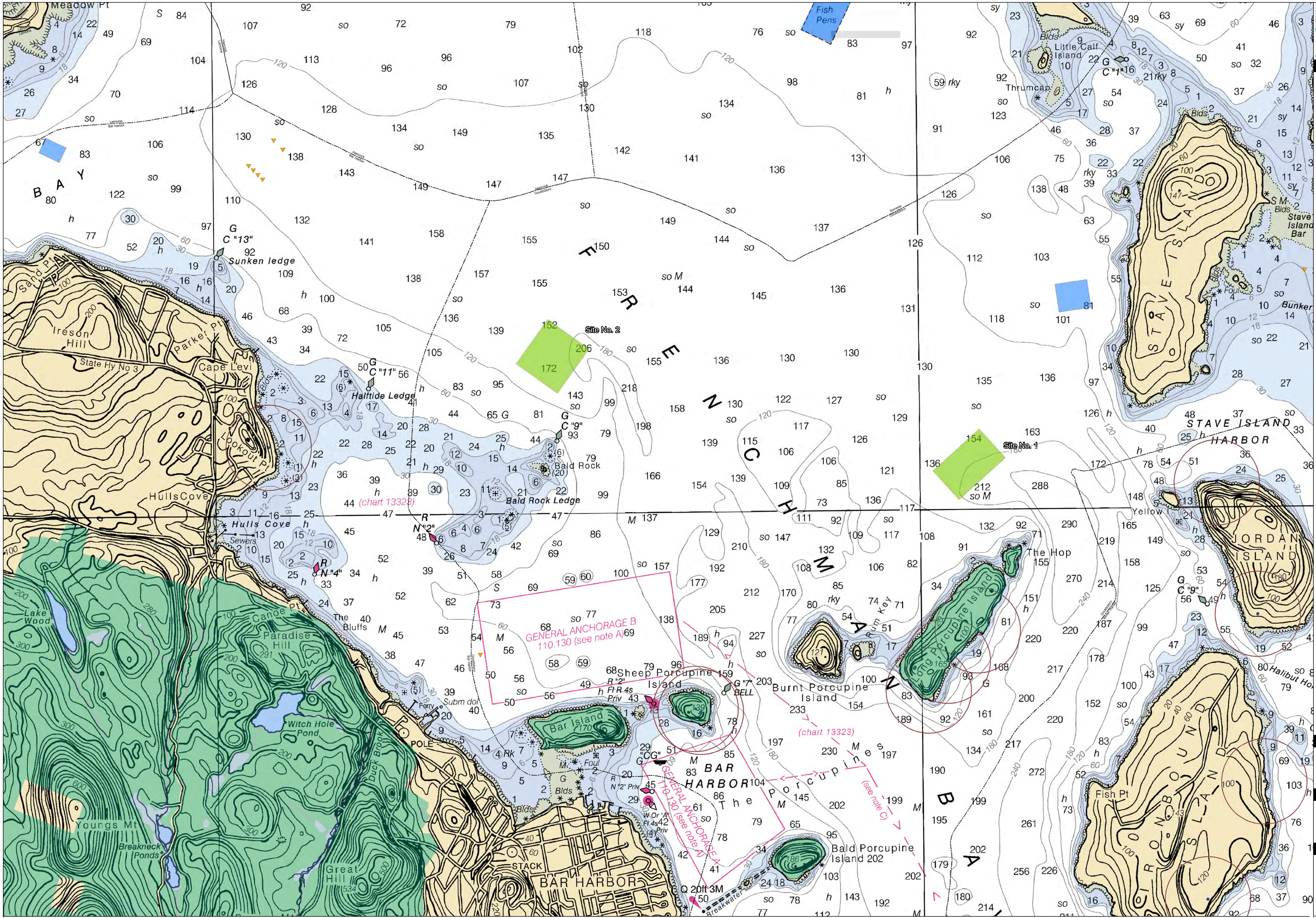
2. MEDEP, Bureau of Water Quality, Marine Unit, Division of Environmental Assessment, February 15, 2009; *“Standard Operating Procedure No. OC-QM-003, Sampling and Analysis Plan Approval, Revision No. 02.”*

PROJECT OVERSIGHT CERTIFICATION

To be signed by all persons, whether MEDEP employees or not, who are responsible for overseeing any and all work, including laboratory analysis, carried out under the terms of this Sampling and Analysis Plan.

"I certify that I have read and understand the requirements of this Sampling and Analysis Plan and will fulfill them to the greatest extent possible. I will assure that all staff, volunteers, etc., for whom I am responsible are familiar with these requirements and competent to carry out their responsibilities."

Name	**Position**	**Date**	**Signature**
Elizabeth Ransom, P.G.	Project Manager		
Thomas Neilson	Field Lead / Project Scientist		
Erik Phenix, L.G.	QA Officer		
Melissa Gulli	Alpha Analytical Inc. Laboratory Project Manager		



RANSOM
Consulting Engineers and Scientists

Legend and Notes

🟩 Potential Sites

LPA Lease Sites

▼ Active

Outstanding Natural Resources

🟩 National Park

▭ WATERBODY

Active/Pending Standard Lease

🟦 Shellfish/Algae/Other

Essential Habitats Nest Sites

● Essential Habitats Nest Sites

Historic EH Buffers

▭ Essential Habitat Boundary

Maine Town Boundaries

▭ Maine Town Boundaries

General Notes:

1. Basemap from NOAA RNC server.
2. Potential lease sites are shown based on available bathymetry, relative shelter, and additional available data.
3. Some features shown are approximate in location and scale.
4. GIS data accessed and downloaded from the Maine Office of GIS, NOAA Digital Coast, and/or The National Map.

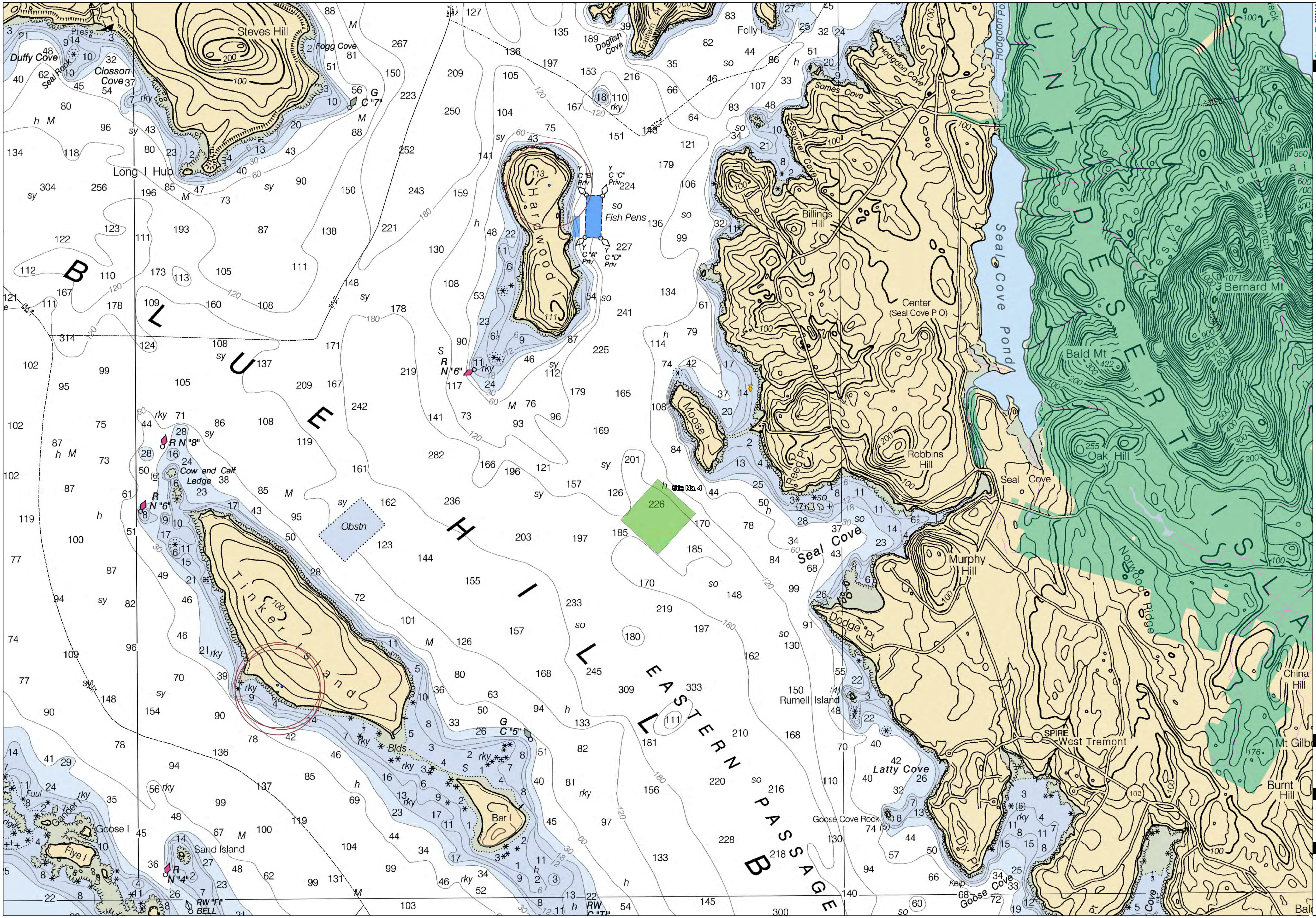
Prepared For

Orientation and Scale

0 625 1,250 2,500 Ft

1 inch = 1,250 feet

Figure 2-1
Proposed Lease Areas
Frenchman Bay



RANSOM
Consulting Engineers and Scientists

Legend and Notes

🟩 Potential Lease Areas

LPA Lease Sites

▼ Active

Outstanding Natural Resources

🟩 National Park

🟨 National Wildlife Refuge

Active/Pending Standard Lease

🟦 Shellfish/Algae/Other

Essential Habitats Nest Sites

● Essential Habitats Nest Sites

Historic EH Buffers

🟥 Essential Habitat Boundary

Maine Town Boundaries

⬚ Maine Town Boundaries

General Notes:

1. Basemap from NOAA RNC server.
2. Potential lease sites are shown based on available bathymetry, relative shelter, and additional available data.
3. Some features shown are approximate in location and scale.
4. GIS data accessed and downloaded from the Maine Office of GIS, NOAA Digital Coast, and/or The National Map.

Prepared For

Orientation and Scale

0 625 1,250 2,500 Ft

1 inch = 1,250 feet

Figure 2-2
Proposed Lease Area
Blue Hill Bay

APPENDIX A

Field Sampling Standard Operating Procedures

Sampling and Analysis Plan
Water Quality Monitoring, Frenchman Bay and Blue Hill Bay

RANSOM
Consulting, LLC

Consulting
Engineers
and Scientists

SOP FOR VESSEL NAVIGATION AND SAMPLE SITE LOCATION

Date: August 31, 2020
Revision Number: 0
Revision Date: August 31, 2020

This standard operating procedure (SOP) is written to describe the general procedures to be utilized by employees of Ransom Consulting, LLC (Ransom) in navigating a vessel to a sample site location in a marine environment. This SOP is not intended to describe specific navigational techniques, rules of the road, or the safe and proper operation of a vessel. It is the responsibility of the vessel operator to know and obey all required safety and navigational rules while operating the vessel, as well as to be familiar with all navigational techniques employed. Should the Vessel Operator feel that these conditions cannot be satisfied, the proposed work should be postponed until the situation is remedied. This SOP does not take the place of proper training of personnel.

General Applicability

This SOP applies to situations in which Ransom personnel are operating a vessel to be used for marine field work. This includes, but is not limited to, water quality monitoring, water sample collection, observation, reconnaissance, hydrographic surveying, and sediment sampling.

Health and Safety

It is the responsibility of all involved in the proposed work to maintain a safe and healthy working environment. Work completed under this SOP should have a dedicated Site-Specific Health and Safety Plan (SSHASP) which includes provisions explicitly addressing vessel safety. The SSHASP should be read and understood by all. Daily health and safety meetings shall be conducted, led by the Vessel Operator.

Equipment

To satisfy the procedures outlined in this SOP the following equipment is required. **Note that two forms of independent navigation shall always be carried. This can be two independent Global Positioning System (GPS) units with separate power sources, or a GPS unit and paper charts with appropriate tools, however, if one system should fail the second system must be able to be utilized without issue.**

1. VESSEL: The vessel used to transport field staff and equipment to and from the field site and serve as the platform from which to conduct operations should be appropriate for the proposed activity, expected range of weather and sea state, and for the level of exposure and distance from shore and save haven it will be operating.

2. GPS: A GPS should be utilized to facilitate easy and efficient navigation to the sampling sites. This can be integrated into the vessel or a handheld unit. It should be loaded with the appropriate up-to-date nautical charts for the area work is occurring, as well as all

transit areas. Whenever possible, geolocated information about sample site locations should be entered directly into the GPS unit. It is critical that thought go into the power source of the GPS unit if it is hardwired into the vessel electrical system. If battery powered, ensure batteries are charged and spares carried if applicable.

3. NAUTICAL CHARTS: Appropriate paper nautical charts should be carried for the areas the work is being conducted in. It is critical that paper charts are carried as a backup should the GPS system fail.

4. PILOTING TOOLS: In addition to nautical charts, dividers and parallel rules or triangles should be carried if manual piloting should be necessary. These are required if paper charts are to be used as the backup navigational system. If paper charts are being used as a primary or backup navigational method, it is good practice to lay out course lines for the route to the sample location with distance (nm) and bearing (magnetic and true) on the chart prior to departure.

5. COMPASS: The vessel should be equipped with a compass. If the vessel is not equipped with a compass, a handheld compass should be carried. A handheld compass should be appropriate for marine use. Compasses designed for orienteering are not an acceptable alternative. The compass should be periodically checked for accuracy against another compass.

6. VERY HIGH FREQUENCY (VHF) RADIO: The vessel should be equipped with a VHF radio. If this is not the case, a handheld radio can be used. Unless engaged in communication on another channel, the VHF should be on and monitoring Channel 16.

Training

All field staff involved in marine sampling activities should be appropriately trained in safety procedures, emergency vessel operation, and the sampling work being completed. The Vessel Operator should possess appropriate training and experience to navigate and operate the vessel and complete the proposed work.

Guidelines and Procedures

Because each field program may have different or unique equipment, this SOP cannot detail the procedures to use the equipment being employed. Instead, it provides a framework from which to work from in planning and navigating to and from field sites.

Prior to Field Program Start

Specific details about the field site(s), planned routes, and weather should be gathered prior to beginning field operations. At a minimum, this should include:

1. Geographic location of all field sites or areas in the appropriate coordinate system. If possible, this information should be pre-loaded onto the primary GPS unit to be used during work.

2. Location the vessel will be launched, or the transit will begin from. It is critical to identify the appropriate launch ramp or starting harbor early in the process. Care should be given to selecting a location with necessary services (e.g. launch

ramp large enough to accommodate the vessel, tide at which the ramp can be used, dockage/mooring for multiday field efforts, fuel and other vessel services, etc.).

3. Planned transit route. Once the field site and starting location information are in hand, it is important to lay out the proposed transit route to each field site. Care should be taken to select a route that is appropriate for the vessel being used and weather/sea state. The planned route should be entered into the GPS, if possible, and should be laid out appropriately on paper charts (course line, distance in nm, bearing in magnetic and true), should they be used as a primary or backup navigational system.

4. Marine forecast for proposed field day(s). Weather should be closely monitored the week before the proposed field operations. If weather is not favorable, the field work should be postponed. Key considerations for weather are:

    a. Wind Speed and direction;
    b. Swell height and direction; and
    c. Visibility.

    If visibility is forecast to be poor (e.g. fog) careful consideration should be given to safe navigation to and from the field site, as well as to safe operation during field activities.

    Weather forecasts should be confirmed each day prior to beginning field operations and should be assessed based on the nature of the proposed work, equipment at hand, and the SSHASP for the field program.

5. A basic field plan outlining the proposed transit route and field areas should be filed with the Project Manager or other project staff not onboard the vessel prior to departure.

Transit To and From Field Sites

Transit to and from field sites should be completed using navigational techniques and methods appropriate to the vessel, weather conditions, and sea state. If visibility is poor, the Vessel Operator should designate another member of the field team to act as lookout during the transit, or until such time as a lookout is no longer needed.

Conducting Field Operations

Once the Vessel Operator has navigated to the field site, field operations can begin. This may include anchoring, determining wind and current directions and drifting, or actively maneuvering. It is the Vessel Operators responsibility to operate the vessel safely during field operations, while also supporting the work mission.

Appendix B2

Water Quality Sonde Profile Data Collection and Processing Standard Operating Procedures

1. **APPLICABILITY.** This Standard Operating Procedure (SOP) applies to employees, contractors, and volunteers of the Marine Unit, Division of Environmental Assessment, Bureau of Water Quality, that are engaged with activities related to water quality sonde profile data collection and processing as part of the Marine Environmental Monitoring Program.

2. **PURPOSE.** Establishing standardized methods for performing common monitoring tasks improves the Marine Unit's efficiency, consistency, verifiability, and credibility. This SOP describes the methodology for preparing, utilizing, and storing a YSI Incorporated (YSI) multiparameter sonde, as well as data management of sonde data. In this context, the multiparameter sonde is used for *in situ* profiling of the water column in the marine (estuarine and coastal) environment. Water column profiles provide information about common parameters including temperature, salinity, optical dissolved oxygen, pH, turbidity and chlorophyll at varying depths from the water surface to the benthic surface.

3. **DEFINITIONS.**

3.1 CALIBRATION: The set of procedures established by the sonde manufacturer to ensure instrument accuracy, a critical quality assurance step in instrument preparation prior to use.

3.2 ECOWATCH. The YSI software used to upload and visualize sonde data, and activate/deactivate individual sensors.

3.3 EGAD. The Department's Environmental and Geographic Analysis Database.

3.4 MULTIPARAMETER SONDE. Water quality monitoring instrument with integrated depth sensor and the capability to accept several probes/sensors to instantaneously measure a variety of real-time conditions. Instrument can store data internally and also integrate with a handheld computer.

3.5 PROBE/SENSOR. Replaceable module that inserts into the end of the multiparameter sonde, and measures a particular water quality parameter. Probes/sensors generally require calibration and may or may not have an attached wiper on the tip to combat fouling.

3.6 PROFILE. The action of lowering or raising a multiparameter sonde slowly through the water column to allow sensor reading stabilization and recording to characterize conditions at discrete depths.

3.7 YSI. YSI Incorporated, the company that manufactured the multiparameter sonde in use by the Marine Unit, that is the subject of this SOP.

4. **RESPONSIBILITIES.**

4.1 TRAINING. The Program Lead or designee is responsible for overseeing staff training for the proper calibration or operation of a multiparameter sonde prior to the start of each field season. The Program Lead must remind staff annually of the steps necessary to ensure data entry, review, verification and validation per specifications in the Marine Environmental Monitoring Program QAPP and this SOP.

4.2 COMPLIANCE. All staff engaged in calibration or operation of a multiparameter sonde or management of sonde data are responsible for becoming familiar and complying with all procedures within this SOP and the associated instrumentation and software.

4.3 DATA RECORDING. Any staff involved in operation of a multiparameter sonde must record associated data on a field datasheet, internally to the instrument, or to the handheld computer, as appropriate. Hard copy datasheets must be placed by field staff in the relevant project folder managed by the Program Lead as soon as possible after return from the field. Field staff must identify for the Program Lead, by e-mail or hard copy record, the whereabouts and name of any electronic files and associated file names.

4.4 REPORTING. Any staff operating a multiparameter sonde must return the instrument in a condition that is equal to or better than when first encountered upon that use. Any problems or concerns noted with the instrument or data generated by the instrument must be reported by the end of the field effort to the Program Lead.

4.5 DATA MANAGEMENT. The Program Lead has ultimate responsibility for data review, verification and validation of all multiparameter sonde profile data. Validated data of sufficient quality should be formatted and uploaded to the EGAD in a timely manner, either by the Program Lead or designee.

5. GUIDELINES AND PROCEDURES. The language below details the proper guidelines and procedures for operation of a YSI multiparameter sonde and management of sonde data. Marine Unit water column sonde data collection will occur using an unvented, YSI 6600 V2 (ID # MARU1). The sonde is connected to a handheld computer, YSI 650 MDS, by a waterproof cable of length most appropriate for the relevant effort. The 650 MDS allows for user manipulation of MDS as well as sonde settings when connected to the sonde via cable. Specific details of sonde (YSI 2012) and computer (YSI 2001) capabilities and procedures are provided at the following links:

sonde (6600): https://www.ysi.com/File%20Library/Documents/Manuals/069300-YSI-6-Series-Manual-RevJ.pdf .
computer (650): https://www.ysi.com/File%20Library/Documents/Manuals%20for%20Discontinued%20Products/655228-YSI-650-Operations-Manual-RevB.pdf

5.1 SONDE CALIBRATION. Prior to each sampling event, the sonde will be calibrated in the DEP field support area or boat barn using the current barometric pressure and fresh, YSI-manufactured standards that are newer than the designated expiration date upon use. Previously used, clean standard may be used to rinse sensors but should not be used for calibration. Calibration values, standard solution expirations, and related information will be recorded on the

Sonde Calibration Form (Appendix C, #1). If water quality profiling is to occur within different estuaries on three subsequent days, for example, only one calibration event need occur at the beginning of the cumulative effort. Detailed calibration language for YSI sensors is presented in the "Continuous Monitoring of Water Quality SOP" (Appendix B, #3) and in YSI (2001; 2012). As such, detailed calibration language is not repeated here.

5.2 FIELD PROCEDURES. Prior to departing for the field, a support line should be attached to the sonde body handle by removable clamp, and the line extended alongside, but not connected to, the sonde cable. The support line is used to alleviate stress on the sonde cable during profiling, and should be held by the operator conducting the profile. The sonde cable should be marked in 0.5 m intervals with brightly colored electrical tape. Tape placement should occur such that the tips of the sensor array should be the starting point for label measurement along the cable. Field staff also need to ensure that new spare batteries are available in the field for both the sonde and computer.

Upon departure from the boat dock or ramp, the sonde calibration cup should be replaced with the sonde protective cage, and sensors then submerged in a bucket of ambient seawater. The bucket and sonde should be secured as appropriate for the trip to the first site given expected conditions based on speed and wave height and direction. After approximately 20 minutes or upon arrival at the first site, a current barometric pressure for the study area should be acquired and the dissolved oxygen sensor recalibrated given the revised pressure and surface water temperature. Failure to recalibrate the dissolved oxygen sensor in the field could result in inaccurate field readings if a considerable difference from calibration barometric pressure occurs.

Once the sampling vessel is anchored on or drifting above over the sample site (see "Site Selection and Location SOP", Appendix B, #1), the datasheet should be populated with the site and sample point names, field crew identifications, date and time of arrival at site, GPS coordinates, and the current barometric pressure and percent dissolved oxygen saturation value after field calibration. The sonde and underwater cable should be connected to the MDS, lead weights attached as necessary to enable sinking of the sonde, and the sonde submerged just under the water surface. The MDS should display the following parameters: temperature (°C), salinity (ppt), pH, dissolved oxygen (% saturation and mg/L), turbidity (NTU), and chlorophyll (µg/l). For profiles, it is not necessary to have optical wipers activated due to the short duration of time in the water. Once determined that sufficient memory and battery power exist to log the profile data and that the MDS displays the correct date, time, and parameters of interest, the logging interval or discrete sample should be selected, the electronic data file named, and the sonde then lowered incrementally through the water column. Work within shallow water (≤5 m depth) will require parameter readings to be collected 0.1 m below the surface and then at every 0.5 m within the water column from the surface to 0.5 m from the bottom. Work within deeper water (>5 m depth) will require data collection at 0.1 m and 0.5 m below the surface, and then every 1.0 m through the water column until reaching 0.5 m from the bottom.

If profiles extend beyond 10 m water depth, sampling intervals may be every 2-5 m based on discretion of the Program Lead or designee.

Data from discrete depths will be recorded either on the MDS, internally to the sonde, or if neither are possible due to malfunction or file storage concerns, data values may be recorded on a datasheet (Appendix D, #4). Data recording need only occur on the sonde downcast unless rapid mixing is occurring, in which case readings on the down- and upcasts are desirable to calculate average localized parameter variability. In order to record the most representative value per sensor per depth, the individual viewing the sonde data readout should allow sensor values to stabilize as much as possible before either choosing the “log one sample” option or recording to a datasheet. The “log one sample” option simultaneously records all sensor values at a given time and depth, and is the preferred data logging/recording method for sonde profiling. The sensor that typically takes the longest to stabilize is the pH sensor, and therefore this sensor should be used to determine when stable readings warrant the logging or recording of a sample. The turbidity and chlorophyll sensor values fluctuate continuously based on passing suspended and fluorescing material, respectively, and so these sensors should not be used to determine stability of readings at a given depth.

Once a profile has been completed, the sonde operator should review the file using the MDS “Quick View File” function. If the profile data appear unusual, uncharacteristic for the particular site, or if one or more depths through the water column were not properly logged, the downcast profile should be repeated. If the data file is acceptable, the sonde should be retrieved from the water, stored with its protective cage over the sensors in a bucket of ambient water that at least covers the sensors, and kept cool and out of direct sun exposure as much as possible. Additional field observations should then be recorded on the datasheet, including weather conditions, wind direction and speed, Beaufort scale, and any qualitative observations of relevant physical or biological conditions.

When sonde use is complete for the day, approximately 0.5 inch of clean tap water should be poured into the sonde calibration cup and then this cup should replace the protective cage over the sensor array. The sonde should be inserted securely into the YSI case to ensure shock protection during transport in the field vehicle.

5.3 SONDE POST-CALIBRATION AND SHORT-TERM STORAGE. Upon arrival at the boat barn, the calibration cup should be unscrewed and any debris on the sonde or sensors, such as filamentous algae, should be removed. Basic cleaning methods, as needed, should be based on YSI (2012) recommendations. The sonde body and sensor array should be rinsed thoroughly with cool tap water, and approximately 0.5 inch of tap water added to the calibration cup. The cup should be screwed on securely over the sensor array. Sonde post-calibration may happen at this time, ideally within a week following the completion of the sampling event, or absolutely prior to the next sampling event.

Post-calibration procedures should follow manufacturer recommendations, and any post-calibration values or errors recorded on the Sonde Calibration/Post-Calibration Form (Appendix C, #1). Since fouling and sensor drift are not anticipated during profiling, post-calibration values should not differ unless there is a substantial difference in barometric pressure or a probe malfunction. If post-calibration values for any probe exceed the acceptable range for measurement accuracy established in QAPP Table 3, then affected data collected during that relevant sampling events will be flagged as estimated ("J") when uploaded to EGAD or rejected ("R") and not uploaded to EGAD.

Short-term storage of the sonde may occur with the sensors in the calibration cup with no more than 0.5 inch of tap water (as described above) to keep the sensors moist but not submerged.

5.4 SONDE MAINTENANCE AND LONG-TERM STORAGE. The Sonde Maintenance Form (Appendix C, #2) is used to record information about sensor maintenance, replacements, and expirations based on manufacture date. Long term storage should include thorough cleaning of the sonde and sensors with tap followed by deionized water, complete drying of all components before storage, removal of batteries and probes from the sonde body, and placement of the sonde, probes and accessories in the proper containers (probes in sealable plastic bags, sonde in YSI case) in a warm location.

5.5 DATA MANAGEMENT. Data should be uploaded to EcoWatch as soon after collection as possible while the sampling event is still fresh in memory. Uploading requires use of a transmission cable and should follow directions provided in YSI (2001). Raw data files must be viewed using EcoWatch plots to identify any immediate data concerns, and then data parsed out into separate Excel files per date and site, as appropriate, and saved to the relevant project folder on a network server. Any data concerns should be noted prominently within the raw data Excel spreadsheet. Raw data in Excel files will later be reviewed as part of the detailed quality checking procedure outlined in the "Continuous Monitoring of Water Quality SOP".

6. REFERENCES.

YSI Incorporated. 2001. *YSI 650 MDS Operations Manual.* Yellow Springs, Ohio. Rev. B.

YSI Incorporated. 2012. *6-Series Multiparameter Water Quality Sondes User Manual.* Yellow Springs, Ohio. Rev. J.

1. **APPLICABILITY.** This Standard Operating Procedure (SOP) applies to employees, contractors, and volunteers of the Marine Unit, Division of Environmental Assessment, Bureau of Water Quality, that are engaged with activities related to Secchi disk transparency data collection and processing as part of the Marine Environmental Monitoring Program.

2. **PURPOSE.** Establishing standardized methods for performing common monitoring tasks improves the Marine Unit's efficiency, consistency, verifiability, and credibility. This SOP describes the methodology for utilizing a Secchi disk for measurements of marine water column transparency or clarity, as well as management of transparency data. Water column transparency provides information about the amount of color, dissolved and suspended material in the water column, and is a proxy for light penetration to greater depths and the benthic surface.

3. **DEFINITIONS.**

3.1 EGAD. The Department's Environmental and Geographic Analysis Database.

3.2 SECCHI DISK. A weighted circular plate 20 cm in diameter with opposing black and white quarters painted on the top surface. The disk is used to determine the transparency/clarity of the marine water and is attached to a low stretch fiberglass tape that is marked to 100th of a meter.

3.3 TRANSPARENCY/CLARITY. The distance that light penetrates through marine water as determined using a Secchi disk, fiberglass tape, and human eye.

4. **RESPONSIBILITIES.**

4.1 TRAINING. The Program Lead or designee is responsible for overseeing staff training for the proper operation of a Secchi disk prior to the start of each field season. The Program Lead must remind staff annually of the steps necessary to ensure data entry, review, verification and validation per specifications in the Marine Environmental Monitoring Program QAPP and this SOP.

4.2 COMPLIANCE. All staff engaged in operation of a Secchi disk or management of transparency data are responsible for becoming familiar and complying with all procedures within this SOP.

4.3 DATA RECORDING. Any staff involved in operation of a Secchi disk must record associated disappearance and reappearance data on a field datasheet. Hard copy datasheets must be placed by field staff in the relevant project folder managed by the Program Lead as soon as possible after return from the field. Field staff must identify for the Program Lead, by e-mail or hard copy record, the whereabouts and name of any electronic files and associated file names.

4.4 REPORTING. Any staff operating a Secchi disk must return the instrument in a condition that is equal to or better than when first encountered upon that use. Any problems or concerns noted with the instrument or data generated by the instrument must be reported by the end of the field effort to the Program Lead.

4.5 DATA MANAGEMENT. The Program Lead has ultimate responsibility for data review, verification and validation of all Secchi disk transparency data. Validated data of sufficient quality should be formatted and uploaded to the EGAD in a timely manner, either by the Program Lead or designee.

5. GUIDELINES AND PROCEDURES. The language below details the proper guidelines and procedures for operation of a Secchi disk and management of transparency data.

5.1 SAMPLING LOCATION. Secchi disk readings should be taken at the site selected by the Program Lead or designee, and should always occur in the shadow of the boat or other structure such as a dock, ideally between 9:00 AM and 3:00 PM. Readings should be taken while the boat is anchored as long as there is minimal surface current and the weighted disk is able to drop through the water column perpendicular to the water surface. If greater surface current prevents the disk from descending vertically through the water column, the boat should be set adrift over the sample location while Secchi readings are taken. If the surface of the water is disrupted by windy conditions or vessel wakes and it is possible to wait until the disturbance has passed, this is advisable. If it is not possible for the disturbance to pass before taking Secchi readings, the operator may take readings as best as possible and appropriately annotate on the datasheet (Appendix C, #4) to what degree the transparency readings may have been compromised.

5.2 COLLECTING DATA. While taking readings, the operator should wear neither a hat nor sunglasses, nor use a view scope. Prior to placing the Secchi disk into the water, the operator should ensure that the disk is securely attached to the end of the fiberglass tape, and then pull an excess amount of tape from the reel to minimize 1) the potential for disk loss into the water and 2) the amount of tape manipulation needed during observations. The operator should then pinch the tape between fingers and allow the disk to slowly descend through the water column. When the disk starts fading from view, the descent speed should be slowed and the observer should stop the disk just when the disk completely disappears from view (i.e. neither the white nor black quadrants or even a glow from the disk are visible). The observer should read the corresponding depth, to the nearest 0.01 m, on the tape where the tape penetrates the water surface, and audibly report this disappearance depth to another field staff member on the boat to record on the datasheet (Appendix C, #4). The observer then lowers the disk down at least a half meter in the water column, and then proceeds to slowly raise the disk through the water column. The depth at which any portion of the disk is visible, including glow from the disk, is then the reappearance depth.

The above procedure should be repeated two more times such that triplicate disappearance and reappearance values are recorded for each sampling location. If the disk hits bottom on any of the replicate casts, then the water depth at that location should be read from the fiberglass tape and recorded on the datasheet along with a notation that the disk hit bottom before disappearing from sight.

5.3 QUALITY CONTROL. If at all possible and to reduce inter-observer variability, the same observer should take all Secchi readings at each site per day of sampling effort. Additionally, a duplicate Secchi reading should be taken upon each tenth sample. The duplicate mean value should be within 0.3 m of the original reading and if not, readings should continue to be taken by the same observer until the readings stabilize and precision increases sufficiently.

5.4 EQUIPMENT MAINTENANCE AND STORAGE. The Secchi disk, attachment hardware, and any portion of the fiberglass tape that was submerged during Secchi readings should be thoroughly rinsed with freshwater upon return to the DEP field support area or boat barn. At least once during the field season and upon completion of the field season, the entire fiberglass tape should be unreeled and soaked in freshwater to remove any residual salt that may have dripped or run onto the unused portion of the tape.

The Secchi disk, attachment hardware and fiberglass tape should be stored between sampling events and seasons in a location that prevents scratching of the disk surface. Prior to the start of the sampling season, Secchi attachment hardware should be tightened or replaced if rusted, and the tape should be replaced if measurement labels are no longer readable or the tape is compromised and would potentially break under the weight of the disk. If the disk surface is faded or significantly scratched, the surface can be revived with a new decal or appropriate color of paint.

5.5 DATA MANAGEMENT. All raw disappearance and reappearance values will be entered by hand into an Excel spreadsheet as soon after collection as possible while the sampling event is still fresh in memory. The Excel spreadsheet should be formatted as an Electronic Data Deliverable (EDD) to enable quick uploading to EGAD. The Excel spreadsheet will be saved on a shared server, and be 100% quality checked by a different individual than entered the data. Once the final EDD is uploaded to EGAD, the Secchi data per site can be reported as three means that represent the average of each triplicate disappearance and reappearance value. The user may then choose to take a single average plus/minus depth deviation depending on intent for the data.

1. APPLICABILITY. This Standard Operating Procedure (SOP) applies to employees, contractors, and volunteers of the Marine Unit, Division of Environmental Assessment, Bureau of Water Quality, that are engaged with activities related to ambient water grab sample collection and processing as part of the Marine Environmental Monitoring Program.

2. PURPOSE. Establishing standardized methods for performing common monitoring tasks improves the Marine Unit's efficiency, consistency, verifiability, and credibility. This SOP describes the methodology for collecting ambient water grab samples to enable measurements of marine nutrient concentrations and chlorophyll *a* biomass. Paired water column nutrients and chlorophyll *a* characteristics indicate the potential relationship between nutrient profile and availability, and phytoplankton response.

3. DEFINITIONS.

3.1 DARK BOTTLE. Opaque, wide mouth Nalgene bottle used to hold water grab samples to later be processed for chlorophyll *a* and phaeophytin ("chlorophyll *a*") analyses.

3.2 EGAD. The Department's Environmental and Geographic Analysis Database.

3.3 SYRINGE FILTER. A large barrel syringe with screw-on filter tip that enables field filtration of ambient water samples.

3.4 VACUUM FILTRATION MANIFOLD. Apparatus used with electrically-powered vacuum suction to filter water samples for processing of chlorophyll *a* samples. Manifold enables simultaneous filtration of up to three water samples.

3.5 VACUUM FILTRATION STAND. Apparatus used with hand-powered vacuum suction to filter a water sample for processing of chlorophyll a samples. Stand allows filtration of one sample at a time.

3.6 WATER SAMPLER. A horizontally-oriented acrylic or PVC tube with ends that close when triggered by messenger, to capture ambient water samples at a given depth. The tube may be opaque or clear, has no metals parts, and includes a port from which water may be dispensed into sample bottles. Sampler is intended to be lowered to the chosen water depth and then moved horizontally prior to closing the ends in order to capture water from that chosen depth.

4. RESPONSIBILITIES.

4.1 TRAINING. Prior to the start of each field season, the Program Lead or designee is responsible for overseeing staff training for the proper operation of the water sampler, field processing of grab samples for nutrient and chlorophyll *a* analyses, and filtration and handling of chlorophyll *a* samples upon return to the laboratory. The Program Lead must remind staff annually of the steps necessary to ensure documentation on laboratory Chain of Custody forms and after analysis, quality control and data handling procedures per specifications in the Marine Environmental Monitoring Program QAPP and this SOP.

4.2 COMPLIANCE. All staff engaged in operation of a water sampler, processing, handling and shipping of grab samples, and review and management of datasheets and laboratory data are responsible for becoming familiar and complying with all procedures within this SOP.

4.3 DATA RECORDING. Any staff involved in collection and field processing of water grab samples must record the time a sample was collected, the bottle identification, number of sample bottles filled at a particular site, whether field filtration occurred, and whether duplicate samples were collected. During laboratory filtration of samples for chlorophyll *a* analyses, volume filtered in mL must be recorded for each sample on a field datasheet as well as on the analytical laboratory Chain of Custody form. All hard copy datasheets must be placed by field staff in the relevant project folder managed by the Program Lead as soon as possible after return from the field and processing in the laboratory. Field staff must identify for the Program Lead, by e-mail or hard copy record, the whereabouts and name of any electronic files and associated file names.

4.4 REPORTING. Any staff operating a water sampler or vacuum filtration stand or manifold must leave the instrument in a condition that is equal to or better than when first encountered upon that use. This includes washing and rinsing of all equipment using fresh water and deionized water when possible, and disposal of filtrate after completion of chlorophyll *a* sample processing. Any problems or concerns noted with the equipment must be reported by the end of the field effort to the Program Lead.

4.5 DATA MANAGEMENT. The Program Lead has ultimate responsibility for data review, verification and validation of all grab sample data. Validated data of sufficient quality should be formatted and uploaded to the EGAD in a timely manner, either by the Program Lead or designee.

5. GUIDELINES AND PROCEDURES. The language below details the proper guidelines and procedures for collection and processing of ambient water samples for subsequent laboratory analysis, as well as for data management of laboratory-generated data.

5.1 GRAB SAMPLE COLLECTION. Water grab samples from relevant sites will be collected from just under the water surface or at depth depending on the monitoring questions. Sites where grabs only need be collected at the surface will be sampled by immersing a large High Density Polyethylene (HDPE) Nalgene collection bottle approximately 0.2 m below the water surface by hand, with the bottle mouth oriented toward the direction of flow. The bottle should be capped underwater and then rinsed three times with ambient marine water. After triple rinsing is complete, the water grab sample should be acquired. The bottle should be filled to ensure that all sample water needed for analyses can be obtained from the same grab.

If one or more grabs is/are desired at depths other than or in addition to at the water surface, a water sampler should be used for grabs at all depths at a particular site. The water sampler to be used may be of Kemmerer or Van Dorn style, and should be constructed of materials that do not contaminate the grab samples that may later be chemically analyzed. The operator should deploy the water sampler to the desired depth by viewing a fiberglass tape or other line that has been marked to show distance above the water sampler opening. During this deployment, the operator should keep water sampler caps open and messenger in hand. When at the correct depth, the operator should move the water sampler at least 1 m horizontally and then drop the messenger with a small amount of downward force. The sensation of the messenger hitting the top of the water sampler and closing the caps should be apparent to the operator holding the tape/line. Once the sampler has closed, the device may be reeled back to the boat. This first drop serves as the first rinse at the sample depth and should be repeated two more times, ensuring that each time the water sampler is brought up to the boat, the water sampler is rotated or agitated thoroughly so that every interior surface is rinsed by the water, including the spigot. The HDPE collection bottle described above should also be triple rinsed as part of this process. After triple rinsing is complete at a particular depth, the grab sample should be collected and dispensed into the collection sample bottle.

5.2 FIELD SAMPLE PROCESSING. Grab samples are collected for whole water analysis of Total Kjeldahl Nitrogen (TKN), Total Nitrogen (TN) and/or Total Phosphorus (TP), dissolved analyses for nitrate + nitrite (NO_3^- + NO_2^-), ammonium (NH_4^+) and/or orthophosphate (PO_4^-), and filtered analysis of chlorophyll *a* and phaeophytin. All laboratory-provided nutrient sample bottles as well as dark bottles for chlorophyll *a* analysis should be labeled before adding sample water, and should include site name, date and time collected, sample depth, analyte(s), and sample collector’s initials. Once laboratory-provided nutrient bottles are open, special caution should be taken to keep those bottles upright that contain preservative (sulfuric acid (H_2SO_4), e.g.).

After gentle agitation, whole water samples for nutrient analyses should be transferred directly from the collection sample bottle to the laboratory-provided bottles and filled 2/3 to 3/4 full and capped tightly. Again after gentle agitation, the remaining sample to be analyzed for dissolved nutrients should be pulled into a new or clean, large barrel syringe, the barrel rinsed thoroughly so that all interior surfaces are touched by the sample water, and then the rinse water disposed. This process should be repeated two more times. Once triplicate rinsing with sample water is completed, the syringe operator should fill the syringe with sample water, screw on a new 0.45 µm pore size syringe filter tip, and then dispense the filtrate into the relevant pre-labeled sample bottles. The sample bottles should be filled 2/3 to 3/4 full and capped tightly. Again after gentle agitation, the remaining sample within the collection bottle should be used to triple rinse a single opaque, wide mouth Nalgene bottle (dark bottle). After the triple rinse is complete, the dark bottle should be filled and capped. If sample water at a particular location is especially lacking in visible particulate material, a second dark bottle should be filled if possible.

All sample bottles, including those for whole and dissolved nutrients, and chlorophyll *a* analyses, should be placed on ice (sample temperature kept at 0-6 °C) in a dark cooler immediately upon collection and until arrival at a laboratory refrigerator or until shipped.

5.3 LABORATORY SAMPLE PROCESSING. Processing of whole water samples for chlorophyll *a* analyses should occur as soon as possible after collection and no more than 24 hours post-collection. The filter stand or filtration manifold should be prepared by making sure that the filtrate flask or carboy has sufficient space to accept additional filtrate, all hoses are tightly attached, and that all components for the filtration apparatus (flask, funnel pedestal with stopper, filter cup) are clean and ready for use. A box of 45 mm diameter, 0.70 µm pore size glass fiber filters and two sets of clean forceps should be located adjacent to the filtration apparatus.

When ready for filtration, the room should be darkened as much as possible and then using forceps, a new filter positioned on the funnel pedestal and the filter cup secured to the pedestal. The dark bottle should be gently agitated and then using a clean graduated cylinder, 100 mL of sample water measured and poured into the filter cup. While 100 mL is a good starting point for sample volume, more or less volume may be required to achieve sufficient color on the filter without clogging the filter with overlying water remaining. The operator should turn on the vacuum pump or start manually pumping, and water should start passing through the filter. If using a filtration manifold, the operator should ensure that the stopcocks are open for only those filter stands for which filtration is desired. As soon as the sample volume has completely passed through the filter, the operator should turn off the pump, assess the filter surface after removing it at least partially from the filter stand to determine if a light amount of color is present, and should not add any further sample water if this is the case. If more color is needed, an additional measured volume should be added to the filter cup.

When filtration is complete, the total volume filtered should be recorded on the field datasheet and then the filter cup removed and the filter gently folded twice into quarters using two sets of forceps. The filter should be placed, being careful not to excessively squeeze the filter, onto a square of foil, and then the foil folded over the filter. The foil should be labeled by indelible marker with site name, date and time collected, sample depth, volume filtered, and sample filterer’s initials. Foil packets should be placed in a Ziploc bag in the freezer until all samples have been filtered and are ready for packing and shipment. The filter cup and filtration surface, graduated cylinder(s) and filter forceps must be rinsed three times with tap water followed by three times with deionized water prior to filtration of the subsequent sample.

5.4 SAMPLE HANDLING AND SHIPPING. The relevant information from nutrient bottles and chlorophyll *a* foil packets should be transferred to the relevant laboratory Chain of Custody form. The remaining project and custody information should be filled out, and samples packed into coolers lined with a trash bag and filled with wet ice. The trash bag should be tied or cinched tightly, necessary paperwork placed within a Ziploc bag on top of the trash bag, and then cooler taped liberally and sealed with a custody seal. Coolers should be relinquished to and shipped by the laboratory-preferred carrier as soon as possible after sample collection and processing to allow the laboratory sufficient time to extract and analyze the samples within the required holding time. Holding times for all analytes noted above is 28 days with the exception of orthophosphate, with a holding time of 48 hours.

5.5 QUALITY CONTROL. Field duplicate samples for nutrients and chlorophyll *a* will be collected for every 10th grab sample and treated identically as the paired sample.

5.6 EQUIPMENT MAINTENANCE AND STORAGE. The collection bottle, syringe (if being reused), and water sampler assembly, including attachment hardware and any portion of the fiberglass tape or line that was submerged during grab sampling, should be thoroughly rinsed with freshwater upon return to the lab or boat barn. The water sampler should be left open to dry between sampling events. At least once during the field season and upon completion of the field season, the entire fiberglass tape or line should be unreeled and soaked in freshwater to remove any residual salt that may have dripped or run onto the unused portion of the tape/line.

The filter stand or filtration manifold, graduated cylinder, and filter forceps should be washed with hot, soapy water and then rinsed thoroughly with tap water followed by deionized water. All parts should be air dried and then stored in a dry location. Any filtrate in the flask or carboy should be disposed.

5.7 DATA MANAGEMENT. All laboratory-analyzed data will be transmitted to the Program Lead in the form of a lab report and Electronic Data Deliverable (EDD), which will be saved to a shared server. The Program Lead is responsible for a complete quality review of the lab report and EDD following the guidelines set forth in “Maine DEP Basic Data Review Checklist, Revision No. 00” (Appendix D). Any data quality concerns will be communicated to and resolved with the laboratory project manager. The final EDD will be uploaded to EGAD and would then be available for reporting and formal use and distribution.

RANSOM
Consulting, LLC

Consulting
Engineers
and Scientists

MARINE MONITORING FORM

Site Information

Site Name:____ Project No.:_______________

Site GPS Coordinates:_______________________________ Date:____________________

Personnel:____

Weather

Wind Direction (360° = N)_____________________________Cloud Cover (±25%): ________

Beaufort Scale (see back):____________________________ Water Depth (m):__________

Sonde e-Logging for Discrete Depth Measurements

Instrument Manufacturer and Model:____

Instrument I.D. Number:_________________Operator:_____________________________________

Date Calibrated: ________________________ Barometric Pressure (mm Hg):_________________

Notes: ____

____

Sonde Manual Logging (*use only if not e-logging*)

Sample Depth (m)	Temperature (°C)	Salinity (ppt)	pH	DO (mg/L)	DO (% sat.)	Turbidity (FNU)

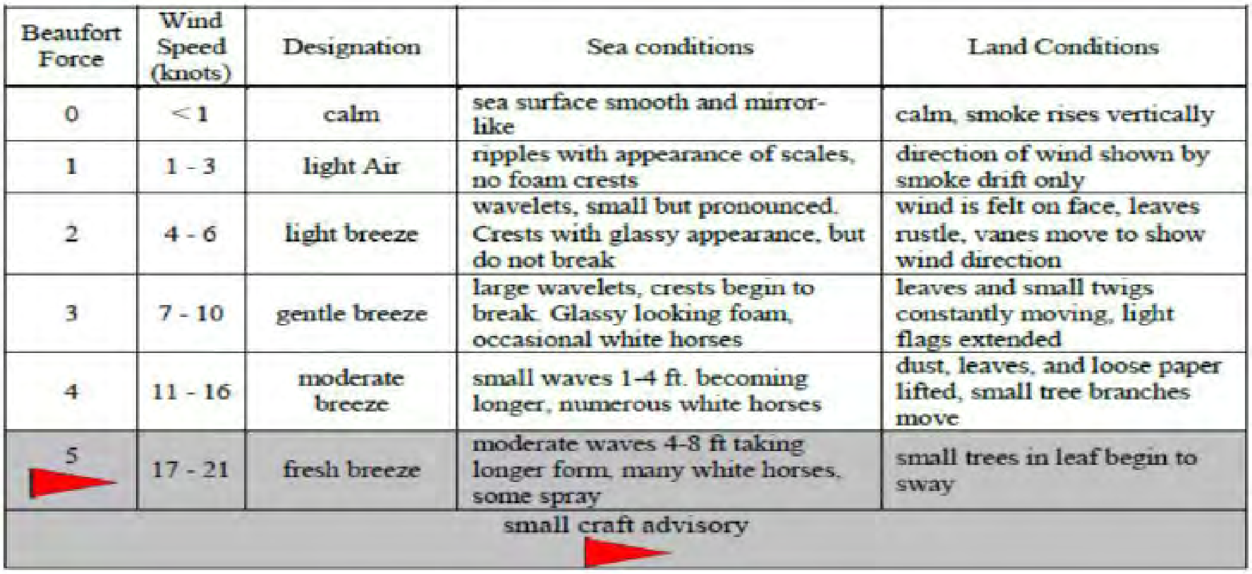
Secchi Disk (20 cm, black & white)

Operator: ____________________________

Replicate	Disappears (to 0.01 m)	Reappears (to 0.01 m)
#1		
#2		
#3		

Grab Sample Notes:

Beaufort Scale:



Beaufort Force	Wind Speed (knots)	Designation	Sea conditions	Land Conditions
0	< 1	calm	sea surface smooth and mirror-like	calm, smoke rises vertically
1	1 - 3	light Air	ripples with appearance of scales, no foam crests	direction of wind shown by smoke drift only
2	4 - 6	light breeze	wavelets, small but pronounced. Crests with glassy appearance, but do not break	wind is felt on face, leaves rustle, vanes move to show wind direction
3	7 - 10	gentle breeze	large wavelets, crests begin to break. Glassy looking foam, occasional white horses	leaves and small twigs constantly moving, light flags extended
4	11 - 16	moderate breeze	small waves 1-4 ft. becoming longer, numerous white horses	dust, leaves, and loose paper lifted, small tree branches move
5	17 - 21	fresh breeze	moderate waves 4-8 ft taking longer form, many white horses, some spray	small trees in leaf begin to sway
small craft advisory				

APPENDIX B

Laboratory Analytical Standard Operating Procedures

Sampling and Analysis Plan
Water Quality Monitoring, Frenchman Bay and Blue Hill Bay

Sample Receipt and Log-In

1. Scope and Application

This procedure is used by the Sample Login and Project Management staff to receive samples into the laboratory and enter information into the Laboratory Information Management System (LIMS). Accurate entry of this information is critical to ensure samples are analyzed properly and to produce a comprehensive final report for each client.

2. Summary of Process

Samples are received into the laboratory by the Sample Login staff. Each job, each sample within each job, and each sample container is assigned a unique ID number. The samples are checked to verify that they are preserved in a satisfactory manner, within satisfactory containers for each analysis and within holding times. The sample information is entered into the Laboratory Information Management System (LIMS) and thus communicated to the entire laboratory. The sample containers are placed in the appropriate refrigerators or freezer.

3. Safety

The toxicity or carcinogenicity of each reagent used in this method is not fully established; however, each sample should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan (Document ID 2121 for Westborough, ID 2124 for Mansfield).

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices, otherwise known as universal precautions, for handling known disease causative agents.

4. Equipment and Supplies

4.1 Preservatives: All preservatives listed below are prepared by the laboratory staff with reagent-grade acid or base and de-ionized water. All preservative preparation information is contained in logbooks located within the laboratory area.

The date, time, preservative name and ID#, department that prepared the preservative and the initials of the Organic Prep or Metals Prep staff member filling the bottle are recorded in the Login Department Preservative Tracking Log.

4.1.1 Hydrochloric Acid, HCl, 1:1

4.1.2 Sulfuric Acid, H_2SO_4, 1:1

4.1.3 Nitric Acid, HNO_3, 1:1

4.1.4 Sodium Hydroxide (NaOH) (6N or 10N)

4.1.5 Sodium Bisulfate (NaHSO4)

4.2 Thermometers:

4.2.1 Omega, infrared – Westborough and Mansfield

4.2.2 Mercury - Mansfield

4.3 Computer with LIMS access

4.4 Writing implements: black or blue pen, colored permanent markers.

4.5 pH Paper: Wide-range (0-14), multi-banded

4.6 Pasteur Pipettes

4.7 Label Writer

4.8 Stickers: Orange (HEXCR ONLY), Blue (Blank and VOA), Yellow (SUB-UPS, SUB-COURIER), Green (Blank and MANSF.), Pink (QC), Headspace (HS)

4.9 KI Paper Strips: 0.05mg/L residual chlorine sensitivity

4.10 Sodium Thiosulfate Crystals ($Na_2S_2O_3$): J.T. Baker; 5-Hydrate crystal

4.11 Sodium Sulfite Crystals (Na_2SO_3)

5. Procedure

5.1 Sample Receipt

Samples may be received at the laboratory via the Client, Alpha Courier, or US Mail / FedEx / UPS.

5.1.1 Retain or copy any custody seals or bills of lading that may have been affixed to the cooler(s). Condition of custody seals will be noted on the SDG. Custody seals are considered ‘present’ for Air samples if the crates have cable ties intact. If no cable ties are present, there is no custody seal present. This information is noted on the LIMS SDG form. Custody Seals are considered 'present' for non-air samples if the cooler is sealed with a signed and dated seal of any kind that must be removed prior to opening the cooler.

5.1.2 Determine if the samples were kept cool with either ice cubes ice packs, or dry ice. Note this information on the back of the Chain of Custody (COC). If the samples did not arrive cooled, then note this on the back of the COC. This information will later be used in the LIMS Sample Delivery Group Form (SDG, refer to section 5.10).

5.1.3 If there is a temperature blank: Using the Omega infrared digital thermometer, check the temperature of the Temperature Blank by aiming it 1-2 inches from the temperature blank container and waiting for the temperature reading to come to equilibrium. Record the temperature on the back of the Chain of Custody (COC) along with the last four digits of the thermometer’s serial number. The temperature should be within 4 ± 2°C. If the temperature reads >6°C, indicate whether the samples were delivered "Direct From Site", defined as having been delivered to the lab directly, after the samples were collected, without being held at another location in between. If not, the client must be notified to give authorization to proceed prior to analysis. If the temperature is <2°C, then it must be noted on the back of the COC whether the samples were received frozen. If frozen, then the Project Manager must be contacted prior to analysis. They will confer with the client if needed and determine whether the analysis should be performed.

5.1.4 If there is not a Temperature Blank: Use the Omega Infrared Thermometer to take the temperature of a sample within the cooler, or the cooler itself, by aiming the thermometer into the cooler while avoiding the ice or other cooling materials present. Continue to follow instructions in 5.1.3

5.1.5 If the samples did not arrive cooled or if the temperature is >6C but not delivered directly from the site, determine if the client needs either MCP or CT-RCP criteria. If so then the client must be informed at the time of sample drop off that because the samples were not cooled they will not receive either “Presumptive Certainty” (MCP) or “Reasonable Confidence” (CTRCP) and so we will need their approval to continue with the analysis at the time of sample drop off.

Please Note: Samples received in the proper temperature range but not on ice will not receive Presumptive Certainty or Reasonable Confidence.

5.1.6 If the client is a new client, payment is due upon receipt. The amount due is determined by a Project Manager or by the Login Supervisor. Alpha accepts personal checks, credit card (MasterCard, Visa or American Express) or cash.

5.1.6.1 **Personal Check:** Record the check number and the amount in the comments section of the COC. Give the original check and a copy of the COC to the Accounts Receivable department.

5.1.6.2 **Credit Card:** Record the credit card received (MasterCard, Visa, or American Express) in the comments section of the COC. Have the client complete the Credit Card Form. Verify card information. Give the completed Credit Card Form and a copy of the COC to the Accounts Receivable department.

5.1.6.3 **Cash:** Record “Cash Received” and the amount in the comments section of the COC. Give the cash and a copy of the COC to the Accounts Receivable department.

5.1.7 If a client is dropping samples off at the lab, inquire about any discrepancies prior to the client leaving. The client must sign the COC, relinquishing the samples to the laboratory. The Alpha Login employee must then sign the COC, and record the date and time the samples were received. Give a copy of the completed COC to the client.

5.1.8 Any coolers of unused sample containers returned by the client must be visually inspected to ensure that no actual samples were delivered in them. If any samples are found the client will need to be notified and asked for additional instruction. The cooler and all unused containers are to be stored outside the Hazardous Waste disposal room.

5.2 Sample and Paperwork Inspection

5.2.1 Environmental Samples

5.2.1.1 Upon receipt of a chain of custody, the chain and associated paperwork are emailed to the login department who will assign a job number to the samples (see section 5.8.1). The data entry group will record the information from the chain of custody into the LIMS excepting product codes and sample comments. Product Codes and Sample Comments will be applied by the project management group. As the information is entered into the LIMS it will be visible to sample receipt staff via the pivot tables used to forecast and prioritize work.

5.2.1.2 Verify that the courier has signed “Receiving and Relinquishing” samples.

5.2.1.3 If the samples have rush turn around times or short hold parameters, they are to be processed immediately. If receiving samples directly from a client at the lab, inspect the chain for analysis with approaching holding times and ensure these are processed immediately.

5.2.1.4 Locate the Alpha Job Number in LIMS by one of two methods.

5.2.1.4.1 From the Specify Login window (see section 5.8.1.3), select “Search”. In the table that opens a list of all jobs beginning with P will be visible. Enter search terms into the top row and click refresh to refine search results. Double click on a line to return to the Specify Login window with the job # copied into the proper field and select OK to enter the job.

5.2.1.4.2 Refer to the Google Doc titled “Daily Courier List 20XX”. On the tab for the day the samples were received search by client name, courier code and region for the pickup that corresponds to your chain of custody. Copy the Job # from the same titled field and enter into the Specify Login window.

5.2.1.5 Upon entering the job, check the Sample Comment table for a comment that indicates the job was split. If the job was split, make a copy of the chain of custody to use for each split job. See section 5.3 for more information about splitting jobs. Also check the Call Tracker form for information being communicated to the Sample Receiver.

5.2.1.6 Remove the samples from the coolers and verify that the number of samples, containers, labeling of containers and their preservatives agree with the Chain of Custody (COC). Any discrepancies must be noted in the SDG form. As samples are removed from coolers login staff will assign each cooler a letter designation (A,B,C,etc...) which will be used to track which bottles were received in each specific cooler. Mark the caps of each container to identify which cooler each container was received in. If coolers were labeled by the courier picking them up, the cooler marked as 1 of X should be designated as A, 2 of X as B and so on.

5.2.1.6.1 **DOD Samples**: If samples received are from a DOD project, then coolers are to be unpacked in Mansfield, inside the hood in login area. If larger cooler or air crate is received, then the larger hood in the Tissue Prep lab will be used to unpack and process samples for all DOD projects.

5.2.1.6.2 **Method 537 (PFA)** If aqueous samples received are for 537(PFA) analysis please follow these protocols:

- Gloves must be worn at all times when handling sample containers.
- Do not open or check the pH of sample containers.

- Do not write on caps or containers with Sharpies, Permanent Markers, or Paint Markers. Ball point pens are acceptable.
- Do not apply any stickers/labels to containers or caps other than the barcoded LIMS labels.
- Always put containers back into Ziploc bags they were received in, for transfer to fridge or to Mansfield facility.

5.2.1.7 If any samples are either missing, leaking, broken, inappropriately preserved, in inappropriate containers, or are outside of their holding time, the client must be contacted. This information is recorded in the LIMS SDG Form. Also, if there are any questions concerning analysis to be performed, inform the Login supervisor or Project Management prior to inputting the information into the LIMS. If a requested analysis cannot be run due to a container discrepancy, the product code must be removed from the sample.

5.2.1.8 If bottles are submitted for analysis not requested on the chain of custody:

5.2.1.8.1 For analyses with remaining Holding Time greater than 3 days (See Table I): log on Hold until the client decides whether the samples are to be analyzed.

5.2.1.8.2 For analysis with remaining holding times less than 3 days the applicable product code is to be logged in and the client contacted to confirm whether they would like the results reported.

5.2.1.8.3 If the containers are vials preserved with water, the product code HOLD-8260HLW is to be added to the sample and the containers added to this product.

5.2.1.8.4 Non-water preserved vials should be applied to the ARCHIVE product.

5.2.1.9 If the client must be contacted, the Call Tracker Form located in the LIMS is used to document the details of this communication.

5.2.1.10 If a client requests that analysis not be conducted until further notice, these samples are put on “HOLD”, and this is noted on the Chain of Custody. Containers are to be assigned to the appropriate HOLD product codes.

5.2.2 Chain of Custody

5.2.2.1 Chain of Custody forms are to be numbered with the Alpha Job number associated with it in LIMS, any fields available for numbering individual samples, or for indicating sample receipt information are to be completed.

5.2.2.1.1 Write the new Alpha job number on the Alpha COC in the “Alpha Job #” box. If the Client’s COC is not an Alpha COC, write the “Alpha job #” on the top left or right corner of the COC.

5.2.2.1.2 Each unique sample ID will also get its own “Alpha Lab #”. This number is the job number plus .1, .2, .3, etc. numbered sequentially for each unique sample ID defined by the client on the COC. This should be written to the right of each sample ID on the COC in the space provided if there is one present.

5.2.2.2 In Westborough all records are transferred to the front office daily to be filed away and stored in filing cabinets. This includes chain of custody forms and the bill of lading forms, purchase orders, custody seals or any other information submitted with the chain of custody and/or samples.

5.2.2.3 In Mansfield all records are filed away and stored in filing cabinets in the login department. This includes all paperwork listed in Section 5.2.2.2.

5.2.2.4 Save the chain of custody to LIMS (see section 5.17)

5.2.3 Project Communication Form (PCF)

The Project Communication Form (PCF) contains information regarding the client, project number, project location, quotes, specific comments or product codes needed for a job. For each job the Login Notes section for any PCF attached to the job must be reviewed.

5.2.3.1 Applicable PCFs must be attached in the LIMS by the project manager logging the job in. This is done by accessing the Job Definition/Login Info section of the LIMS. Double-click on the “Project Information” line to view a list of PCFs. A list of possible PCFs, or an error message will appear (“Query caused no records to be retrieved”). If an error message appears, there are no PCFs to choose from.

If there are PCFs to choose from and one is relevant, double-click on the PCF number to attach the PCF to the job.

Next, double-click on the “Scanned Information” line. If Scanned Information is present and the title matches the job being processed or if it is referenced by the PCF, attach it to the current job by double-clicking on the Scanned Information number.

Review the PCF and Scanned Info loaded and follow their instructions when processing or logging the samples.

5.2.4 Rush Jobs

5.2.4.1 If the client requests the analysis to be completed within a shorter Turn-Around Time (TAT) than what is standard for their account, change the TAT field and Due Date in LIMS to reflect the due date requested by the client. If the client requests a rush but leaves the due date blank on the chain, then default to a 48 hour TAT and surcharge.

5.2.4.2 Containers for the Rush job must be provided immediately to the appropriate departments either by placing on the Rush/Short Hold cart in Login, or by scanning the samples to the appropriate departments and hand delivering them. If no staff are available to analyze the samples they should be placed in their appropriate custody locations. A camera linked to staff monitoring it in Wet Chemistry, Extractions and Volatiles is used to inform labs of available work.

5.3 Splitting a Job

A single job sometimes needs to be split into more than one job for reasons dictated by the client or project manager. A copy of the original Chain of Custody must be made for each additional job that must be created. Add a comment in the LIMS regarding the job number(s) the job was split with.

5.4 Subcontracted Samples

5.4.1 Chain of Custody

An additional Chain of Custody must be completed and be sent to the laboratory that is to receive the samples for analysis we subcontract to them. This chain is generated as a PDF through LIMS by selecting “Sub COC” at the top of the Sample Definition window. It is to be printed and placed on the appropriately labeled shelves in the Walk-In Refrigerator along with the bottles being subcontracted. Containers should be scanned to the location they are placed in the walk-in. When they are taken to be delivered by an Alpha Courier or shipped by login staff they will be scanned to the LIMS location “TRANSFER-SUB”.

For the Mansfield facility, the samples are packed into a cooler and prepared for either shipment or courier drop-off at the appropriate sub-contract laboratory.

5.4.2 Courier Request Form & Shipping via UPS

A Courier Request Form must be completed for all samples being delivered to a subcontractor by an Alpha Courier. This form needs to include all pertinent information, e.g. where the samples are being subcontracted to, the sample number, and the delivery date. This task is completed utilizing the Courier Request Program within the LIMS System. By default Alpha will courier samples to subcontractors located in Massachusetts, Connecticut, Rhode Island, New Hampshire, Vermont and Maine or when directed to courier by the Project Manager or logistics department. The logistics department will then handle the delivery of the samples to the subcontractor.

If shipping samples via UPS the Courier Request Form is not completed and a request for a pickup is made via UPS’ website. The shelving in the Walk-In refrigerator is checked daily for work to be shipped and pivot table is used to view and check for samples to be shipped by the Log-In department. This board is located in the Log-In department and is updated both by the persons processing samples to be shipped and by those shipping the samples.

5.5 Westborough/Mansfield Sample Transfer Procedure

When one facility logs in a job, and it is deemed that some of the analysis must be performed at the alternate facility, the following procedure is initiated.

1. Analysis must be logged using the appropriate product codes of the lab performing the tests. When choosing the test codes, the facility performing the analysis, Westborough or Mansfield, must be chosen in the "TESTING SITE" field located in the Product Code section of the Login Screen, after the Container Key. All Mansfield product codes will start with "A2" with the exception of Metals analysis and Dissolved Gasses. If the testing site of the analysis logged on a container is WESTBORO, the letter W will print on the login generated label, if the testing site is MANSFIELD an M will be printed in the same location. If analysis for both testing sites are logged on the container, both letters will appear.
2. The procedure outlined in the Sample Custody and Tracking SOP (Qualtrax ID 1560) in section 5.7 is to be followed.

5.6 pH Determination

Determine the pH of all water and liquid samples as indicated below. The pH must be verified for each container to ensure proper preservation prior to analysis.

NOTE: Do NOT take the pH of any VOA vials, TOC or DOC containers, or samples for the analysis of sulfite (SO3), acidity, alkalinity, carbon dioxide (CO2), dissolved oxygen (DO), Oil & Grease, TPH-1664, Perchlorate, Chlorophyll, 537 (PFAA's) or any bacteria samples.

5.6.1 The pH must be taken by using a glass Pasteur pipette dipped into the sample container. Transfer a drop of sample onto wide-range, multi-banded pH paper. Refer to Table II for the proper pH of preserved samples. Under no circumstances should the pH paper be dipped directly into a sample container.

5.6.2 Record the pH in the SDG form.

5.6.2.1 If the samples are from a DOD project, then the lot# of the pH strips used must be recorded in the SDG form.

5.6.3 If a sample is preserved with a preservative different than what is indicated in Table II the lab must be notified to ascertain if the analysis can still be run.

5.6.3.1 If the analysis cannot be run this must be noted in the LIMS SDG and the client must be notified

5.6.3.2 If the analysis can still be performed, then this must be noted in the SDG, a comment must be added for the lab as to what the container is preserved with, and the client must give authorization if we are to proceed with analysis outside the method. In many cases this may cause the client to lose "Presumptive Certainty" for MCP, or "Reasonable Confidence Protocols" for CTRCP requirements.

5.6.4 If the pH of preserved samples is not as indicated in Table II and the sample is either unpreserved or contains the preservative indicated in Table II: Record the original pH in the SDG and adjust the pH using the appropriate preservative as indicated for each analysis in Table II. Check the pH after each addition of preservative. The addition of preservatives must not exceed 1% of the total volume of sample received. See Bottle Preservation Guide (Qualtrax No. 17996) for volume of preservative to add to unpreserved sample containers. Make a record on the LIMS SDG Form to indicate that the samples were not properly preserved. For all metals containers that required a pH adjustment, write the date/time acid was added on the cap of the bottle along with the initials of the person who added the acid to the sample, also add a comment to the sample with this information; this is to ensure that the lab will meet the requirement to wait 24 hours (16 hours for DW samples) prior to beginning preparation for Metals analysis.

5.6.5 Note the addition of preservative and result in the SDG. Bottles containing sample that are not in the proper pH range are to be marked with a blue sticker on the cap to alert the chemists to this issue. If the pH does adjust into the proper range, affix a green sticker to the cap

5.6.6 **EPA 522:** Samples must be checked for residual chlorine.

5.6.6.1 Samples must be checked for residual chlorine prior to any pH adjustments or extraction. Invert the sample several times to ensure that sample is well mixed. Remove a 20mL aliquot of sample and dip one KI test strip into the aliquot for 30 seconds with a gentle constant back and forth motion. (Refer to the chart on the KI strip container.) If chlorine is detected at a level greater than or equal to 0.1mg/L. Document the performance of this check in the LIMS SDG Form by checking the box labeled "Chlorine Check". If Chlorine is detected at a level greater than or equal to 0.1mg/L, this should be noted in the Container level comments field in the SDG form.

5.6.6.2 **EPA 522** Samples should have a pH of <4. If the sample is not within this range, it should be noted in the LIMS SDG that samples were received above the appropriate pH. A Blue circle sticker should be added to each container with a high pH. No additional preservation is needed.

5.6.6.3 **EPA 608** Samples must have a pH within the range of 5.0 – 9.0 pH units. If the sample is not within this range, it is adjusted using either NaOH to increase the pH or with H_2SO_4 to decrease the pH. Make a record in the LIMS SDG to indicate the volume of acid or base that was added to the sample. Write the preservative added on the cap of the container with the date/time added.

If the pH will not adjust into the proper range, note this in the LIMS SDG. The bottles are marked with a blue sticker to alert the chemists to this issue.

5.7 Sample Storage

For both the Westborough and Mansfield facilities refer to SOP 1560 for instructions regarding the storage and tracking of samples after the login process outlined in this SOP have been followed.

5.8 Data Entry into the Laboratory Information Management System(LIMS)

5.8.1 Access the LIMS

5.8.1.1 Access the LIMS by clicking on the WEBLIMS / Seedpak icon located on the Windows desktop screen.

5.8.1.2 A password screen will open. Each person is given a name and password to access the LIMS. Type in your name, password and directory “Bowzer”. Select < Enter >.

5.8.1.3 The main screen will open. From the Main Menu, select < Sample Management>, then select < Login > (or the keys: “Alt + S”, then “L”). This will open the < Specify Login > screen.

5.8.1.4 Type in the Alpha Job # for which data is to be entered. Select < Enter >. This will open the Sample Definition Screen. To generate a new Alpha Job # select New.

5.8.2 Sample Definition

Referring to the Client’s original COC, input the following information for each sample. (If an Alpha sample number is inadvertently deleted during this process, refer to Section 5.8.6.)

5.8.2.1 **Client ID:** This is the Sample ID from the COC.

5.8.2.2 **Collect Date:** This is the Sampling date and time. These are entered in the following format: day-month-year, and military time.

5.8.2.3 **Project:** This is the Client’s Project #, if provided.

5.8.2.3.1 If the project number is new for the client, and therefore has not been previously used in the LIMS, an error message will appear. Prior to proceeding, the new project number must be entered into the LIMS as follows (New project numbers should be created by project managers or login data entry staff only):

5.8.2.3.1.1 At the top of the screen, choose <File> and then <Main Menu>.

5.8.2.3.1.2 Select <Maintenance> and then select <Accounts>. The Client Information screen will open. Click on the arrow next to the box labeled <Account Definition>.

5.8.2.3.1.3 Select <Project Definition> and then enter the Account Number / Account Code, and click on <OK>.

5.8.2.3.1.4 To clear the field, press the < F6 > button.

5.8.2.3.1.5 Enter the new project number, if provided on COC, the Project Name (Site), and Location as it appears on the COC. Leave the other spots blank.

Double-check that the correct information has been entered, and then click on < Save > (or < Alt + S >).

5.8.2.4 **Account:** This is an abbreviated account name given for the client name in the LIMS. A list of account names can be accessed by pressing the “F9” key on the keyboard.

If the job is for a new client and the account name cannot be found in this list, the Login will log the job under the account “NONE”.

Note: new accounts should be created by project managers only.

5.8.2.5 **Account Name:** This is the full client name. This field will fill in automatically after the “Account” is entered.

5.8.2.6 **TAT:** This is the standard turn-around time for the client. (Either 1 week, 2 weeks or the RUSH TAT requested and approved). Turn-around time is entered in # of business days.

5.8.2.7 **Due Date:** This field will automatically fill in when the Received Date and Time are entered.

5.8.2.8 **Receive Date & Time:** This is the date and time samples were first received by an agent of Alpha Analytical. This must match the date and time noted on the signed COC. The following format must be utilized: day-month-year and military time (i.e. 31-AUG-07 14:20).

5.8.2.9 **Lab Due Date**: This is an internal laboratory due date which prints on worklists and batch sheets. This is entered on an as needed basis and must be entered in the following format: day-month-year.

5.8.2.10 **LVL5 Due Date**: This is the due date of the data package that is needed for any project requiring additional reporting packages. Packages requiring due dates are ASP-A, ASP-B, DPKG-RED, DPKG-FULL and NJ-RED. This is the date that will automatically print as a comment for the lab and is to be entered in the following format: day-month-year.

5.8.2.11 **Location:** The project location such as the town, state, etc. This should be taken directly from the client’s Chain of Custody.

NOTE: If information has been entered in the Project Definition, it will automatically load.

5.8.2.12 **Project Name (Site):** The project name, or specified site. This should be taken directly from the Chain of Custody.

NOTE: If information has been entered in the Project Definition, it will automatically load.

5.8.2.13 **Discount:** This is the discount given to the client, if applicable. (The discount is already entered into the LIMS and the field will fill in automatically when the "Account" is entered) An automatic discount may be overwritten in this field if a special discount is given to a particular job (Refer to the PCF).

5.8.2.14 **Priority:** This refers to TAT: either "standard", "rush", or "no charge". A list of priority codes can be accessed by pressing the "F9" key on the keyboard.

5.8.2.14.1

Priority	TAT Description
NC	No charge for analysis
S0	Standard turnaround time rates/fees
SD	Same day rush – 200% surcharge rate
1A	1 day rush for 1 week clients – 100% surcharge rate
1B	2 day rush for 1 week clients – 75% surcharge rate
1C	3 day rush for 1 week clients – 50% surcharge rate
1D	4 day rush for 1 week clients – 25% surcharge rate
2A	1 day rush for 2 week clients – 100% surcharge rate
2B	2 day rush for 2 week clients – 85% surcharge rate
2C	3 day rush for 2 week clients – 75% surcharge rate
2D	4 day rush for 2 week clients – 65% surcharge rate
2E	5 day rush for 2 week clients – 50% surcharge rate
2F	6-7 day rush for 2 week clients – 25% surcharge rate
2G	8-9 day rush for 2 week clients – 10% surcharge rate

5.8.2.15 **Factor:** This is the charge or surcharge relating to the priority code.

5.8.2.16 **Comments:** This field is only used on Dummy Jobs to reference the job # requested by the lab.

5.8.2.17 **Matrix Description:** For samples that are sediments, it is necessary to indicate this in this field. This is important for any samples associated with the Mansfield facility. For any solid (matrix 4) samples the specific matrix is noted here.

5.8.2.18 **Quote Reference:** This field is used only if a quote has been generated in the LIMS. The information is contained on a Project Communication Form, may be indicated on the COC, or may be obtained by pressing "F9" on the keyboard which brings up a listing of Quotes associated with a particular Account. Clicking on a Quote # in the list and pressing "OK" will attach it to the job.

5.8.3 Analyte Definition

Move to the middle section of the < Sample Definition > screen by pressing the < ctrl + page down > keys on the keyboard.

Referring to the Client's original COC, enter the requested analyses using the information from the Analysis Request section, as follows:

5.8.3.1 **Matrix:** This is the sample type (water, soil, solid, etc.). There are specific matrix codes used for this field. A list of matrix codes can be accessed by pressing the “F9” key on the keyboard.

5.8.3.1.1

Matrix	Type of Sample
1	Water
2	Liquid/Solvent
3	Soil
4	Solid
5	Sludge
6	Oil / Product
7	Wipe
8	Miscellaneous
9	Drinking Water
10	Air
11	Soil Vapor
12	Air Cartridge
13	Tissue

5.8.3.2 **Product Code:** This is the analyte requested. The correct Product Code for any given request dependent on the matrix of the sample. Different matrices have different methodologies based on the established State and Federal regulatory requirements. The proper code to log by default, absent any project specific instructions, can be determined by referencing the document LOGIN GUIDE 20XX on the Google Drive.

If a client specifies a particular method it is to be logged in as such, unless that is not an option in the LIMS. If this is the case, the Login Supervisor should be contacted for further instructions.

A full list of analytes for each matrix can be accessed by pressing the “F9” key on the keyboard.

HOLD Samples: There is a product “HOLD” that is entered into the LIMS for those samples that the client has requested not to be analyzed until further notice.

> **NOTE:** This is only to be entered if the entire sample is to be placed on Hold. If specific analysis are contingent on other analysis, or certain analysis is on hold specific HOLD codes exist for each product and contingency.

CANCELLED Samples: There is a product called “CANCELLED” that is entered into the LIMS for those samples that the client has requested to be cancelled.

> **NOTE:** This is only to be entered if the entire job is cancelled.

SUBCONTRACT Samples: For each analysis being subcontracted there is a product beginning with “SUB-” that is entered into the LIMS for those samples that will be sent to another Laboratory for analysis. If a product does not exist contact the Log In Supervisor or Alpha Project Manager to have one created.

MCP / CT-RCP / NJ DEP Samples: There is a product “MCP” that is entered into the LIMS on the first sample of any job that require the MCP Data Enhancement Package. The product “CTRCP” should be entered on the first sample of any job requiring CT Reasonable Confidence Protocol. The product "NJDEP" should be entered on the first sample of any job from the state of New Jersey.

Data Deliverable Packages: Various Package products exist for projects that request additional data deliverables. Some examples of these are: DPKG-RAW, DPKG-FULL, NJ-RED, ASP-A, ASP-B. These products will need to be added to only the first sample of any job requesting it. When adding this code the Level5 Due Date section will automatically populate a date. This date will have to be copied down to all samples.

5.8.3.3 **Type:** This field will be entered automatically based upon what is entered for the Product Code. It refers to whether the analyte is a single (s), parent (p), or child (c) product.

A single product is a product that is alone.

A parent product is a product that contains two or more analytes (these analytes are the children). The majority of Types are single (s), but there is a list of parent products that may be accessed by pressing the “F9” key on the keyboard.

When a parent product is entered, the children will be entered automatically. Verify that the “show children” box is checked.

5.8.3.4 **Surcharge:** This field will be entered automatically. The surcharge is automatically populated based on the Priority Code.

5.8.3.5 **Discount:** This field will be entered automatically.

5.8.3.6 **Price:** This field will be entered automatically. It is the price charged to the Client for the analyte.

5.8.3.7 **Cost:** This field will be entered automatically. It is the cost to Alpha to run the analyte.

5.8.3.8 **Price List:** This field will be entered automatically if there is QUOTE or ACCT level pricing. Otherwise the field will default to STD.

5.8.3.9 **Default Type:** This field is the type of container the analyte is normally from from. A full list of container types can be accessed by pressing the “F9” key on the keyboard.

5.8.4 Container Key and Type

5.8.4.1 Double click one of the Container Key fields next to the default type field to open the “Maintain Containers” table. In this table each analytical product is associated with a container key, container types can be edited for each key, and associations, types and keys can be copied between samples.

5.8.4.2 **“Split”/”Filtrate”/”Extract” Containers:** Containers not originally provided by the client are designated as such by adding a prefix to their container types. Raw samples split into separate containers in house whether for compositing, replacing broken containers or creating additional aliquots must have the prefix

"S" in front of the container type. Filtrates created in the lab must have their post-filter volume identified with an "F" prefix. Extracts, if stored in general custody locations must be identified with an "E" prefix.

5.8.4.3 **Container Key:** These are individual identifiers for each container included on a sample. Letters and letter-number combinations can be used. Each container key represents a single unique container and is used to track that container through the laboratory. Place all container keys for containers to be used for any given product in the container key field to its right. The container type will default to the value entered in the "Actual Container Type" field.

5.8.5 Job Definition / Log-In Info

Select the < Login Info > button on the screen main Sample Definition screen to enter the following information:

5.8.5.1 **Login By:** This field is for the initials of the person who is entering the sample information into the LIMS.

5.8.5.2 **State of Origin:** This is the state where the sample was collected.

5.8.5.3 **Scanned Information:** This is the field where Scanned Information is attached, if available.

5.8.5.4 **Project Information:** This is the field where the Project Communication Form (PCF) is attached, if available.

5.8.5.5 **Dispose Samples:** "Y" will automatically populate indicating that samples are to be disposed of. If samples are to be returned to the Client or to be held for an extended period of time, change this to "N". If answered with an N the method of storage (Refrigerated, Frozen or Ambient) must be specified and the date when the samples are able to be disposed must be entered in the comment field adjacent to the question.

5.8.6 Reseeding a Job

Reseeding a job is only necessary if a sample number is inadvertently deleted while entering the job information. If the job is not reseeded, the job will have skipped sample numbers.

5.8.6.1 Save the job that is being worked on by pressing the <Enter> button.

5.8.6.2 At the top of the screen, select <File> and then <Main Menu>.

5.8.6.3 Select <DBA> and then click on Seeds/Config/Printers. Click on the arrow next to "Seeds", and then click on <Login Seeds>.

5.8.6.4 Place the cursor in the box called "Seed Identifier" and press the <F7> key.

5.8.6.5 Type in the full Alpha job number that is being reseeded, and press the <F8> key. This will show the number of seeds/samples.

5.8.6.6 Change the number to either the number before the skipped number, or to zero if the entire job is to be redone.

5.8.6.7 Click on the <Save> button and exit the screen.

5.9 Saving Data

5.9.1 **Field Filtered Analysis:** Before exiting the < Sample Definition > screen, save data for field filtered sample as follows: While on the < Client ID > field for a sample with field filtered volume check the <Fld Filt>.

5.9.2 Air volumes for Metals analysis in Air Media must be entered for each sample. Place curser on the first metals product code and then select the DATA button at the top of the login screen. Enter the volume provided by the client on their COC for that sample, then hit <Save>. Repeat this for each sample.

5.10 Electronic Sample Delivery Group (SDG) Form

This info must be completed and saved for all jobs logged. Select the <S17> Button at the top of the Login Screen. The SDG is comprised of 5 pages.

5.10.1 Delivery

5.10.1.1 Delivered by: Drop down menu to select Alpha Courier, Client, FedEx, UPS or another form of delivery can be hand typed.

5.10.1.2 Tracking # can be entered if samples came from a shipping service like FedEx or UPS.

5.10.1.3 Chain of Custody: Indicate if a Chain of Custody was provided.

5.10.1.4 Coolers

5.10.1.4.1 Loginnum: Will auto-fill when clicked on.

5.10.1.4.2 Coolers: Enter Cooler “A”, “B”, etc.

5.10.1.4.3 Receive Date : will default to the current date once the cursor is placed in this field, change as needed.

5.10.1.4.4 Custody Seal: This is a drop-down menu to choose between Absent, Present/Intact, or Present/Not Intact.

5.10.1.4.5 Custody Seal #: if a number was on the custody seal enter here. If not enter “NA”

5.10.1.4.6 Cooler Preservation: Boxes to be checked for all that apply.

5.10.1.4.7 Temp Blank/IR Gun: Temperatures to be filled in if one or both methods of obtaining temperatures are used. If both methods are used, then the acceptable temperature must be double clicked and highlight in blue.

5.10.1.4.8 Frozen: Check if samples are received frozen.

5.10.1.4.9 Site Direct: Check if samples are received outside of the acceptable temperature range but were delivered directly from sampling site.

5.10.1.4.10 Reported Temp: Will auto-fill

5.10.1.4.11 “IR Gun Serial Number” use F9 to view a list of all serial numbers of IR Thermometers in the facility chosen as the OWNER for the job. To view numbers from the alternate facility change the job OWNER, enter the number and revert back to the correct owner.

5.10.1.5 VOA/VPH

5.10.1.5.1 If vials submitted for low level 8260 analysis containing Reagent Water as the preservative were frozen by the client answer the question on this page “Reagent Water Vials Frozen by Client?” as YES. This will allow the time of freezing to be entered in the CONTAINER table described in section 5.10.1.6.7

5.10.1.5.2 If vials submitted for low level 8260 analysis containing Reagent Water as the preservative were frozen by login staff prior to being assigned a container and labeled due to an approaching holding time answer the question on this page “Reagent Water Vials Frozen Prior to Login” as YES. This will allow the time and LIMS location of freezing to be entered in the CONTAINER table described in section 5.10.1.6.7.

5.10.1.6 Condition

5.10.1.6.1 First select the button at the bottom of the page labeled “Recheck Containers and Hold Times”

5.10.1.6.2 All samples on COC received? ; Extra Samples Received?: Upon changing the answer to either question a table will open where any samples that were received, but that were not listed as a sample on the chain can be selected. Any samples listed on the chain but not received are also indicated in the same table.

5.10.1.6.3 Are there any sample container discrepancies?: Any bottles that were provided for analysis not listed on the chain, any tests requested for which we did not receive any volume, any bottles received broken or damaged or sent in containers that are not appropriate for the requested analysis are indicated in the table that opens when changing the answer of this question to YES.

5.10.1.6.4 Are there any discrepancies between sample labels and COC?: if the Sample ID, or the Collection Date/Time on the chain disagrees with the information written on the bottles this information is to be documented in the table that opens when changing the answer of this question to YES.

5.10.1.6.5 Are samples in appropriate containers for the requested analysis?: If any containers were indicated as inappropriate in section 5.10.1.6.3 this question should be answered with YES.

5.10.1.6.6 Are samples properly preserved for requested analysis?: If any headspace was present in samples where headspace cannot be present, if any samples had incorrect preservatives or had to had their pH adjusted, if chlorine needed to be neutralized or methanol was not fully covering soil for any VOCs submitted preserved with Methanol than this question is to be answered as NO.

5.10.1.6.7 Select the box labeled VIEW CONTAINERS to the right of the question regarding proper preservation of samples to display the CONTAINER table.

5.10.1.6.8 Enter the cooler key each container was received in, pH’s of the containers recorded, any inappropriate headspace or methanol preservation

5.10.1.6.9 If pH of the samples were adjusted by the Login Staff, this initial pH must be noted, then the final pH. If the incorrect preservative was used this should be indicated as well.

5.10.1.6.10 Containers requiring a chlorine check will have the checkbox in the column titled Chlorine Check highlighted yellow.

5.10.1.6.11 The comments field can be used for any issues for which there is no automated question or table. F9 can be used to view a list of pre-typed comments.

5.10.1.6.12 SAVE: Close out of all subtables and then the SDG. The Electronic SDG must now be saved to a PDF in the LIMS. Choose to generate the PDF when prompted.

5.11 Addresses

5.11.1 In this section, the billing address and who the report goes to may be changed if necessary by the project management staff. Sample Login staff should not alter the information in this table.

5.11.2 **Billing Address:** If the Billing Address needs to be changed, hit the “F9” key while the cursor is on the Billing line. Then double-click on the correct billing address, and click the “Save” button. If this is a third-party billing, hit the “F9” key while on the Billing line. In front of the “%” on the top of the screen, enter the LIMS account number which is to be billed and hit the “Enter” key. Now double-click on the correct billing address and click the “Save” button. If the correct address is not in the LIMS listing, do not make any changes and alert the Project Management department to make the appropriate corrections.

5.11.3 **Report to:** If the person listed to receive the final report needs to be changed, click on the “Report” line and hit the “F9” key. Double-click on the appropriate person and click the “Save” button.

5.11.4 If the correct person’s name is not in the LIMS listing then Login must create a new “Report To” address in the LIMS. To add a “Report To” address perform the following:

- Select <Maintenance>, then <Accounts>.
- The Client Information Screen will open. Click the arrow next to the box labeled Account Definition. Select <Address Information>.
- Enter the Account #/ Account Code and check <OK>.
- Use the down arrow to go down thru “Report To” addresses until you reach the last “Report To” address#.
- Hit <F6> key to clear the field
- Hit <F4> key to copy from the previous field.
- Change the Report__ (#) to the next consecutive # and then change all information as needed. Project Manager’s name, phone#, fax#, email address.

5.12 Comments

5.12.1 When a project requires a comment that will be seen by the Laboratory, the comment must be loaded in the Comment table of the Login screen rather than the comment field adjacent to the sample info. These comments may include special instructions for the Lab, cautionary notes about the matrix of a sample, compositing instructions, or special limits needed.

5.12.2 When entering a comment put the cursor on the CLIENT ID of the sample needing the comment and click on the COMMENTS button located at the top of the Login screen.

5.12.3 Hand type the comment in on the yellow highlighted line or choose from the “Canned” comments list.

5.12.3.1 To choose from the “Canned” comments, click on the Green down arrow located all the way to the right of the yellow highlighted line you are working on. Click on the comment that applies and click on OK at the bottom. This will populate the comment you choose in the yellow highlighted line.

5.12.4 After you are done entering the appropriate comment either by hand typing or canned comment, you must check off all of the Lab departments that will need to see this comment. For example: if the comment is about VOC 8260, the Volatiles button must be checked and Reporting. (Reporting must be included for all comments.) When this is complete, click on the SAVE button at the bottom.

5.12.5 Comments can be ADDED to all samples, or DELETED from all samples by clicking on the designated buttons located in the comments screen. Individual comments can be deleted by placing the cursor on the yellow highlighted comment and hitting Shift F6.

5.13 Printing the Bottle Labels

To print the bottle labels for a job entered, select <Report> and then select <Labels>. This will open the “print” screen. Press <Enter> and then <Enter> again. The individual bottle labels will be printed, as well as a label with the Client and Job number.

The bottle labels will contain the job number, sample number and the container letter. Also on the labels will be the analytes to be tested from that particular container, the container type and the sampling date and time.

5.14 Labeling the Sample Containers

Using the labels printed in Section 5.13, place the labels on the appropriate containers horizontally, without covering client info, and as close to the top of the container as possible. Any split, extract or filtrate labels are to be provided to the laboratory that will create these containers. All labeled containers must be peer reviewed by another Alpha employee prior to being released to the Laboratory.

5.15 Additional Container Flagging:

Soils from Foreign Country:
If the samples are soils from a foreign country, they will need to be sterilized prior to disposal. In this case, each container is marked with a yellow sticker indicating that the soils need to be sterilized prior to disposal. This will alert the chemists to the origin of the samples and will alert the Hazardous waste disposal staff of the special precautions necessary. (For Westborough see Qualtrax ID. 2296; for Mansfield see Qualtrax ID. 1526).

Samples Potentially Containing Asbestos:

Any sample with Asbestos analysis requested or that is noted by the client to contain or potentially contain Asbestos, should be labeled with yellow stickers indicating “May Contain Asbestos” and each container double bagged. Please refer to “Asbestos Handling and Sample Receiving Guidelines” (2532) for additional specific instructions for logging these jobs..

SPME Samples:

SPME samples cannot be frozen and must be refrigerated only. For all SPME samples received, orange stickers reading “DO NOT FREEZE” must be applied to the caps of the containers.

MS/MSD: For all sample containers that are to have MS or MS/MSD run on them, Pink labels are to be placed on the cap with QC indicated on them. For VOA Vials stickers can be placed on the side.

All Caps: For all containers large enough, caps are marked with the last 3 digits in the Alpha Job# followed by the sample # separated by a hyphen in colored marker. This is for ease of locating samples in refrigerators. Do not mark the caps of plastic containers for Method 537(PFA) analysis.

PH: If the pH of a sample container does not adjust into the proper range for the analysis being run on it after the maximum volume of preservative is added, a blue sticker is placed on top of the container cap. This alerts the lab personnel to the fact that the sample received was not properly preserved. If the sample is successfully preserved then a green sticker is placed on top of the container cap.

Soil Hexavalent Chromium: For separate soil containers provided by the client for MCP or CTRCP Hexavalent Chromium analysis, Orange “HEXCR ONLY” stickers must be placed on the cap and samples should be bagged together. This should also be done for any MCP or RCP sample requesting HexCr on a contingent basis.

5.16 Scanning the Chain of Custody

The Chain of Custody (COC) and any other pertinent information sent by the Client must be scanned into the LIMS for each job.

For MWRA jobs, the SMART sheets are scanned in the order the samples were logged in, followed by the COC. Any other information provided by the Client is scanned after the COC.

Chains can be scanned into the LIMS using the scanner program located on the copier in Login, Reporting or the Front Office. Save files with the file name as the job # with a “b” at the end, Ex: L1700001b. This will append the pages to any documents already saved as the chain of custody for this job number. If you need to completely replace the documents scanned as the chain of custody than scan without the “b” at the end of the file name. To add a page in front of the already scanned pages add an “f” to the end of the job #.

6. Quality Control and Operational Assessment

6.1 The digital and IR thermometers (Section 4.2) are calibrated on a semiannual basis by an instrument service company. Certificates are kept on file.

6.2 All Chains of Custody are reviewed by the appropriate Project Manager or Client Service Representative for approval.

7. Attachments

Table I: Hold-time Criteria
Table II: Analyte Preservatives

TABLE I: HOLD TIME CRITERIA

All analyses refer to aqueous matrices unless otherwise specified

15 minutes	pH
8 Hours	All Bacteria for non-drinking water samples Enterococcus Heterotrophic Plate Count Fecal Coliform Dissolved Oxygen
24 Hours	Hexavalent Chromium pH (soils, if also being run for MCP/RCP-Hexavalent Chromium) ORP (soils, if also being run for MCP/RCP-Hexavalent Chromium) Odor Total Residual Chlorine Ferrous Fe Chlorophyll A Chlorine Demand Residual Free Chlorine Hydrazine Free Cyanide (4500) ORP USP-TOC Conductivity Carbon Dioxide (Wet Chemistry) Sulfite MCP Metals filtration DOC filtration SDS ME-BOD ME-CBOD
30 Hours	Drinking Water Bacteria analysis (except Heterotrophic Plate Count & Fecal Coliform)
48 Hours	Surfactants-MBAS Nitrate, NO3 Nitrite, NO2 BOD Soluble BOD (SBOD) filtration w/ in 24 hours Carbonaceous BOD (CBOD) UV 254 Total Settlable Solids Orthophosphate Color Turbidity

	Tedlar Bags Perox Fix Gas Low Level 8260 preserved w/ Reagent H2O
3 Days	Formaldehyde Acrolein (624), unpreserved
7 Days	Ash (soils, solids) Moisture (Soils/Solids) Solids (aqueous, soils, solids) Sulfide Reactivity Semivolatiles (aqueous)-625,8270,AEXT,PAH Organic Extractables-TPHDRO, 8082,8081,8151,608 Unpreserved volatiles Dissolved Gas CO2
14 Days	Total Cyanide Physiologically Available Cyanide Amenable Cyanide Free Cyanide 9016 Reactivity (soil) 537 (PFAS) Semivolatiles (soil)- 8270, BEXT, AEXT, PAH Organic Extractables (soil)- TPHDRO, 8081,8151,8082, EPH EPH-Extractable Petroleum Hydrocarbons VPH-Volatile Petroleum Hydrocarbons 8260 524.2
28 Days	Total Organic Nitrogen-NH3, TKN, TON Total Organic Carbon Mercury APH

TABLE II: ANALYTE PRESERVATIVES

Unpreserved liquids

TPH-DRO
PAH-8270
8270
8082
8081
608 (Non-chlorinated, pH must be between 5 and 9)
8151
ETPH
625 (Non-chlorinated)
Formaldehyde
TCLP Metals
Pre-filter Soluble Metals
Chlorophyll A
Sulfite
Other Wet Chem liquid analytes not listed elsewhere
Pre-filter DOC
UV 254
624 (Non-chlorinated)
Ferrous Fe
Dissolved Gas CO_2
Volatile Organics (624,8260)

Unpreserved solids

8260
TCLP 8260
8021
TOC
Analytes in Oil
EPH
TCLP*
Wet Chem
Metals
Organics
TPH-DRO
TCLP Lamps
Chlorophyll (wipe/filter)

1:1 HCl pH<2

8260
624
524.2 (Non-chlorinated)
Dissolved Gases (Methane, Ethane, Ethene)
VPH
8021
EPH
Oil & Grease
TPH-1664

1:1 HNO_3 pH <2

Total Metals
Field-Filtered Soluble Metals

NaOH pH >12 (except wipes)

TCN
ACN
PACN
FCN-9016

1:1 H_2SO_4 pH <2

NH3
TKN
NO3/NO2
TON
TPHOS
COD
TOC
Field Filtered DOC

TPH-1664

Oil & Grease

TABLE II: ANALYTE PRESERVATIVES

(continued)

Ascorbic Acid /HCl

THM
524.2 (Chlorinated)

Sodium Thiosulfate

624 (Chlorinated)
625 (Chlorinated)
608 (Chlorinated, pH must be between 5 and 9)
504

Zn Acetate / NaOH pH >=9

Sulfide

MeOH -solids

8260/5035 High
VPH
8021/5035 High

NaHSO$_4$ solids

8260/5035 Low

Reagent Water -Solids

8260/5035A Low

0.2%HNO$_3$ – wipes

Total Metals

Hexane -Wipes

PCB
8270

Acetone – Wipes

Herbicides

0.1% NaOH –Wipes

Hexavalent Chromium

Hexane & Acetone-Wipes

PCB (from State of New Jersey)

Sodium Sulfite / NaHSO4 pH <4

522 - 1,4 Dioxane (Chlorinated)

Phosphoric Acid <2

TOC-5310

1:1 H2SO4 pH <4

TPhenol

Trizma Crystals

537 (PFAS)

Sample Custody and Tracking

1. Scope and Application

This procedure is used by the laboratory staff to track sample containers throughout the Laboratory using the Laboratory Management Information System (LIMS). This procedure is critical to ensure full sample container traceability.

2. Summary of Process

Sample containers are stored in predetermined refrigerators, freezers, or shelving units located in the Walk-In refrigerator and in certain areas of the Laboratory. Utilizing the LIMS, sample containers may be transferred to an Individual's Department, back to their proper storage location, or to Sample Archive. The temperatures of the refrigerators and freezers are checked and recorded daily to ensure that temperatures are being maintained.

3. Safety

The toxicity or carcinogenicity of each reagent used in this method is not fully established; however, each sample should be treated as a potential health hazard. From this viewpoint, exposure must be reduced to the lowest possible level by whatever means available. A reference file of safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices, otherwise known as universal precautions, for handling known disease causative agents.

4. Equipment and Supplies

4.1 Refrigerators

4.2 Thermometers and Vials with DI H2O

4.3 Freezers

4.4 Computer with LIMS access

4.5 Infrared Barcode Scanner

4.6 Carts

4.7 VOA Racks

4.8 Data Logger

5. Procedure

5.1 Sample Transfer from Login to Custody

5.1.1 Upon completion of sample Log-in (Sample Receipt and Log-In 1559), the sample containers are designated in the LIMS as being located in "Custody". Containers logged in Mansfield will reflect a location designation beginning with "A2-".

5.1.2 All samples are scanned to a specific custody location, and placed in the appropriate freezers or refrigerators by the Login Staff. Samples with rush turnaround times or short holding times may be taken by the lab directly after the login process is complete and in this case are placed in the appropriate storage areas by the lab staff once the container is no longer in use. When taking samples directly from login, as when taking from custody locations, the containers must be scanned out by the chemist to themselves and their department. See section 5.3.

5.1.3 Transfer of samples to Laboratory Custody.

5.1.3.1 All samples must be scanned to a specific custody location after log in and sample receipt procedures have been completed and when laboratory staff are no longer using the container. Refer to section 5.3 for scanning procedure.

5.2 Sample Storage

Sample containers are stored in specific freezers, refrigerators, or shelving units within the laboratory. Refer to Table I for Sample Placement / Custody Locations –Westboro.

5.2.1 Aqueous Samples

5.2.1.1 Wet Chemistry

Westboro: Samples for Wet Chemistry analyses are to be stored in the Walk-In refrigerator and scanned to the name of the specific shelving section where the sample is placed. The shelves within this refrigerator are named and labeled as follows: "W#-S#-X". Where W# indicates the rack number, S# the shelf within that rack and X the section of that shelf. (Example: W21-S3-C would be on rack 21, shelf 3 section C)

Mansfield: Samples are placed numerically into a refrigerator in Sample Management.

5.2.1.1.1 Exceptions

Samples to be analyzed for OG-1664, TPH-1664 and plastic containers preserved with Sulfuric Acid have their own separate refrigerators located in the Wet Chemistry lab.

5.2.1.2 Metals

Westboro: Samples requiring Metals analysis that are received in the Westborough facility, as well as extracts produced in the Westborough facility that are to be analyzed in the Mansfield lab are to be transported to the Mansfield facility using specially marked coolers located in Sample Login and the Walk-In Refrigerator. See section 5.7.

Mansfield: For the Mansfield facility, samples are placed on the metals shelf in the Metals Prep Laboratory.

5.2.1.3 Semi-Volatiles

Westboro: Samples for Semi-Volatile analysis are to be stored in the designated Organic Prep refrigerators located in the Organic Prep Laboratory. The shelves within these refrigerators are named and labeled as follows: "RXX-YY" where XX is the refrigerator's number and YY is the shelf number within the fridge.

5.2.1.3.1 Exceptions

Samples to be analyzed for Perchlorates, and EDB/DBCP by 504 or 8011 Methods have their own separate refrigerator and the shelves are labeled according to the analysis. This fridge is located in the Semi-Volatile Organics Laboratory.

Mansfield: For the Mansfield facility, samples are placed numerically into a refrigerator in Sample Management.

5.2.1.4 Volatiles

Westboro: The vials for Volatiles analysis are to stored in refrigerators located within the volatiles laboratory. 40mL vials are scanned to and stored in racks within the refrigerator named and labeled as follows: "VXX-YY" where XX is the refrigerator's number and YY is the rack number within the fridge. Containers that do not fit in the racks are placed in other refrigerators designated for this purpose in the volatiles lab and scanned to the specific refrigerator they are stored in. Containers in these refrigerators are to be placed on the shelf numbered with the last digit of the container's job number.

Mansfield: For the Mansfield facility, samples are placed into A2-CUSTODY-VOA8 for Piano Waters or A2-CUSTODY-VOA9 for Dissolved Gas and are bagged and placed into these refrigerators in AIR/Volatiles department.

5.2.2 Soil/Solids/Sludges/Oil & Wipe Samples

Westboro: Soil/Solid/Sludges/Oil & Wipe samples are to be stored in the Walk-In refrigerator and scanned to the name of the specific shelving section where the sample is placed. See section 5.2.1.1 for further detail on naming conventions in this refrigerator. All Sludges and Oil samples should always be bagged when being stored.

Mansfield: For the Mansfield facility, samples are placed numerically into A2-CUSTODY-REFRIG-D1, D2, or D3 refrigerator, located in the Custody Room. For extended storage soil samples are boxed and scanned to specific shelves in A2-CUSTODY-FRZ1 walkin Freezer.

5.2.2.1 Exceptions:

Westboro:

Containers submitted for volatiles analysis are to be stored in the same manner as aqueous samples as explained in 5.2.1.4 unless the vials are preserved with deionized water for low level volatiles analysis.

DI Water preserved Low Level Soil Vials are stored separately in specifically designated freezers. DI preserved Low Vials are always stored laying in a horizontal position until frozen to ensure that they do not break during freezing. Once frozen the vials are then transferred to racks located within freezers in the volatiles lab. These racks are named and labeled as follows: “FXX-YY” where XX is the freezer’s number and YY is the number of the rack within the freezer.

Vials submitted for Volatile Organics analysis for Oils, Product, or any sample with strong odors should be bagged, placed in the appropriately labeled Odor Seal containers located with the vials that have already been analyzed. These containers are to be stored in the walk-in refrigerator in the bin labeled for this purpose and scanned to the shelf the bin is located on.

Metals samples are transported to Mansfield in accordance with the instructions in 5.7.2 and stored in refrigerators located in the Metals Analysis lab. Each shelf of each fridge has a unique LIMS location name and samples should be scanned to those specific locations.

Mansfield: For the Mansfield facility, Methanol preserved High vials are scanned to A2-CUSTODY-VOA7, then bagged and placed into this fridge, and DI Water preserved soil vials are scanned to A2-CUSTODY-VOA9, bagged and placed into this freezer located in the AIR/Volatiles department. All oil samples, Voa or otherwise are to be scanned to either A2-CUSTODY-REFRIG-E1 or E2 located in the sample custody room.

5.2.3 Subcontracted Samples

Samples to be subcontracted to another Laboratory for analysis are to be scanned to and placed on the rack in the walk-in labeled for this purpose. Login staff and couriers are to transfer the containers to the location TRANSFER-SUB once taking the samples to deliver or ship to the subcontracting laboratory.

5.2.4 Extended Long Term Sample Storage

Westboro: Samples which the Client doesn’t want the requested analyses performed until further notice, are stored together with the rest of the Alpha Job# in the appropriate refrigerators. If a client has requested extended long-term storage, sample containers are scanned to the shelves in the Walk-In refrigerator reserved for this purpose.

Mansfield: All samples rec’d on hold in the Mansfield facility are placed into refrigerators/freezers as indicated above for soils/waters.

5.2.5 Air Samples

Whole air samples for volatile analysis are brought directly to the Air Laboratory, located at our Mansfield Facility. These samples are kept at room temperature. Tedlar bags are placed in a covered box or in a dark trash bag, without exposure to light. Samples collected using sorbent media should be stored according to method requirements, and locations are defined by the department conducting the analysis.

5.2.6 Sample Filtrates

Samples filtered in the Laboratory are assigned a new container ID and Label from the LIMS. Sample filtrates are collected in containers appropriate for the analysis requested, and labeled by the lab staff doing the filtration.

5.2.7 Sample Extracts

5.2.7.1 TCLP Samples

Samples extracted for TCLP analysis are collected in appropriate containers, and are assigned a new container ID and label from the LIMS. The containers are stored in the location appropriate for the analysis being run on them.

Exception: Metals extracts are collected in containers appropriate for the analysis requested and labeled by the lab staff completing the extraction. These extracts are stored in the Metals Prep Laboratory on the appropriate shelves, located in the Mansfield facility.

5.2.7.2 Organic Samples

Organic sample extracts are transferred to autosampler vials, hand-numbered and stored in refrigerators/freezers located within the analytical laboratory areas. Transfer of the extracts from the Organic Prep Department to the appropriate Analytical Department is recorded in the Prep logbooks. Management of Organic extracts is handled by the semi-volatiles department.

5.2.7.3 Metals Samples

Metals sample extracts are hand-numbered and stored in the Metals Analytical Laboratory area. Transfer of the extracts from the Metals Prep Department to the Metals Analytical Department is recorded in the Prep logbooks.

5.3 Sample Transfer from Custody to Individual

The LIMS is utilized to transfer a sample container out of Custody locations, or to transfer samples directly from login into a chemist's possession. The sample login department assigns each test to at least one container, if no container is applied to a test the sample level comments should be referenced for further instructions, in the absence of these comments the project manager or login staff must be consulted prior to analysis. If a test cannot be performed from the volume assigned in LIMS, login or the project manager must be consulted prior to using any volume that is not assigned to the test.

5.3.1 Using the computer located outside the Walk-In refrigerator, access the LIMS, by clicking on the "Seedpak LIMS" icon located on the Windows desktop screen.

5.3.2 A password screen will open. Type in the Username (CUSTODY) and Password (CUSTODY) and Database (BOWZER). Then click on <Connect> to the Bowzer database.

5.3.3 The main screen will open. From the Main Menu, select <Containers>, and then select <Transfer>. This will open the Container Transaction screen.

Type in the appropriate Username (Each staff member is given a Username from the IT Department to enable access the LIMS) in CAPITAL letters.

5.3.4 Select the appropriate department from the pull down menu where the analysis will be performed or, if returning a sample to storage, the location the container will be stored:

5.3.5 Click the <Check Out> button.

5.3.6 Scan the barcoded sticker on each sample container to transfer.

5.3.7 When scanning is complete, click the <Done/Save> button.

5.3.8 Samples are now in the analyst's custody from that department. Samples may be shared with other analysts within this department but must be checked back in to their proper storage location by the same department that scanned the sample out of storage. The LIMS recognizes the location of the sample container(s) has been changed from the storage location to the selected Department.

5.4 Sample Transfer from Individual to Custody

Once the chemist no longer requires the sample containers, they are returned to the proper custody location using the LIMS (as per Sections 5.3.1 – Section 5.3.6).

5.4.1 If a sample container is empty then it is returned as such. In the Status column, change "Intact" for that particular sample container to "Empty". (Follow RCRA guidelines to determine if container is empty.)

5.4.2 When scanning is complete, click the <Done/Save> button.

5.4.3 The LIMS recognizes the location of the sample container(s) has been changed from the Individual back to a Custody Location.

5.4.4 Sample containers are returned to the appropriate refrigerator, freezer or shelf. (See Table I). Empty containers are placed in a trash receptacle.

5.4.4.1 **Exceptions:** Bacteria sample containers and TOC vials utilized for sample analysis are placed into respective bins and shelves stored in the Dead Room, these do not require refrigeration after analysis.

5.5 Sample Transfer from Custody to Archive (Dead Room)

After a job is complete and a final report has been sent to the Client, all sample containers are transferred from their custody locations to the Dead Room. Extracts stored within the custody refrigerators can be disposed of 8 days after creation. The date of extraction is written on the tops of the caps

If the job has not yet been invoiced/reported to the Client, a window will pop up that says "Job has not been invoiced, containers may still be needed by chemists." Click <Reject> button and return samples to appropriate custody refrigerator.

If samples need to be held for extended long term storage, or containers returned to the Client, a window will pop up that says "STOP SCANNING. Disposal question answered with an N." These containers are separated from the rest of the samples so they can be assessed by the Sample Custody staff. They will then be put on the specified shelves or refrigerators for long term storage, or returned to the client. Refer to section (5.2.4).

5.6 Refrigerators and Freezers

The Industrial refrigerators undergo maintenance/cleaning by an outside vendor as needed. If a refrigerator is ever taken out of use and/or requires repairs this information is documented in the Refrigerator Maintenance Logbook located in the Login Department. For the Mansfield facility, any maintenance/issues with the refrigerators are listed on the daily temperature tracking sheet in the comment section.

5.6.1 Temperature Check

Each refrigerator or freezer contains a thermometer or thermocouple for the datalogger. The temperature is checked and recorded on a daily basis to ensure the appropriate temperature. The refrigerators must be at a temperature of $\leq$ 6 degrees Celsius, but $\geq$ 2 degrees Celsius. The temperature is still within acceptable criteria if it drops below 2° and the samples are not frozen. The freezer must be at a temperature of less than -7 degrees Celsius but greater than -20 degrees Celsius. Periodic adjustment to temperature controls are made as necessary.

5.6.1.1 Data Logger – Westboro and Mansfield

The majority of the freezers and refrigerators are connected to the Data Logger by a numbered thermocouple.

Refer to Form No.: 08-104 for a Westboro listing of refrigerators and freezers and the corresponding datalogger input number.

Refer to Form No.: 108-27 for a Mansfield listing of refrigerators and freezers and the corresponding datalogger input number.
The Data Logger is programmed to continuously record the temperature of each refrigerator or freezer. The temperatures recorded from the Data Logger are verified once each day by a Login/Custody Staff member to ensure that temperatures are within acceptance criteria. See Data Logger Operation SOP document #1747.

5.6.1.2 Manual Temperature Check

Westboro & Mansfield:
All freezers and Refrigerators in the Westboro and Mansfield facility are connected to the Data Logger. In the case of a new refrigerator/freezer being installed, or issues with the Data Logger, then they would be manually checked daily by a representative of the department where the fridge is located and recorded in the Refrigerator/Freezer Temperature Logbook (Form No.: 08-05), to ensure that temperatures are within acceptance criteria. Refer to Work Instruction #14006 on instruction on how to use Min/Max Thermometers and record temperatures manually.

5.6.1.3 Disposal

Prior to disposal of any refrigerators or freezers the Sample Management department must notify the Environmental Health and Safety Department of the need to decontaminate the item prior to removing the item from service. The EH&Safety department will coordinate the Refrigerant removal by a Certified Refrigerant Removal Company. Once the Freon has been removed, the EH&Safety Department will notify the Facility Maintenance department to have the item removed from the facility.

5.7 Sample Transfer Between Mansfield and Westborough Facilities

5.7.1 Scanning Samples and Extracts to Coolers

Any samples or extracts that are located in one Facility but are needed for analysis in the other must be transported by an Alpha Courier in specifically designated coolers that are individually numbered and used only for this purpose. All samples and extracts must be scanned in LIMS to the cooler they are to be transported in using the Transaction Screen (see section 5.3.2). The location will consist of the word "COOLER" followed by the cooler number and either "TRANSFER_TO_MANSFIELD" if the Mansfield lab is the destination or "TRANSFER_TO_WESTBORO" if the Westborough lab is the destination.

5.7.1.1 Method 537(PFAS) aqueous sample containers submitted for analysis by EPA method 537 must be retained in the Ziploc bags they were received in, and transferred in a separate cooler from other containers and/or analysis.

5.7.2 Checking Coolers Into or Out of a Facility

When a courier is ready to transport the coolers to their destination location they must log into LIMS following 5.3.1 and 5.3.2 above and select Check Out under the "COOLERS" heading at the bottom of the transaction window. This will open the COOLER_TRANSFER window. The courier must enter their username, ensure the correct facility is displayed in the "Transporting To:" field, and then select Show Coolers. Once Show Coolers is selected, any coolers that contain samples will be displayed in the table, the courier then must select using the checkboxes to the left of the table, which coolers they are transporting and then press the large yellow box labeled "TRANSFER". All samples being transported, with the exception of aqueous samples for metals analysis only, must be transported covered in ice.

When arriving at the destination facility the courier must repeat the instructions in 5.3.1 and 5.3.2 and then select Check In under the "COOLERS" heading at the bottom of the transaction window. The courier than enters their username, ensures the Site Receiving Containers matches the facility they are in and then selects Show Coolers. Once Show Coolers is selected, all coolers that have been checked out to the LIMS username entered will be displayed. The temperature of each cooler must be taken in accordance with SOP 1559 5.1.1.1.4, except the temperature recorded and IR Gun Serial Number used are to be entered into the fields in the table to the right of each cooler. The TRANSFER button is then selected and the location of all containers in the selected coolers will be changed to match the LIMS location in the field above labeled "Location Receiving Containers".

Sample containers should then be transferred to their proper storage locations depending on their matrix and the department performing the analysis as indicated in section 5.2.

6. Quality Control and Operational Assessment

6.1 Calibration

6.1.1 Thermometers

The thermometers in each refrigerator or freezer are calibrated prior to use and on an annual basis by the QA Department. Records are maintained on file by the QA Department.

6.1.2 Data Logger

The Data Logger is calibrated by an outside vendor. See Data Logger Operation SOP document #1747.

7. Corrective Actions

7.1 Broken Sample Containers

If at any time a sample container is broken, a Login/Custody Staff member must be notified immediately. The Login/Custody Staff member changes the container status in the LIMS from “Intact” to “Broken” and determines whether the entire sample has been compromised or if any can be saved.

7.1.1 Refer to Cleanup and Disposal of Broken Samples (2691).

7.2 Improper Temperature

If any of the refrigerators or freezers are above or below the required temperature (Section 5.6.1) the cause of the exceedance must be investigated. If the door is open it will be closed and the temperature checked again within three hours to ensure it is back within the proper range. If the door is not open, then the temperature must be read by using an infrared thermometer and taking a temperature reading of the probe. If it is determined that there is a problem with the refrigerator or freezer then all samples must be removed from that refrigerator or freezer and placed in another appropriate refrigerator or freezer. This ensures that the integrity of the samples is maintained. An outside vendor is contacted for service.

8. Attachments

Table I: Sample Placement / Custody Locations – Westboro

TABLE I: SAMPLE PLACEMENT / CUSTODY LOCATIONS – Westboro

(VOA CUSTODY) Volatile Organics Laboratory (R-23, R-24, R-33, R-41, R-48, R-49)

8260	VPH
624	TPH-GRO
524.2	ME4217

(VOA DEAD CUSTODY) Volatile Organics Laboratory & Walk-In Refrigerator (R-48)

All Volatiles Vials that have been utilized for sample analysis.

(OPREP CUSTODY) Organic Prep Laboratory (waters and liquids) (R-12 & R-27, R-60)

8082	625
8081	8270
608	8151
ETPH	8270

TPH-DRO	EPH

(R-46 CUSTODY) Organics Laboratory	**(R-47 CUSTODY) Organics Laboratory**
PERC	TCLP/SPLP & ASTM Leachate Extracts
504	for Organic analysis
8011	

TABLE I: SAMPLE PLACEMENT / CUSTODY LOCATIONS - Westboro-Cont.

(Waters for Wet Chemistry and non-volatiles soils) Walk-In Refrigerator (W#-S#-X)

Water and Liquid samples for Wet Chemistry only

Soils, sludges, solid materials, oils, wipes, unknown liquids, mixed liquids, solvents, etc. for all non-Volatiles analysis

Sludges and oils must be placed into plastic locking bags.

(R-62 CUSTODY) Wet Chemistry Laboratory

OG-1664

TPH-1664

(R-44 CUSTODY) Wet Chemistry Laboratory (aqueous only)

COD	NO3/NO2
NH3	TPHOS/SPHOS
TKN	TPO4

(FREEZER) Volatiles Laboratory (F-11 & F-12, F-24, F-26)

8260 Reagent Water vials	8260 Reagent Water Vials (hold)
5g/25g Encores	5g/25g Encores (hold)

Bottle Order Preparation

1. Scope and Application

This procedure is used by the Alpha Staff to correctly set-up, package, and distribute a Bottle Order to an Alpha Client. This information is critical to ensure that the Client submits samples to the laboratory that are correctly preserved, in the appropriate containers, and have sufficient volume needed for the analysis requested.

2. Summary of Process

Bottle Order requests are entered into the Laboratory Information Management System (LIMS) by authorized Alpha staff that have been trained to enter orders. The Bottle Order is filled and packaged according to the information provided in the Bottle Order request. The packaged Bottle Order is delivered to the Client via Alpha Courier, FedEx, UPS, or is picked up by the Client themselves.

3. Safety

The toxicity or carcinogenicity of each reagent used in this method is not fully established; however, each reagent should be treated as a potential health hazard. From this viewpoint, exposure to chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene plan.

All personnel handling these reagents must follow safety practices, otherwise known as universal precautions.

4. Equipment and Supplies

4.1 Preservatives Prepared by Bottle Prep staff: All preservatives listed below are prepared by the Bottle Prep staff with reagent-grade acid or base and Deionized Water. All preservative prep information is contained in logbooks located within the bottle prep rooms. The date, time, preservative name and ID#, department that prepared the preservative and the initials of the staff member filling the bottle are recorded in the Bottle Room Preservative Tracking Log located on wall under the hood in the Bottle Room. (See Section 6.3).

4.1.1 **Hydrochloric Acid (HCL), 1:1:** Prepare by adding 1L of DI water to an acid rinsed 2L Erlenmyer flask followed by 1L of concentrated HCL. Store at room temperature. Expires one year from preparation or according to manufacturer's expiration date, whichever is sooner.

4.1.2 **Sulfuric Acid (H_2SO_4), 1:1:** Prepare by placing an acid rinsed 2L Erlenmyer flask in an ice bath. Add 1L of DI water then 1L of H_2SO_4. Allow to cool. Store at room temperature. Expires one year from preparation or according to manufacturer's expiration date, whichever is sooner.

4.1.3 **Nitric Acid (HNO_3), 1:1:** Prepare by adding 1L of DI water to an acid rinsed 2L Erlenmyer flask followed by 1L of concentrated HNO_3. Store at room temperature. Expires one year from preparation or according to manufacturer's expiration date, whichever is sooner.

4.2 Pre-packaged Preservatives:

4.2.1 Sodium Hydroxide Pellets (NaOH): American BioAnalytical

4.2.2 Sodium Thiosulfate Crystals ($Na_2S_2O_3$): J.T. Baker; 5-Hydrate crystal

4.2.3 Acetone/Hexane: Pesticide quality or equivalent; stored in the Bottle Prep Department

4.2.4 Manganous Sulfate Powder Pillows: HACH brand. Labeled as “#1”, located in the Microbiology Lab.

4.2.5 Alkaline Iodide-Azide Reagent Powder Pillows: HACH brand. Labeled as “#2”, located in the Microbiology Lab.

4.3 Pre-Preserved Containers (See Table II): With Certificates of Analysis

4.4 Unpreserved Containers (See Table III)

4.5 Certified Containers: With Certificates of Analysis

4.6 Glass BOD Bottles, stoppers and plastic caps

4.7 Computer with LIMS access

4.8 HP Laserjet P2055dn Printer

4.9 Zebra Label Writer (Labels, Ribbon)

4.10 Writing Implements: Pens, Markers, Highlighters

4.11 Corrosive Preservative Stickers: HNO_3, HCL, H_2SO_4, NaOH, Na2S2O3, Hexane. For use on individual sample containers.

4.12 Custody Seal Stickers: Name, date, time

4.13 Encore Sample Packs & T-Handles: 5g, 25g

4.14 Plastic 2mL Transfer Pipettes: Samco

4.15 Soil Syringes: 5mL, individually wrapped

4.16 20mL Syringes: Disposable, sterile

4.17 Syringe Filters: Sterile, with 0.2µm Cellulose Acetate membrane

4.18 Cardboard Boxes (various sizes)

4.19 Packing tape

4.20 Packing paper

4.21 Coolers

4.22 GHS Stickers

4.23 Temperature Blanks: 60mL plastic containers filled with tap water and capped tightly. “Temp Blank” is written in permanent marker on the cap and side of the bottle.

4.24 Liquinox Cleaner: Dilute as 100 parts DI water: 1 part cleaner.

4.25 Universal Sorbent Pads

4.26 DI Water in Spray Bottle

4.27 DI Water in carboy: Carboy filled from DI unit in either Wet Chemistry Department or Metals Prep Department.

4.28 VOC-free water: Taken from the Volatiles Department. This is municipal water, passed through a column of activated carbon.

4.29 Bubble Bags (various sizes)

4.30 Ziploc Bags (various sizes)

4.31 Bubble Wrap

4.32 Gauze pads: 2” x 2” size

4.33 Alpha Chains of Custody

4.34 Sampling Instructions:

4.34.1 Perchlorate: Alpha WI/01-07 - 2287

4.34.2 Dissolved Oxygen: Alpha WI/01-03 - 2262

4.34.3 for Ascorbic Acid/HCL preservation: Alpha WI/01-04

4.34.4 Sulfide: Alpha WI/01-02 - 2277

4.34.5 Volatile Organics by 5035 (8260, 8021 & VPH): Alpha WI/01-05 - 2281

4.34.6 **Encore Sampling:** Alpha WI/01-16 - 2415

4.35 GHS Labeling:

4.35.1 GHS Labeling for Sample Containers with Preservatives (Qualtrax ID 17976) document.

5. Procedure

5.1 Log on to the LIMS

Access the LIMS, by clicking on the ACS LIMS / Seedpak icon located on the Windows desktop screen.

A password screen will open. (Each person is given a name and password to access the LIMS.) Type in your name and password, then tab down and type in “bowzer” in the database space, and select < Enter >.

5.2 Match Bottle Order with Labels:

A Bottle Order prints from the LIMS onto the printer in the Bottle Room. It is matched up with labels that print from the Zebra Label printer also located in the Bottle Room. Both the Bottle Order sheet and the Labels will have a Bottle Order # located at the top of the sheet and down the right side of labels.

5.2.1 **Air Orders, Mansfield only:** If a bottle order prints for Air Canisters, Tedlar Bags or PUF Cartridges, the Bottle Order and Labels are brought to the Air Lab and given to Air Lab personnel. All Air Orders and Sampling Instructions are completed and maintained by Air Lab personnel.

5.3 Organize Bottle Orders:

After the labels and bottle order sheets are matched up, they are organized according to deliver date. There is a plastic crate on the counter next to the Zebra label printer that holds hanging folders for each day of the week. After the orders are printed, the sheet and labels are attached together with a paper clip and placed in the proper folder. This allows the Bottle Order staff to prioritize fulfilling the orders, and ensures orders are completed on time.

5.4 Pull Bottles:

Pull all containers listed on the Bottle Order from stock supplies, matching up size, container type and correct preservation. Many vials in stock are pre-preserved and when first opened will contain “Certificates of Analysis”. See Section 6.1 for further instructions.

If Certified Bottles are requested on Bottle Order, only use Certified/Pre-cleaned containers located at the back wall of Bottle Room, and/or pre preserved vials. See Section 6.2 regarding the procedure for retaining Certified/Pre-clean Certificates. Section 6.2.1 outlines the cleaning procedures followed by the Manufacturer for all Certified/Pre-cleaned containers.

All Bottle Orders including MeOH-, NaHSO4-, and/or Reagent H_2O- preserved vials for soil must include one (1) Soil Syringe (Section 4.16) per sample and appropriate Sampling Instructions.

5.5 Bottle Preservation:

Containers requiring preservation are preserved using acids/bases located under the hood in the Bottle Room. [See Bottle Preservation Guide (Table I) for correct volume of acids to add to each container type.] The containers preserved in-house / if needed (HCl-preserved Ambers, H_2SO_4-, HNO_3- and NaOH-preserved plastics) are stored on labeled shelves within the Bottle Room. Plastic containers preserved with the following must have the corresponding dot sticker placed on the cap: NaOH, H2SO4, HNO3. Exceptions to the Bottle Preservation Guide (Table I) include the following:

5.5.1 **Dissolved Oxygen (DO)**: A plastic BOD bottle is provided along with the glass stopper and tall plastic cap located in the Microbiology Lab. Each bottle, stopper and cap is bagged in a 6X8 Ziploc bag with a #1 (Section 4.2.6) and #2 (Section 4.2.7 & 4.2.8) Powder Pillow, clearly marked with a marker. Appropriate Sampling Instructions are included.

5.5.2 **Wipes:** 4oz Amber jars are used for all wipes. Using the Tweezers, add one 2” x 2” gauze pad in each jar. Add the appropriate preservative on top of the wipes, as follows:

5.5.2.1 **SVOCs, AlkPAH, O&G, PCB, P/H, TPH, ETPH**: Add 5mL Acetone/Hexane

5.5.2.2 **Cyanide, Inorganics, HexCr, Metals**: Add 5mL of DI Water.

5.5.3 **VOCs**: Add 5mL of MeOH. Include a 15 ml MeOH vials.

5.5.5 **GHS (Global Harmonized System) Labeling:** reference the GHS Labeling guide for containers with Preservatives (Qualtrax ID 17976) document. All containers with preservatives require all applicable GHS labels to be affixed to them.

5.6 Trip Blanks:

Bottle orders for 504 and 524 analyses must contain two Trip Blanks; otherwise Trip Blanks are only supplied upon Client request. 40mL Amber vials with Teflon septa are utilized. Trip Blanks are prepared and filled by the Bottle Order staff at the Laboratory per the directions below:

5.6.1 Water vials (those vials preserved with HCl, Ascorbic acid, H_2SO_4, Ammonium Chloride, or Sodium Sulfate) are filled with VOC-free water taken from the Volatiles Department (Section 4.29). Ensure no headspace or air bubbles are present in the vial. The bottle order sticker is attached to the vial (Section 5.5) and the date and initials of the staff member filling the vial are all recorded on the sticker.

5.6.2 Soil vials containing preservative (Table II) are affixed with the bottle order sticker (Section 5.5). The date the label is affixed is recorded as the sampling date on the sticker. Also recorded are the initials of the staff member affixing the label.

5.7 Attach Labels: Labels are attached to the appropriate container horizontally (left to right) in the middle of the container with the exception of:

5.7.1 **Vials**: All vials are separated by test/preservative and bagged in appropriate sized Ziploc bags. Labels are placed in the bag with the vials, not attached.

5.7.1.1 **Methanol (MeOH) preserved Vials**: Labels are placed on vial in the Vertical position with “CORROSIVE” listed at the top of label. The left hand side of the label must be level with the bottom of the vial and as close to the bottom of the vial as possible. The label must be placed directly to the left of, but not covering the lot #, Serial#, and Production Date on the pre-printed manufacturer's label on vials. At no time should the vial tare weight, located at the top right hand side of the manufacturer’s label, be covered.

5.7.1.2 **Sodium Bisulfate (NaHSO4) & Reagent Grade H2O preserved Vials**: See instructions above for Methanol.

5.8 Packaging Bottle Order

5.8.1 **Cooler:** If the “Special Shipping Requirements” section at the bottom of the Bottle Order request has “Cooler” checked off, then the bottle order must be sent to the Client in a cooler.

5.8.1.1 Choose an appropriate sized cooler from the cooler storage room. Wipe the inside of the cooler of any excess water and clean with Non Phosphorous Spray Cleanser (Section 4.25), wipe again and dry. Next, spray the cooler down with DI water and wipe dry.

5.8.1.2 Load all containers into the cooler. All vials are bagged in Ziploc bags and placed in the upright position, and all preserved containers should be in an upright position. All Amber containers are placed in Bubble Bags (Section 4.30) if being sent via UPS or FedEx.

5.8.1.2.1 **NOTE:** Verify that all containers with preservative are tightly closed.

5.8.1.3 Include a “Temperature Blank” in every cooler.

5.8.1.4 Use crumpled newspaper and/or bubble wrap around the containers to fill any gaps in the cooler and also ensure the containers stay in place during cooler delivery. Make sure there is enough space left in the cooler to account for the addition of ice prior to return of samples to the Laboratory.

5.8.1.5 Include Paperwork: All paperwork is bagged in a 10X12" Ziploc bag in the following order, from top to bottom: Bottle request, Chains of Custody (1 per 10 sample sets, then add 1 extra), and all applicable Sampling Instructions. The Ziploc bag containing the paperwork is be placed on top of sample containers in the cooler. Also include soil syringes, if appropriate.

5.8.1.6 The cooler is taped closed with packing tape and a "Box Label" (the first label printed containing Client's name, project name, and date required, Section 5.1) is placed on the top of the cooler.

5.8.1.6.1 If more than one cooler is filled for a particular bottle order, additional "Box Labels" will need to be printed. Do this by accessing the LIMS system. Under the "Applications" menu, select "Bottle Orders" and <OK> at prompt. Next click on <QUERY> at the bottom, and enter the Bottle Order # at the top of screen. Click on "Enter Query". After verifying that this is the Bottle Order required, click on "Print Bottle Labels" at the right hand side of screen. Then tag the "Box Label Only" square, choose the amount of box labels needed, and click on <PRINT>.

5.8.2 **Box:** If "Cooler" is <u>not</u> checked in the "Special Shipping Requirements" section, the bottle order may be submitted in a box. Choose an appropriate sized box. All procedures above are followed except a Temp Blank is not included.

5.8.2.1 If more than one box is filled for a particular bottle order, additional "Box Labels" will need to be printed. Do this by accessing the LIMS system. Under the "Applications" menu, select "Bottle Orders" and <OK> at prompt. Next click on <QUERY> at the bottom, and enter the Bottle Order # at the top of screen. Click on "Enter Query". After verifying that this is the Bottle Order required, click on "Print Bottle Labels" at the right hand side of screen. Then tag the "Box Label Only" square, choose the amount of box labels needed, and click on <PRINT>.

5.8.3 **Certified Containers:** If "Certified Containers" is checked in the "Special Shipping Requirements" section, then only certified containers (Section 4.5) may be used in filling the bottle order.

5.9 Saving Bottle Orders as DONE: When completed, all bottle orders are saved in the LIMS system as DONE. Do this by accessing the LIMS system. Under the "Applications" menu, select "Bottle Orders" and <OK> at prompt. Next click on <QUERY> at the bottom, and enter the Bottle Order # at the top of screen. Click on "Enter Query". Verify that this is the Bottle Order required. The initials of the person filling the Order must be entered in as "Completed by:" and in the Bottle Schedule window on Right side, click on "Done" and then click on "Save" at the bottom.

5.10 Placement of Completed Order: All orders are divided according to whether they are to be picked up by the Client, brought with a Courier, or shipped via UPS or FedEx.

5.10.1 **Client Pickup**: All orders to be picked up by Client are placed on the wire rack labeled "Bottle Orders for Client Pickup", located in the Courier staging room.

5.10.2 **Courier**: All orders taken to Clients by Alpha Couriers are placed in the Courier staging area.

5.10.3 **Shipping**:

5.10.3.1 **UPS:** All orders shipped via UPS are placed inside the sliding door to the Courier staging area by 4:30pm. See procedure for shipping via UPS (Table IV). Service centers should check with local drop locations on acceptance of packages for shipping via air or ground.

5.10.3.2 **FedEx:** All orders shipped via FedEx are placed on the floor inside the sliding door to the Courier staging area. FedEx orders must have a pickup requested. See procedure for shipping via FedEx (Table V). Service centers should check with local drop locations on acceptance of packages for shipping via air or ground.

5.11 Bottle Order Status

The status of Bottle Orders is verified on a daily basis to ensure all Bottle Orders are completed in a timely manner. A list of Bottle Orders that have not been completed is printed from the LIMS as follows:

- Under the "Reports" menu, select "Status", then select "Bottle Orders".
- Enter the date in the "Report Date" screen and then select "Background (IDXL)".
- Click <OK> and enter the printer class to print to, and select <Print>.

6. Quality Control and Operational Assessment

Certificates of Analysis supplied from the Bottle Distributer are retained to ensure validity of pre-preserved vials and Certified/Pre-cleaned containers. Log books are kept, detailing when preservatives are made and the Lot# with which they were made with. Each new Lot of bacteria sampling containers are verified to be sterile by the Microbiology Department. A procedure is in place with multiple checks during the process of entering bottle orders into the LIMS.

6.1 Certified Bottle Orders: All Bottle orders are filled with certified containers. Upon request copies of the "Certificate of Analysis" are included with the bottle order. The Lot numbers of certified containers are saved in the LIMS.

6.1.1 Certified/Pre-clean Procedure: A cleaning procedure is followed by the manufacturer for all Certified/Pre-cleaned containers and is as follows:

Ambers:
1) Laboratory-grade phosphate-free detergent wash.
2) Acid rinse.
3) Multiple hot and cold Deionized water rinses.
4) Oven Dried.
5) Capped and packed in quality controlled conditions

Plastics:
1) Laboratory-grade phosphate-free detergent wash.
2) Acid rinse.
3) Deionized water rinse.
4) Air Dried.
5) Capped and packed in quality controlled conditions.

6.2 Preservatives: All preservatives prepared by the laboratory staff are made with reagent-grade acid or base and Deionized Water. All preservative preparation information is contained in logbooks located within the appropriate prep laboratories. The date, time, preservative name and ID#, department that prepared the preservative and the initials of the staff member filling the bottle are recorded in the Bottle Room Preservative Tracking Log located on wall next to the hood in the Bottle Room.

6.3 Sterile Bacteria Containers: With each new Lot of sterile sample cups, a single item from the Lot is sent to the Microbiology Lab for sterility testing. The Microbiology Analyst gives a copy of the Microbiology analysis logbook documenting the results to a member of the Login staff. Only after sterility is confirmed may any sterile item be used. Results are entered into a spreadsheet format in the Bottle Room.

7. Corrective Actions

Not applicable.

8. Attachments

Table I: Bottle Preservation Guide
Table II: Pre-preserved Vials
Table III: Un-preserved Bottles
Table IV: UPS Shipping Instructions
Table V: FedEx Shipping Instructions

9. Referenced Documents

(WI 01-07)	Perchlorate Collection Instructions.
(WI 01-06)	Residential DW Collection Instructions.
(WI 01-03)	Dissolved Oxygen Collection Instructions.
(WI 01-04)	524.2 Collection Instructions for Ascorbic/HCL Preservation.
(WI 01-02)	Sulfide Collection Instructions.
(WI 01-05)	Volatile Organics Collection Instructions for 5035 (8260, 8021, & VPH).
(WI 01-16)	Encore Sampling Instructions.
(WI 01-15)	Perchlorate ICMSMS Sample Collection
Form No.: 01-05	Login Preservative Tracking Log

TABLE I

Bottle Preservation Guide

Bottles are preserved with the following volumes according to bottle size.

(Exceptions are listed in Sections 5.3.1 through 5.3.3)

VIALS

20 mL Vial – 1 drop of Acid.
40mL Vial – 2 drops of Acid.

PLASTIC BOTTLES

Plastic 120mL – 0.5mL of Acid
Plastic 250mL – 1mL of Acid
Plastic 500mL – 2mL of Acid
Plastic 1000mL – 4mL of Acid

Plastic 250mL – 3 NaOH Tablets
Plastic 500mL – 6 NaOH Tablets
Plastic 1000mL – 9 NaOH Tablets

AMBER BOTTLES

Amber 250mL – 1mL of Acid
Amber 500mL – 2mL of Acid
Amber 950mL – 5mL of Acid

Amber 950mL – 6 Tablets (0.08g) $Na_2S_2O_3$

When pre-preserved containers are unavailable

40mL Vial – 2 drops of HCl
40mL Vial – 2 drops of H_2SO_4

TABLE II

Pre-preserved Containers

(Refer to Section 4.3)

40mL Vial w/ 0.2ml Hydrochloric (HCL)

40mL Vial w/ 0.5 ml Sulfuric (H_2SO_4)

40mL Vial w/ 25 mg Ascorbic Acid

40mL Vial w/ 65 mg Ammonium Chloride (NH_4CL)

40mL Vial w/ 10 mg Sodium Thiosulfate ($Na_2S_2O_3$)

40mL Vial w/ 15 ml Methanol (MeOH)

40mL Vial w/ 5 ml Sodium Bisulfate ($NaHSO_4$) w/ large stir bar

40mL Vial w/ 5 ml Reagent Grade H_2O

120mL Bacteria Cup w/ 10mg $Na_2S_2O_3$

TABLE III

Unpreserved Containers

(Refer to Section 4.4)

H2O	**SOIL**
40mL Vials	4 oz Soil Vial w/Septa cap
100mL Amber	100mL Amber Soil Jar
250mL Amber	250mL Amber Soil Jar
500mL Amber	2 oz Plastic for Soil (TS)
950mL Amber	
120mL Bacteria Cup	
120mL Plastic	
250mL Plastic	
500mL Plastic	
950mL Plastic (Wide Mouth)	
1 Liter Plastic Cylinder	
120mL Plastic – Pre-cleaned (used for Perchlorate)	

1 Liter Black Plastic (used for Chlorophylls)
1 Gallon Cubitainer
2.5 Gallon Cubitainer
10L Carboy container

Certified & Pre-cleaned
100mL Amber Soil Jar
250mL Amber Soil Jar
120mL Amber (Wide Mouth)
950mL Amber
250mL Plastic
500mL Plastic
950mL Plastic (Wide Mouth)

TABLE IV

UPS Shipping Instructions

1. Log onto the Internet
2. Type in "www.campusship.ups.com", press enter.
3. Type in user ID and password. Click on "Login".
4. Click on "Create a Shipment"
5. Scroll down to bottom of next screen and click on "Create a Shipment" again.
6. Enter Address Info by choosing to enter "New Address" or a stored address from the drop down menu in "My UPS Address Book".
7. Under "Shipment Information" enter Service Type, Pkg Weight, Pkg Value (usually $100 will do), and dimensions of package or packages.
8. Click on "Preview Shipment" to make sure all information is accurate. This screen will also give a guaranteed date/time of delivery.

   NOTE: If Saturday delivery is required, under "Shipment Information" ensure that the check box for "Saturday Delivery" is checked. Affix 'Saturday' stickers onto the package(s).
9. Click on "Ship Now"
10. Click on "View/Print Label"
11. Click on "File", then "Print", then click "OK"
12. Remove printed Label from Printer and cut along borders.
13. Place face down on the wide shipping tape dispenser, pull out desired amt of tape, then cut and place on top of package.
14. Place the shipment in the UPS drop zone. They will pick up automatically everyday around 4:30 pm.

TABLE V

FedEx Shipping Instructions

1. Log onto the Internet.
2. In the browser, type in "www.fedex.com" and press enter, then select "Login to My FedEx".
3. Type in user ID and password. Click on "Login".
4. Click on "Ship", then "Prepare Shipment On line" in drop down menu.
5. Enter Address Info. A new address may be entered or use a stored address from the drop down menu.
6. Under "Shipping Information" enter Service Type, Pkg Weight, Pkg Value (usually $100 will do), and dimensions of package or packages.

7. Click on "Continue" or "Get Rate" to make sure all information is accurate. This screen will also give a guaranteed date/time of delivery**.**
8. In Special Services, click on Signature and/or Saturday pickup or delivery.
9. Schedule a pickup (all FedEx orders must be scheduled, they do not automatically come).
10. Click on Continue, and then confirm pickup.
11. Click on Continue again, then Print label.
12. Remove printed Label from Printer and cut along borders.
13. Place upside down on the wide shipping tape dispenser, pull out desired amt of tape, then cut and place on top of package.
14. Place the shipment next to the UPS drop zone (not in the drop zone).

Total Suspended Solids Dried at 103-105ºC

Total Volatile Suspended Solids Dried at 500°C

References: **SM 2540 D (for TSS) and SM 2540 E (for TVSS)**, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is suitable for the determination of Total Suspended Solids (TSS) and Total Volatile Suspended Solids (TVSS) in potable, surface, and saline waters, as well as domestic and industrial wastewaters.

Definitions: Refer to Alpha Analytical Quality Manual.

"Solids" refer to matter suspended or dissolved in water or wastewater. Solids may affect water or effluent quality adversely in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction in the transient consumer. For these reasons, a limit of 500mg dissolved solids/L is desirable for drinking waters. Highly mineralized waters also are unsuitable for many industrial applications. Waters high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

"Total solids" is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include "total suspended solids," the portion of total solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

"Fixed solids" is the term applied to the residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature. The weight loss on ignition is called "volatile solids." Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A well-mixed sample is filtered through a weighed standard glass-fiber filter, and the residue retained on the filter is dried to a constant weight. The increase in the weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and the total dissolved solids may provide an estimate of the total suspended solids. Samples submitted for Total Volatile Suspended Solids analysis are also dried at a higher temperature to a constant weight.

2.1 Method Modifications from Reference

Filters are prepared and analyzed at 500°C.

Method specifies that duplicate determination should agree within 5% of their average weight; Alpha is using in-house control limits.

3. Reporting Limits

The RDL for both TSS and TVSS analysis is 5mg/L using 1000mL of sample and an analytical balance sensitive to 0.1mg. A lower RDL may be achieved by using a larger sample (more volume) or the use of a more sensitive balance.

Note: TSS-LOWmethod should be used for samples where client requested reporting limit lower than 5 mg/L. Reporting limit for low TSS-LOW method is 1.0 mg/L. Analyst must use 5 digit balances in order to achieve low reporting limit.

4. Interferences

4.1 Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing.

4.2 Mix small samples with a magnetic stirrer. If suspended solids are present, pipet with widebore pipets. If part of a sample adheres to the sample container, consider this in evaluating and reporting results. Some samples dry with the formation of a crust that prevents water evaporation; special handling is required to deal with this. Avoid using a magnetic stirrer with samples containing magnetic particles.

4.3 Exclude large, floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not desired in the final result.

4.4 Disperse visible floating oil and grease with a blender before withdrawing a sample portion for analysis.

4.5 Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200mg residue.

4.6 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material.

4.7 Prolonged filtration times resulting from filter clogging may produce high results due to increased colloidal materials captured on the clogged filter.

4.8 If the sample has high sediment/sand content, it is necessary to use a smaller volume for analysis. This will allow for a smooth transfer of the filter to the drying tin.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles, provided that the material in suspension does not adhere to container walls.

Sampling, subsampling, and pipetting two-phase or three-phase samples may introduce serious errors. Make and keep such samples homogeneous during transfer. Use special handling to insure sample integrity when subsampling.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4 ± 2ºC up to the time of analysis to minimize microbiological decomposition of solids. In no case hold sample more than 7 days. Bring samples to room temperature before analysis.

7. Equipment and Supplies

7.1 Drying Tins: Aluminum.

7.1.1 140mL capacity: For use with 90mm filter disks (Section 7.2)

7.2 Glass-Fiber Filter Disks: 90mm diameter, without organic binder, Type A/B, size 1.0um; use with drying tins from Section 7.1.

Whatman Grade 934-AH ready-to-use (RTU) filters must be used : part# 9907-90 for 90 mm filters

Note: 7.2.1 Regular TSS method: Ready to use filters (RTU) can be used immediately from the package, and the preweighed vendor weight can be used for the analysis.

7.2.2 TSS-LOW method: Ready to use filters (RTU) must be prepped first in the 103 -105 C oven for a minimum of 2 hours, followed by proper dessciation, prior to manual weighing on a 5-digit balance. **Filtration Apparatus:** With a membrane filter funnel.

7.3 Side-arm Flask: Of sufficient capacity for sample size selected.

7.4 Dessicator: With a dessicant containing a color indicator of moisture concentration.

7.5 Drying Oven: For operation at 103 - 105ºC.

7.6 Analytical Balance: Capable of weighing to 0.1mg. % digit balances (capable of weighting 0.01 mg) must be used for TSS-LOW method.

7.7 Magnetic Stirrer: With TFE stirring bar.

7.8 Wide-bore Pipets: Various volumes.

7.9 Graduated Cylinders: Glass or plastic at 100mL, 500mL, 1000mL volumes.

7.10 Muffle Furnace: Capable of 500°C.

7.11 Porcelain Crucibles

7.12 Tweezers: Flat tip, non-piercing.

7.13 Wax crayon or Sharpie pen

7.14 Vacuum Pump for use at filtration station

7.15 Volumetric flasks, 1000mL

8. Reagents and Standards

8.1 Reagent Water: Deionized (DI) water.

8.2 Universal Solids Standard: Environmental Express part #F95005H , #F95005UH or equivalent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Filter one blank of 1000mL of DI water per batch of 20 samples or less. Results must be less than the Reporting Limit (RL). If Blank results are less than 10 times the RL and the sample results are greater than 10 times the RL, the data is reported. If Blank results are greater than 10 times the RL, and there is sufficient sample volume remaining, the entire batch is reset and reanalyzed. A narrative is included with the report.

9.2 Laboratory Control Sample (LCS)

Prepare standard (8.2) according manufacturer specification using 1L volumetric flask. Split 1L into five 250ml containers, using 200ml portions; mix standard well before every measurement.

Label each of the containers, making sure that ID Number and exact concentration is written on each container. Standard will expire in 7 days after preparation.

Filter one LCS standard (200ml) per batch of 20 samples or less. The results of the LCS and must be within ± 20% of the true value. If criteria is not met, then entire batch of samples has to be reset.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Not Applicable.

9.6 Laboratory Duplicate

Filter one sample in duplicate per batch of 10 samples or less. Duplicate determinations should be within in-house control limits. Current control limits are $\leq$ 11% and may be re-evaluated. If acceptance criteria are not met, and there is sufficient sample volume remaining and sample is within holding time, the sample is reset. Otherwise, the data is submitted a narrative is included with the final report.

Additionally, the average between sample and duplicate tare-corrected weights in g must be calculated. Duplicate determinations should agree within 5% of their average weight. This calculation has to be performed on excel format.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Prepare the filters.
- Weigh the clean filters, and/or drying tins.
- Filter the appropriate volumes of sample, and QC samples.
- Rinse the filter with DI water.
- Transfer the filter into to its corresponding drying tin.
- Total Suspended Solids are dried for a minimum of 2 hours in a 103 – 105 °C oven.
- Cool in a dessicator until temperature is constant.
- Weigh the filters until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.
- Total Volatile Suspended Solids are also dried for a minimum of 2 hours in a 500 °C muffle furnace.
- Cool in a dessicator until temperature is constant.

- Weigh the filters until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.
- Calculate results.

Note: For regular TSS method (not TSS-LOW) use prepped filters (RTU filters). Record filter weight provided by the vendor.

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of Glass-Fiber Filter Disk

10.1.1.1 Attach a side-arm flask to the filtration apparatus.

10.1.1.2 Insert a glass-fiber filter disk (Section 7.2) with the wrinkled side up into the filtration apparatus.

Note: Record the manufacturer and lot number of the filter in the batch computer worksheet, when generated.

10.1.1.3 Apply the vacuum, and wash the disk with three successive 20mL portions of DI water.

10.1.1.4 Continue the suction to thoroughly remove all traces of water, and discard washings.

10.1.1.5 Using tweezers remove the filter from filtration apparatus and transfer to a drying tin (Section 7.1).

10.1.1.6 Dry the filter and tin in an oven at 103-105ºC for at least two hours.

Note: For TVSS filters must be pre-washed and dried at 500ºC for at least 2 hours.

10.1.1.7 Cool in a dessicator to a constant temperature, and weigh. Record the weights on the computer worksheet. Print a copy of the worksheet for later use in Section 10.3.

10.1.1.8 The dried filters may be used immediately, or stored in a dessicator until needed. Proceed to Section 10.3.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Label drying tins with sample IDs. At the beginning of analysis, record the date/time/analyst's initials in the computer worksheet. Batch samples in groups of 10 or less, for each product, and enter the associated WG# into each computer worksheet. The worksheet is then ready for use when sample filtration begins (from Section 10.3.4).

10.3.2 Assemble filtering apparatus with a prepared filter from Section 10.1.1, attach a side-arm flask and apply vacuum. Wet filter with a small volume of DI water to seat it

10.3.3 Selection of Sample Sizes

Choose a sample volume to yield between 10 and 200mg dried residue. If more than 10 minutes are required to complete filtration, it is necessary to use another prepped filter (from Section 10.1.1) with a decreased sample volume.

If Total Dissolved Solids are to be determined, collect the filtrate into a clean side-arm flask. Refer to SOP/2219.

10.3.3.1 **Clean samples:** Shake sample container to ensure a well-mixed solution prior to pouring. Use a graduated cylinder to measure sample volume. Begin with a sample volume of 100mL. Continue filtering successive volumes of 100mL until the desired volume of 1000mL is reached. However, if the filtration begins to slow in efficiency during this time, do not add any more additional sample.

10.3.3.2 **Samples containing sediment or other material:** Begin with a smaller sample volume. (An initially large volume of these types of samples will likely clog the filter and require reanalysis.)

10.3.3.2.1 These types of samples require initial stirring on a magnetic stirrer. While stirring, withdraw a volume of sample using a wide-bore pipette (Section 7.9). Pipette the sample onto the filter, and then while holding the pipette over the filter, rinse the pipet with DI water and filter this rinseate as well. This will ensure that all of the solid material is transferred to the filter.

10.3.3.3 **Sludge samples:** These samples are always pipetted to the filter as stated above in Section 10.3.3.2.1. A small sample size is desirable to prevent crust formation and filter curling during the drying step of the procedure.

10.3.4 Write the volume of sample filtered on the computer worksheet that was printed in Section 10.1.1.7. next to the corresponding sample number, along with any necessary comments regarding matrix interference, limited volume, etc.

10.3.5 Wash with three successive 10mL volumes of DI water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete.

10.3.6 Samples with high dissolved solids may require additional washings.

10.3.7 Using tweezers carefully remove the filter from filtration apparatus and transfer to the corresponding, labeled drying tin.

10.3.8 Place the filter in the tin into a 103-105ºC oven to dry for a minimum of 2 hours. Record the date, time in and the oven temperature in the computer worksheet.

10.3.9 After drying, remove tin from the oven to cool in a dessicator to a constant temperature. Record the date, time out and the oven temperature in the computer worksheet.

10.3.10 Weigh the tin on an analytical balance. Record the weights on the computer worksheet, and the sample volumes filtered as written down from Section 10.3.3. Enter the sample volumes from the logbook onto the computer worksheet discussed in Section 10.1.1.7.

10.3.11 Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less. Record each weight on the computer worksheet.

10.3.12 Total Volatile Suspended Solids

10.3.12.1 If TVSS analysis is requested, using tweezers, transfer the filter from the drying tin to a labeled crucible.

10.3.12.2 Place the crucible into a 500ºC muffle furnace for a minimum of 2 hours, and record the date, time in, and the oven temperature in the computer worksheet.

Following removal from muffle furnace, place in a dessicator to cool to a constant temperature. Record the date, time out and the oven temperature in the computer worksheet.

10.3.12.3 Using an analytical balance, tare a clean drying tin. Remove the filter from the crucible with tweezers and transfer the filter to the drying tin.

10.3.12.4 Record the weights on the computer worksheet.

10.3.12.5 Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less. Record each weight on the computer worksheet.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

10.5.1 Prior to each use, the filtering apparatus is rinsed thoroughly with DI water.

10.5.2 As needed, the filtering apparatus is washed in the industrial dishwasher.

10.5.3 As necessary, wipe the inside of the filtering apparatus with a paper towel and then rinse thoroughly with DI water

10.5.4 Ensure that there is an adequate supply of oil in the vacuum pump.

10.5.5 Monitor automated data logger system once daily for temperature of oven.

11. Calculations

$$\text{mg TSS / L} = \frac{(\mathbf{A} - \mathbf{B}) \times 1000}{\text{sample volume, (mL)}}$$

where:

A = Final weight (weight of filter + dried residue, mg)
B = Initial weight (weight of clean filter, mg)

$$\text{mg TVSS / L} = \frac{(\mathbf{G} - \mathbf{N}) \times 1000}{\text{sample volume, (mL)}}$$

where:

G = Final Weight at 105 °C (weight of dish + dried residue,mg)
N = Weight at 500 °C (weight of dish + dried residue, mg)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance as outlined in Section 10.5.

Review of blanks and duplicate samples for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. If Laboratory doesn't have sample for re-analysis, project manager has to be notified and data will be release to customer with a narrative.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

Custom standards are used for MDL studies, LOD and LOQ studies.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

Analysis of a weight-bearing, real-world sample is chosen to run four times in duplicate between two analysts (one of which must be a trained analyst), and the results must be within 10%.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

Analysis of a weight-bearing, real-world sample is chosen to run four times in duplicate between two analysts (one of which must be a trained analyst), and the results must be within 10%.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Nitrate, Nitrite and Nitrate/Nitrite Nitrogen

Reference: **Methods 353.2:** Methods for the Determination of Inorganic Substances in Environmental Samples, EPA 600/ R-93/ 100. August, 1993.

Methods 4500NO_3-F, 4500NO_2-B: Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-107-04-1, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1992

1. Scope and Application

Matrices: This method is limited to optically clear water samples with a total concentration of nitrite and nitrate below 8mg N/L.

Definitions: Refer to Alpha Analytical Quality Manual.

In waters and wastewaters, the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea. Numerous concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Total oxidized nitrogen is the sum of nitrate and nitrite nitrogen. Nitrate generally occurs in trace quantities in surface water but many attain high levels in some groundwater. In excessive amounts, it contributes to the illness known as methemoglobinemia in infants. A limit of 10mg nitrate as nitrogen/L has been imposed on drinking water to prevent this disorder. Nitrate is found only in small amounts in fresh domestic wastewater but in the effluent of nitrifying biological treatment plants, nitrate may be found in concentrations of up to 30mg nitrate as nitrogen/L. It is an essential nutrient for many photosynthetic autotrophs and has been identified as a growth-limiting nutrient.

Nitrite is an intermediate oxidation state of nitrogen, both in the oxidation of ammonia to nitrate and in the reduction of nitrate. Such oxidation and reduction may occur in wastewater treatment plants, water distribution systems, and natural waters. Nitrite can enter a water supply system through its use as a corrosion inhibitor in industrial process water. Nitrite is the actual etiologic agent of methemoglobinemia. Nitrous acid, which is formed from nitrite in acidic solution, can react with secondary amines (RR’NH) to form nitrosamines (RR’N-NO), many of which are known to be carcinogens. The toxicologic significance of nitrosation reactions in vivo and in the natural environment is the subject of much current concern and research.

Within this SOP, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3^--N, and nitrite nitrogen as NO_2^--N.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Lachat Analyzer and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color, which is read at 520nm. Nitrite alone can be determined by removing the cadmium column. The nitrate is calculated as the difference between the reduced and non-reduced sample

2.1 Method Modifications from Reference

Soils can be analyzed using 1:10 ratio soil to water extraction, following filtration.

3. Reporting Limits

This method has an analytical range of 0.1 to 8.0mg N/L in the form of nitrate, and 0.05 to 8.0mg N/L in the form of nitrite.

The Reporting Limit is 0.1mg/L for Nitrate and 0.05 mg/L for Nitrite. Reporting limit is 1.0 mg/kg for soils

4. Interferences

4.1 Suspended matter in the column will restrict sample flow.

4.2 For turbid samples, filter through 0.45µm membrane filter prior to analysis.

4.3 Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

4.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. In this case, only the water phase of the sample is used for analysis and a narrative is submitted with the data. Dilutions are performed as necessary.

4.5 Residual chlorine can interfere by oxidizing the Cd column, reducing its efficiency. Prior to analysis, check wastewater and drinking water samples for residual chlorine and record results in the Laboratory Notebook. If residual chlorine is present, and the samples are preserved with H_2SO_4, the sample may be analyzed for NO_3/NO_2 determination. However, NO_2 must be performed by a manual method. If it is not possible to analyze NO_2 by a manual method, the result is reported as NA and a narrative is submitted.

4.6 Sample color interferes if it is absorbed at about 540nm.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles; 250mL minimum volume. Soils can be collected in plastic or glass containers.

6.2 Sample Preservation

Refrigerate samples at 4 ± 2 °C.

For Nitrate/Nitrite analysis, the samples are preserved with 1:1 H_2SO_4.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Begin NO_3^- and/or NO_2^- determinations promptly after sampling. If storage is necessary, store for up to 48 hours at 4 ± 2 °C.

NOTE: If the 48-hour hold time cannot be met, the sample is to be handled as follows, only in an emergency situation. These instructions are not to be used on a regular basis.

Prior to the expiration of the 48-hour hold time, the following three steps are executed:

1. A manually colored Nitrite test is performed by Method 354.2. Results are recorded in the Laboratory Notebook.
2. A 50mL aliquot of the sample is preserved to a pH of <2 with concentrated H_2SO_4. Preservation is recorded in the Laboratory Notebook.

Prior to analysis, within 14 days of preservation, the preserved sample is neutralized using 6N NaOH. The sample is analyzed using only the Lachat Instrument.

CAUTION! Samples must NOT be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column

7. Equipment and Supplies

7.1 Lachat 8000 Automated Ion Analyzer or Lachat QuickChem 8500 Automated Ion Analyzer

7.2 Nitrate+Nitrite Lachat Board

7.3 Nitrite Lachat Board

7.4 Pre-packed Cadmium Columns: Available from Lachat.

7.5 Ottawa sand.

7.6 Disposable Culture Tubes 13x100 ml

7.7 Disposable pipettes.

8. Reagents and Standards

8.1 Stock Nitrate Standard, 1000mg N/L as NO_3^-: Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.1.1 Stock Nitrate Standard, 200.0mg N/L as NO_3^-: Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 1.444g potassium nitrate (KNO_3) in about 600mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at 4±2°C. This solution is stable for six months.

8.2 Stock Nitrite Standard, 1000mg N/L as NO_2^-: Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.2.1 Stock Nitrite Standard, 200.0mg N/L as NO_2^-: Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 0.986g sodium nitrite ($NaNO_2$) or 1.214g potassium nitrite (KNO_2) in approximately 800mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at 4±2°C. This solution is stable for six months.

8.3 Intermediate Nitrate Working Standard, 20 mg N/L as Nitrate: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_3^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at 4±2°C.

8.4 Intermediate Nitrite Working Standard, 20 mg N/L as Nitrite: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_2^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at 4±2°C.

8.5 Set of Six Calibration NO_3^- Standards, 8.0, 4.0, 1.00, 0.40, 0.20 and 0.1mg N/L as Nitrate: These standards are stable for 2 weeks. Refrigerate at 4±2°C.

To four 200mL volumetric flasks, add respectively: 8.0, 4.0, 1.0 and 0.4mL of the 200mg N/L NO_3^- stock standard. Bring to volume with DI water.

To two 200mL volumetric flasks, add respectively: 2.0 and 1.0mL of the 20mg N/L NO_3^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared. If an autodiluter is used then it must be checked in an analytical tray by autodiluting 8.0mg N/L as Nitrite. The recovery for NO2 must be within 10% of the true value.

8.6 Set of Six Calibration NO_2^- Standards, 8.0, 4.0, 1.00, 0.40, 0.10 and 0.05mg N/L as Nitrite: These standards are stable for 2 weeks. Refrigerate at 4±2°C.

To three 200mL volumetric flasks, add respectively: 8.0, 4.0 and 1.0 of the 200mg N/L NO_2^- stock standard. Bring to volume with DI water.

To three 200mL volumetric flasks, add respectively:4.0, 1.0mL and 0.5mL of the 20mg N/L NO_2^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared.

8.7 Ammonium Chloride Buffer, pH 8.5: In a 2L volumetric flask, dissolve 170g ammonium chloride (NH_4Cl) and 2.0g disodium ethylenediamine tetraacetic acid dihydrate ($Na_2EDTA \bullet 2H_2O$) in about 800mL water. Dilute to the mark with DI water and invert to mix. Adjust the pH to 8.5 with concentrated ammonium hydroxide. This solution is prepared monthly and stored at room temperature.

8.8 Sulfanilamide Color Reagent: To a 2L volumetric flask add about 1200mL water. Then add 200mL of 85% phosphoric acid (H_3PO_4), 80.0g sulfanilamide, and 2.0g N^-(1-naphthyl)ethylenediamine dihydrochloride (NED). Shake to wet, and stir to dissolve for 30 minutes. Dilute to the mark with DI water and invert to mix. Store in a dark bottle. This solution is stable for one month. Store at room temperature.

8.9 200ppm Nitrate Stock Standard, (for ICV/LCS): Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at 4±2°C. Expires six months from preparation or upon manufacturer's expiration date.

8.10 200ppm Nitrite Stock Standard: Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at 4±2°C. Expires six months from preparation or upon manufacturer's expiration date.

8.11 Initial Calibration Verification Standard (ICV)/Laboratory Control Sample (LCS): Store refrigerated at 4±2°C. Expiration is 2 weeks from date of preparation.

8.11.1 Nitrate LCS, 5.0ppm: Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.

8.11.2 Nitrate ICV, 0.5ppm: Pipet 10.0mL of 5.0ppm standard (Section 8.11.1) into a 100mL volumetric flask and bring to volume with DI.

8.11.3 Nitrite LCS, 5.0ppm: Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.

8.11.4 Nitrite ICV, 0.5ppm: Pipet 10.0mL of 5.0ppm standard (Section 8.11.3) into a 100mL volumetric flask and bring to volume with DI.

8.12 DPD Free Chlorine Reagent Powder Pillows: HACH brand, for 25mL sample. Store at room temperature. Expires upon manufacturer's expiration date.

8.13 1N Hydrochloric acid (HCL): To a 1L volumetric flask add about 600mL DI. Then add 83mL of concentrated hydrochloric acid (HCL) Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.

8.14 1N Sodium Hydroxide (NaOH): To a 1L volumetric flask add about 600mL DI. Then add 40 g of sodium Hydroxide. Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

8.15 6N Sulfuric Acid (H2SO4): To a 1L volumetric flask add about 600mL DI. Then add 140 ml of concentrated Sulfuric Acid (H2SO4). Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

One Method Blank is analyzed per batch of 20 samples or less The Method Blank consists of DI.

For soils: 5g of Ottawa sand extracted with 50 ml of DI. Results must be < 0.1mg/L. If this criterion is not met, the blank is re-analyzed. If there is still failure, the problem must be found and corrected prior to any sample analysis

9.2 Laboratory Control Sample (LCS)

The 5ppm ICV is reported as the LCS for the batch. See Section 9.3.

For soil LCS: 5g of Ottawa sand extracted with 0.25 ml of 1000 mg/l nitrate (8.1) (or 1000 mg/l Nitrite standard (8.2)) and 50 ml DI. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. LCS recoveries must be recovered within ± 10% of the true value. If these criteria are not met, LCS's must be re-analyzed. If failure continues, the batch has to be re-extracted and re-analyzed.

9.3 Initial Calibration Verification (ICV)

Two ICVs are analyzed at the beginning of the analytical sequence. One is at a concentration of 0.5ppm, and the other is at a concentration of 5.0ppm.

Both must be recovered within ± 10% of the true value. If these criteria are not met, the ICVs must be re-analyzed. If failure continues, the ICVs are to be re-made and/or a new calibration curve is to be generated.

The 5ppm ICV is reported as the LCS for the batch.

9.4 Continuing Calibration Verification (CCV)

At the beginning of the first tray, after every ten samples and at the end of every analytical sequence, a CCV and a CCB pair must be analyzed to verify both calibration curves.

1.0ppm Nitrate CCV (Section 8.5)

1.0ppm Nitrite ICV (Section 8.6)

Calibration Blank (DI)

The results of the CCVs must be within ±10% of the true value, otherwise re-calibration is required.

The results of the CCBs must be less than our standard limit of detection, otherwise the analysis is stopped and the problem corrected.

9.5 Matrix Spike

One Matrix Spike is analyzed per batch of 20 samples or less. Separate spikes are performed for Nitrate and Nitrite. In a 25mL volumetric flask, 0.5mL of 200ppm stock calibration standard (Section 8.1 or 8.2) is added to the sample. The final concentration of the matrix spike is

4.0ppm. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

For soils: weigh 5.0 g of sample, add 2.0 ml of 200 mg/l Nitrate or Nitrite standard and 48 ml of DI. The final concentration of the matrix spike is 80.0 mg/kg. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

% Recovery for the Matrix Spike must be within in-house control limits. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

Note: For samples, analyzed by method **353.2** (NO2-353 and NO3-353) maximum batch size is 10 samples; every 10 samples required separate matrix spike (MS) to be analyzed. % Recovery for the Matrix Spike must be within +/- 10% of true value. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

One Duplicate sample is analyzed per batch of 20 samples or less. A separate aliquot of the sample is analyzed for this purpose.

% RPD for the Duplicate must be within in-house control limits. If acceptance criteria are not met, the Duplicate is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Calibration
- ICV/LCS – both levels
- Sample analysis
- CCV – every ten samples and at the end of the analytical sequence

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation

10.1.1.1 Place the Nitrate+Nitrite board (containing the cadmium column) in Channel 1. Place the Nitrite board in Channel 2. Make sure the valve to the cadmium column is closed prior to starting to pump the reagents.

10.1.1.2 Commence pumping of reagents.

10.1.1.3 Once the lines are full of reagent and free of gas bubbles, open the valve to allow reagent to flow through the cadmium column.

NOTE: Be sure to switch the valve back before rinsing the manifold with DI water at the completion of the run.

NOTE: DO NOT LET AIR ENTER THE CADMIUM COLUMN.

10.1.2 Column Efficiency Procedure

10.1.2.1 Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column or any change in the cadmium surface characteristics, (cadmium granules should be dark gray). If air bubbles are present in column, connect the column into the manifold, turn the pump on maximum and tap firmly with a screwdriver handle, being careful not to break the column, working up the column until all air is removed. If air cannot be removed, the column should be repacked. Cadmium columns should be stored filled with buffer. If air enters the column, efficiency will decrease. Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green pump tube. Pump buffer through the packed column and collect in a graduated cylinder. The flow rate with the column connected should be greater than 4.0 mL/minute.

10.1.2.2 **Column Efficiency – Slope Ratio Method:** Calibrate with the mid-range NO_3–N standards. Calibrate with a matching concentration range of NO_2–N standards. The column efficiency is determined by the equation:

$$E = \frac{S_{NO3\text{-}N}}{S_{NO2\text{-}N}} \times 100$$

where:

$S_{NO3\text{-}N}$ = slope of NO_3 calibration
$S_{NO2\text{-}N}$ = slope of NO_2 calibration
E = % efficiency

10.1.2.3 **Column Efficiency – Concentration Ratio Method:** Calibrate with the mid-range NO_2-N and NO_3-N standards. Run a known concentration NO_2-N standard. Run a matching concentration NO_3-N standard. The column efficiency is determined by the following equation:

$$E = \frac{C_{NO3\text{-}N}}{C_{NO2\text{-}N}} \times 100$$

where:

$C_{NO3\text{-}N}$ = concentration of NO_3 standard
$C_{NO2\text{-}N}$ = concentration of NO_2 standard
E = % efficiency

10.1.2.4 **Column Efficiency Result:** If the efficiency is <75%, the column is repacked. All results are recorded and maintained on file in the QC department.

10.1.3 Residual Chlorine Screening

Check all wastewater and drinking water samples for residual chlorine prior to analysis.

10.1.3.1 Add 1 DPD Free Chlorine powder pillow (Section 8.12) to 25mL of sample in a centrifuge tube. An immediate color change to pink indicates residual chlorine is present. If residual chlorine is present, add a small amount of ascorbic acid to a sample aliquot (record this in logbook) and check for residual chlorine presence again. If residual chlorine remains, notify the Department Manager and/or the Laboratory Director. Results will be reported as Not Applicable (N/A).

If residual chlorine is not present, continue with sample analysis.

10.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

10.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has seven calibration points. The correlation coefficient of each curve must be $\geq$0.995, otherwise recalibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NO_3+NO_2 as N and NO_2 as N concentrations in standards.

10.2.1.1 Channel 1 is used to generate a calibration curve for Nitrate/Nitrite ranging from 0 to 8.0ppm.

10.2.1.2 Channel 2 is used to generate a calibration curve for Nitrite ranging from 0 to 8.0ppm.

Note: Instrument is calibrated daily, fixed calibration range is used; linearity is verified daily; three standards are used for linear calibration verification (low ICV (0.5 mg/l), High ICV (5.0 mg/l) and CCV (1.0 mg/l)). All standards must be within 10% of true value

10.2.2 Initial Calibration Verification (ICV)

10.2.2.1 Prior to sample analysis, the following ICVs must be analyzed to verify both calibration curves.

10.2.2.1.1 Nitrate ICV, 0.5ppm (Section 8.12.2)

10.2.2.1.2 Nitrate ICV, 5.0ppm (Section 8.12.1)

10.2.2.1.3 Nitrite ICV, 0.5ppm (Section 8.12.4)

10.2.2.1.4 Nitrite ICV, 5.0ppm (Section 8.12.3)

10.2.2.2 The results must be within $\pm$10% of the true value, otherwise re-calibration is required.

10.3 Equipment Operation and Sample Processing

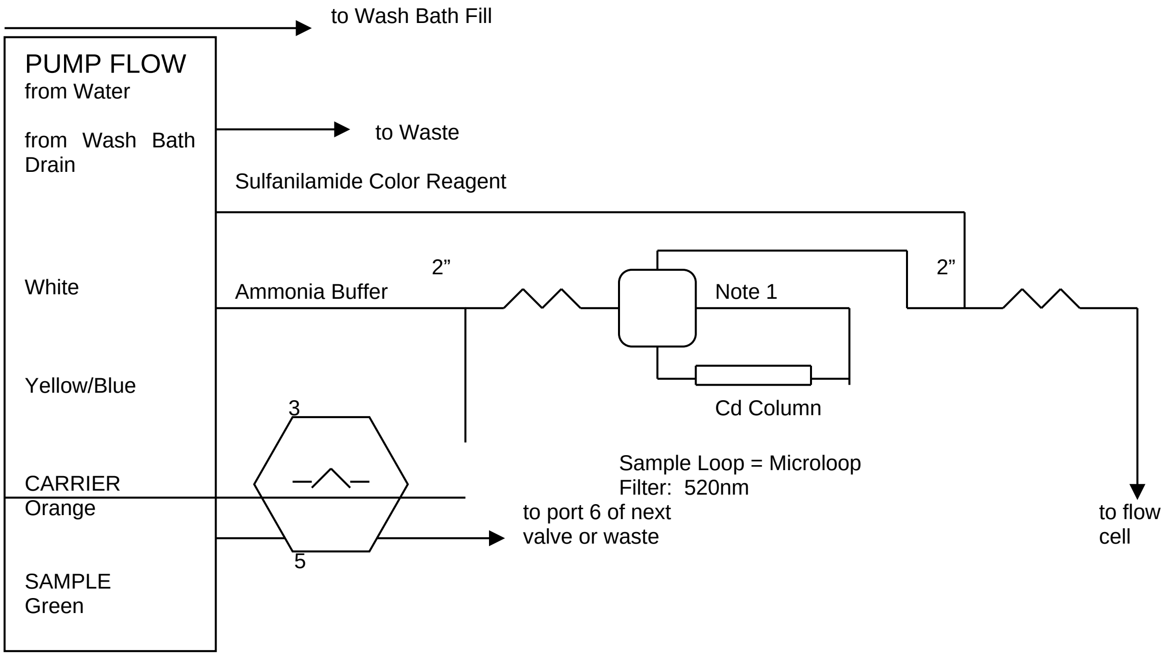
Follow the manufacturer's directions for the operation of the Lachat 8000.

All samples have to be inspected prior to analysis. Samples that are turbid or have sediment have to be filtered prior to analysis.
Check pH of the samples. If pH is less then 5 or greater then 9, then adjust pH using 1N Hydrochloric Acid (HCl) (8.13), 6N Sulfuric Acid (H2SO4) (8.15) or 1N Sodium Hydroxide (NaOH) (8.14). Record pH adjustment in the log book.

For soils: extract soils samples prior to analysis: take 5g of sample, add 50 ml of DI, extract for 30 min, then filter thorough 0.45 nm filter. Record all weights for calculations.

Note: if samples are filtered, then Method blank also have to be filtered

The Manifold Diagram follows:



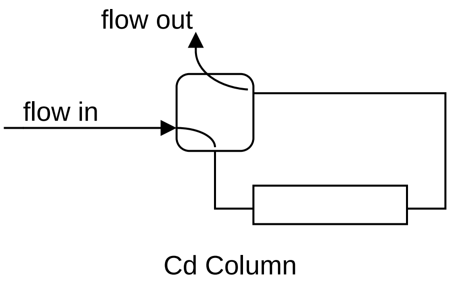
CARRIER is water.

2” is 135cm of tubing on a 2-inch coil support.
APPARATUS: Standard valve, flow cell, and detector head modules are used.

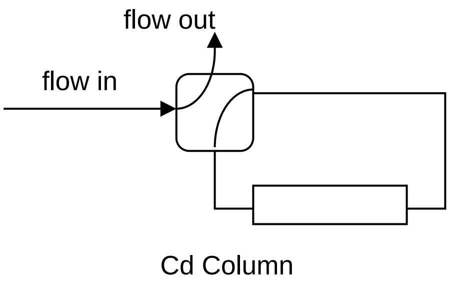
All manifold tubing is 0.8mm (0.032”) i.d. This is 5.2µL/cm.

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

State 1: Nitrate + Nitrite



State 2: Nitrite Only



10.4 Continuing Calibration

See Section 9.4

10.5 Preventive Maintenance

Tubing is changed monthly or as needed.

At the end of each analytical sequence, the valve to the column is closed. DI is rinsed through the Lachat for five minutes followed by five minutes of air.

All maintenance is documented in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 **Nitrate/Nitrite:** When the software is set up according to the manufacturer's recommendations, the concentration of nitrate plus nitrite in mg NO_3/NO_2-N/L is reported directly when the Cd column is included in the sample train in Channel 1.

11.2 **Nitrite:** When the software is set up according to the manufacturer's recommendations, the concentration nitrite in mg NO_2-N/L is reported directly when the Cd is not included in the sample train in Channel 2.

11.3 **Nitrate:** The concentration of nitrate is determined by the subtraction of the nitrite concentration, (Section 11.2 above), from the nitrate-nitrite concentration, (Section 11.1 above).

11.3.1 If the sample was preserved initially as described in Section 6.3, subtract the Nitrite value generated manually from the Nitrate/Nitrite value generated by the Lachat Instrument. This value is reported as the Nitrate result.

When the sample is preserved initially as described in Section 6.3, the value generated by the Lachat instrument for Nitrite is invalid and therefore disregarded.

11.4 If any sample exceeds a concentration of 8.0 mg/L, the sample must be diluted and re-analyzed. All sample concentrations must fall within the calibration curve.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

None.

Nitrogen Ammonia

Reference: **SM4500 NH_3-BH,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 350.1, Methods for the Chemical Analysis of Water and Wastes, EPA 600/ 4-82-055, Revision 2.0, August 1993.

Method 10-107-06-1-A, Methods for Automated Ion Analyzers, May 20, 1998.

1. Scope and Application

Matrices: Ammonia nitrogen can be determined in potable, surface, and saline waters as well as domestic and industrial wastewaters and in soils.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Tecator and/or Lachat Instrument and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

In waters and wastewaters the forms of nitrogen of greatest interest are, in order of decreasing oxidation state: nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons. Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea, and numerous synthetic organic materials. Typical organic nitrogen concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Ammonia is present naturally in surface and wastewaters. Its concentration generally is low in groundwaters because it adsorbs to soil particles and clays and is not leached readily from soils. It is produced largely by deamination of organic nitrogen containing compounds and by hydrolysis of urea. At some water treatment plants ammonia is added to react with chlorine to form a combined chlorine residual.

In the chlorination of wastewater effluents containing ammonia, virtually no free residual chlorine is obtained until the ammonia has been oxidized. Rather, the chlorine reacts with ammonia to form mono- and dichloramines. Ammonia concentrations encountered in water vary from less than 10µg ammonia nitrogen/L in some natural surface and groundwaters to more than 30mg/L in some wastewaters.

In this discussion, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3-N, nitrite nitrogen as NO_2-N, and ammonia nitrogen as NH_3-N.

2. Summary of Method

The sample is buffered at pH 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds. It is distilled into a solution of boric acid. The ammonia in the distillate is determined colorimetrically by the phenate method.

The phenate method is base 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 product is measured at 630nm, and is directly proportional to the original ammonia concentration

2.1 Method Modifications from Reference

Method was modified to include soil sample distillation. Samples are buffered at pH 9.5. pH is checked using pH paper 7.5-14 (not pH meter).

3. Reporting Limits

The RL is determined to be 0.075mg/L based on a 100mL sample size for waters and 7.5 mg/kg based on 1g of soil material used for distillation.

4. Interferences

4.1 Most of the following interferences refer to the direct analysis of a water sample. The distillation steps remove most of these interferences. Residual chlorine must be removed prior to distillation by pretreatment of the sample with sodium thiosulfate.

4.2 Samples with a high concentration of NH_3 may carry-over into the next sample and therefore yield false high results in that next sample. If a sample with a low concentration follows a sample with a high concentration, re-analyze the low sample to ensure results are accurate.

4.3 Glycine, urea, glutamic acid, cyanates, and acetamide hydrolyze very slowly in solution on standing but, of these, only urea and cyanates will hydrolyze on distillation at pH of 9.5. Hydrolysis amounts to about 7% at this pH for urea and about 5% for cyanates. Some of the organic compounds that may interfere, such as formaldehyde, may be eliminated by boiling off at a low pH before nesslerization.

4.4 Color and turbidity of the sample distillate may interfere with analysis. These interferences are removed by manual filtration of the distillate prior to Lachat analysis, or by dilution of the sample prior to distillation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Phenol used in this method is hazardous and general laboratory safety practices must be observed.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

The most reliable results are obtained on fresh samples. Use plastic or glass containers.

6.2 Sample Preservation

If prompt analysis is impossible, preserve samples with approximately 0.8mL concentrated H_2SO_4/L sample. The pH of the acid-preserved samples should be <2. (Some wastewaters may require more concentrated H_2SO_4 to achieve this pH.) Store samples at 4ºC.

Soils samples are received without preservation and stored at 4ºC

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples (both waters and soils) are analyzed within 28 days after collection.

7. Equipment and Supplies

7.1 **Distillation apparatus:** Tecator Instruments Automatic Distillation Unit. Follow the instrument manufacturer's instructions for proper operation.

7.2 **pH Strips:** Range 7.5 to 14 units.

7.3 **250mL Disposable Polypropylene cups with covers.**

7.4 **Automated Ion Analyzer:** Lachat Instruments.

7.5 **Helium gas, He:** 140kPa (20 lb/in^2).

7.6 **Volumetric flasks,** various volumes.

7.7 **Pipets,** various volumes.

7.8 **Analytical Balance**

7.9 **Centrifuge Tubes:** 50mL volume.

7.10 **Potassium Iodide Starch Paper**: Fisher brand 14-860

8. Reagents and Standards

8.1 **Borate buffer solution:** Add 88mL 0.1N NaOH solution to 500mL of approximately 0.025M sodium tetraborate ($Na_2B_4O_7$) solution (9.5g $Na_2B_4O_7 \cdot 10\ H_2O$/L) and dilute to 1L with DI water. Store at room temperature. Expires one month from date of preparation.

8.2 **Sodium thiosulfate (dechlorinating reagent):** Dissolve 0.35g $Na_2S_2O_3 \cdot 5H_2O$ in DI water and dilute to 100mL. Prepare fresh daily as needed.

8.3 Neutralization agents: Prepare with DI water:

8.3.1 Sodium hydroxide, NaOH, 6N: In a 1L volumetric flask, carefully mix 240g of NaOH (Section 8.11) pellets with 500mL DI water. Dissolve and bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.3.2 Sulfuric acid, H_2SO_4, 10%: To a 1L volumetric flask, add 500mL DI water. Carefully add 100mL of concentrated Sulfuric Acid (Section 8.12). Bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.4 Sodium Phenolate: **CAUTION!** Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin. In a 1L volumetric flask, dissolve 88mL of 88% liquified phenol or 83g crystalline phenol (C_6H_5OH) in approximately 600mL DI water. While stirring, slowly add 32g sodium hydroxide (NaOH). Cool, dilute to the mark, and invert three times. Do **not** degas this reagent. Store at room temperature. Expires one month from date of preparation.

8.5 Sodium Hypochlorite (approximate 2.6%): In a 500mL volumetric flask, dilute 250mL Regular Chlorine bleach [5.25% sodium hypochlorite (NaOCl)] to the mark with DI water. Invert three times to mix. Store at room temperature. Expires one month from date of preparation.

8.6 Sodium Nitroprusside (coloring agent): In a 1L volumetric flask, dissolve 3.50g sodium nitroprusside (Sodium Nitroferricyanide [$Na_2Fe(CN)_5NO_2 \cdot H_2O$]) dilute to the mark with DI water. Degas with helium to prevent bubble formation. Use He at 140kPa (20 lb/in^2) through a helium degassing tube. Bubble He vigorously through the solution for one minute. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.7 Boric 1.5% Boric Acid Solution: To a 1000mL volumetric flask add 15g Boric Acid. Dilute to the mark with DI water. Store at room temperature. Expires one month from date of preparation.

8.8 0.2% Boric Acid Solution (Carrier Solution): To a 2L volumetric flask, dissolve 4g Boric Acid (H_3BO_3) in DI water. Degas by bubbling vigorously with Helium for one minute. Store at room temperature. Expires one month from date of preparation.

8.9 Stock Standard, 1000ppm as NH_3 (for calibration and spike solutions): Commercially prepared. Certificate of analysis is required. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.9.1 Intermediate Calibration Stock Standard, 100ppm as NH_3: To a 100mL volumetric flask, add 10.0mL of Stock Standard (Section 8.9) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.9.1.1 Nine Working Calibration Standards: The following standards are prepared in volumetric flasks fresh each day of use:

8.9.1.1.1 20.0ppm: 40mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.2 10.0ppm: 20mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.3 **8.00ppm:** 8mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.4 **4.00ppm:** 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.5 **2.00ppm:** 2mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.6 **1.00ppm:** 1mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.7 **0.400ppm:** 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.8 **0.200ppm:** 1mL of 20ppm standard (Section 8.9.1.1.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.1.9 **0.100ppm:** 1mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.1.10 **0.050ppm:** 20mL of 0.100ppm standard (Section 8.9.1.1.8) to 40mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.2 Continuing Calibration Standards:

8.9.1.2.1 **0.400ppm Low CCV:** 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.2.2 **4.00ppm Hi CCV:** 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.2 **Spiking solution, 200ppm as NH_3:** To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.9) and dilute to the mark with DI water.

8.10 Stock Standard Solution, 1000ppm as NH_3 (for ICV and LCS): Commercially prepared. Certificate of analysis is required. This must be from a different source than that used for Stock Standard (Section 8.9). Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.10.1 Initial Calibration Verification Standards (ICV):

8.10.1.1 **Hi ICV, 10ppm:** To a 100mL volumetric flask add 1mL of 1000ppm standard (Section 8.10). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.10.1.2 **Hi ICV, 8.0ppm:** To a 100mL volumetric flask add 0.8mL of 1000ppm standard (Section 8.10). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation

8.10.1.3 Low ICV, 1.0ppm: To a 100mL volumetric flask add 10mL of 10ppm ICV (Section 8.10.1.1). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.10.2 LCS solution, 200ppm as NH_3: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.10) and dilute to the mark with DI water. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.11 NaOH Pellets: ACS Grade. Store well sealed at room temperature. This salt does not have an expiration date.

8.12 Sulfuric Acid (Concentrated): Store at room temperature. Expires upon manufacturer's specified date. Use extreme caution when handling.

8.13 Ottawa sand: store at room temperature. Expires upon manufacturer's specified date.

8.14 Thymol Blue Sodium Salt 0.04% water solution: used as indicator during pH adjustment prior distillation. Store at room temperature. Expires upon manufacturer's specified date.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 The Distillation Blank is 100 mL DI for waters or 1g of Ottawa sand for soils. Distill one per batch of 20 samples or less. Results for the Distillation Blank must be less than the Reporting Limit. If results for the Distillation Blank are greater than the reporting limit, and all sample results for the batch are greater than 10x the Blank result, sample results may be reported with a narrative to be included on the final report. Otherwise, all samples must be redistilled and reanalyzed.

9.1.2 The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) for the Lachat analysis are not distilled and are 0.2% Boric Acid Solution (Section 8.8).

The ICB is run after the initial calibration verification standards (ICV) and CCB is run after the continuing calibration standards (CCV).

If results for the ICB are greater than the reporting limit, the ICB is reinjected. If failure continues the instrument must be recalibrated. Samples are not to be analyzed until an acceptable ICB is obtained.

If results for the CCB are greater than the reporting limit, the CCB is reinjected. If failure continues the instrument must be recalibrated. All samples that were analyzed since the last passing CCB have to be reanalyzed.

9.2 Laboratory Control Sample (LCS)

9.2.1 **Distillation:** Distill a Low and a Hi LCS with each batch of 20 samples or less. Recovery of the LCSs must be within +/- 10% of true value for method 350.1 for waters, and within control charts for method 4500 and soils. If recovery is outside of this range, the standard is reanalyzed once. If recovery still fails criteria, the entire sample batch must be redistilled and reanalyzed.

9.2.1.1 **Low 2ppm LCS:** Add 1mL of 200ppm LCS solution (Section 8.10.2) to

100mL DI for waters or for 1.0g of Ottawa sand for soils. This is used for the Low 0 – 2ppm curve

9.2.1.2 **Hi 20ppm LCS:** Add 10mL of 200ppm LCS solution (Section 8.10.2) to 100mL DI for waters or to 1.0g of Ottawa sand for soils. This is used for the Hi 2-20ppm cuve.

9.3 Initial Calibration Verification (ICV)

9.3.1 **Lachat Analysis:** The ICVs are not distilled. Analyze the following Hi and Low ICVs after calibration of the Lachat instrument. Recoveries must be within 10% of the true value. If results are outside of this range, the ICV may be reinjected once. If failure continues, recalibration of the instrument is necessary. Samples are not to be analyzed until acceptable ICV results are obtained.

9.3.1.1 **Low ICV, 1.0ppm** (Section 8.10.1.2)

9.3.1.2 **Hi ICV, 10ppm (**Section 8.10.1.1). **HI ICV** will be **8.0** ppm (Section 8.10.1.2) in case of one board calibration.

9.4 Continuing Calibration Verification (CCV)

9.4.1 **Lachat Analysis:** The CCVs are not distilled. Analyze the following Low and Hi CCVs after every ten samples and at the completion of analysis. Recoveries must be within 10% of the true value.

If recoveries fall outside of this range, the CCV is reanalyzed. If recovery is acceptable, the analysis may continue. If recovery is again unacceptable, the cause for the failure is determined and corrected, and the instrument is recalibrated. All samples that were analyzed since the last CCV that was within range are reanalyzed.

9.4.1.1 **0.4ppm Low CCV** (Section 8.9.1.2.1)

9.4.1.2 **4.0ppm Hi CCV** (Section 8.9.1.2.2)

9.5 Matrix Spike

One matrix spike is prepped and analyzed per batch of **20 samples** or less for **method 4500**. Matrix spike has to be analyzed **for every 10 samples** or **less for method 350.1**. Prior to distillation, use 2mL of 200ppm Intermediate Stock Standard (Section 8.9.2) per 100mL of sample for waters or 1.0g of sample for soils. Recovery of the Matrix Spike must be within +/- 10% of true value for waters and within control charts for soils. If the recovery is outside of acceptance criteria, and the LCS is within acceptance criteria, matrix interference may be assumed. The associated data is reported with a narrative to be included on the final report.

9.6 Laboratory Duplicate

Distill one duplicate sample per batch of 20 samples or less. The %RPD for the duplicate sample must be within Control Chart limits. If results are outside of the acceptance criteria, a narrative to be included on the final report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

9.8.1 Distillation Sequence

3 Rinse tubes
Blank
Low LCS
Hi LCS
Rinse
Samples
Rinse after suspected high samples
Duplicate
Spike
Rinse
Shut-Down

9.8.2 Lachat Analytical Sequence

Instrument Calibration
CCV: Low, Hi,CCB
Low ICV
Hi ICV
IC Blank
Samples
CCV: Low, Hi, CC Blank every 10 samples and at completion of analysis
Shut-Down

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to analysis, organize samples in order from low to high odor.

10.1.2 Clean 250mL Tecator tubes by rinsing twice with approximately 0.5mL of 6N NaOH solution and 100mL RO water. Rinse twice again with DI water.

10.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer’s instructions.

10.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has 7 calibration points. The correlation coefficient of each curve must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

10.2.1.1 Channel 1 is used to generate a calibration curve on the low range from 0.00 – 2ppm.

10.2.1.2 Channel 2 is used to generate a calibration curve on the high range from 0 – 20ppm.

Alternative method: One board can be used to calibrate the Lachat instrument. !0 point calibration curve will be used with calibration standards 10.0, 8.0, 4.0, 2.0, 1.0,04, 0.2, 0.1, 0.05 mg/l each and blank. The correlation coefficient must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.2.2 Initial Calibration Verification (ICV)

Prior to sample analysis, an ICV is analyzed at 1.0ppm to verify the low calibration curve on Channel 1. Another ICV is analyzed at 10ppm to verify the high calibration curve on Channel 2. Both ICVs must yield results ± 10% of their true value, otherwise re-calibration is necessary.

Note: if instrument is calibrated using one board calibration, then both ICV's (Low and High), will be evaluated and high ICV will be 8.0 mg/l ICV standard (Section 8.10.1.2).

10.2.3 Initial Calibration Blank (ICB)

Following the ICV is the analysis of an ICB. The ICB consists of an aliquot of 0.2% Boric Acid (Section 8.8). Results must be less than the Reporting Limit.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Preparation

10.3.1.1 Sample Aliquots

10.3.1.1.1 Aqueous Samples

Add 100mL dechlorinated sample or a portion diluted to 100mL with DI water, to pre-washed Tecator tubes that are numbered to correspond with the samples.

10.3.1.1.2 Soil Samples

One gram of a well-homogenized sample is weighed out into a centrifuge tube (record weight in notebook). The sample is then transferred into a Tecator tube with 100 mL of DI.

10.3.1.1.3 QC Samples

In a similar manner, prepare the QC samples to be distilled with the batch (refer to Sections 9.1.1, 9.2, 9.4 and 9.6).

10.3.1.2 Add approximately 1.0mL of $Na_2S_2O_3$ to each tube in order to remove chlorine, followed by 5.0mL of Borate Buffer solution.

10.3.1.2.1 Aqueous Samples

Add small amounts of 6N NaOH until the pH is adjusted to 9.5. 2-3 drops of indicator (sec 8.14) can be used for pH adjustment. Color of solution will change from pink to blue at pH 9.5. Use pH paper to confirm pH reading. Check each sample with Potassium Iodide Starch paper (7.10) for chlorine presence. If starch paper showing presence of chlorine, add extra 1.0mL of $Na_2S_2O_3$ and check again.

10.3.1.2.2 Soil Samples

No pH adjustment is needed.

10.3.2 Distillation

10.3.2.1 **Equipment preparation:** To clean any prior contamination, first rinse the Tecator distillation unit by running a series of 3 rinse tubes. The first rinse containing 90mL DI and 10mL 6N NaOH. The second and third rinses are with100mL DI and 0.5mL 6N NaOH.

10.3.2.2 **Distillation:** Begin by distilling rinse tubes, Blank, Low and Hi LCS, and then Samples and QC Samples using the Tecator Instruments Automatic Distillation Unit according to the manufacturer's instructions.

Distillate is collected from the Tecator unit in 250mL disposable polypropylene cups that contain 20mL of 1.5% Boric acid (Section 8.7). Distillation is complete after collection of approximately 140mL. Fill each cup to 150mL with DI water. Cover and store at 1-4º C if Lachat analysis is to be delayed

10.3.3 **Ammonia analysis of distillate:** Follow the manufacturer's instructions for the proper operation of the Lachat ion analyzer. The following are specific notes for this analysis.

Sample throughput:	90 samples/hr; 40sec/sample
Pump speed:	35
Cycle period:	40 s
Inject to start of peak period:	25 s
Inject to end of peak period:	63 ± s

10.3.4 System Notes:

10.3.4.1 Allow 15 minutes for heating unit to warm up to 60ºC.

10.3.4.2 System IV GAIN: 175 X 1

10.3.4.3 If standards are not distilled, samples should be multiplied by 1.5 to correct for the increase in volume after the distillation.

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

10.3.4.4.1 Place all reagent lines in deionized water and pump to clear reagents (2-5 minutes).

10.3.4.4.2 Place reagent lines and carrier in 1M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

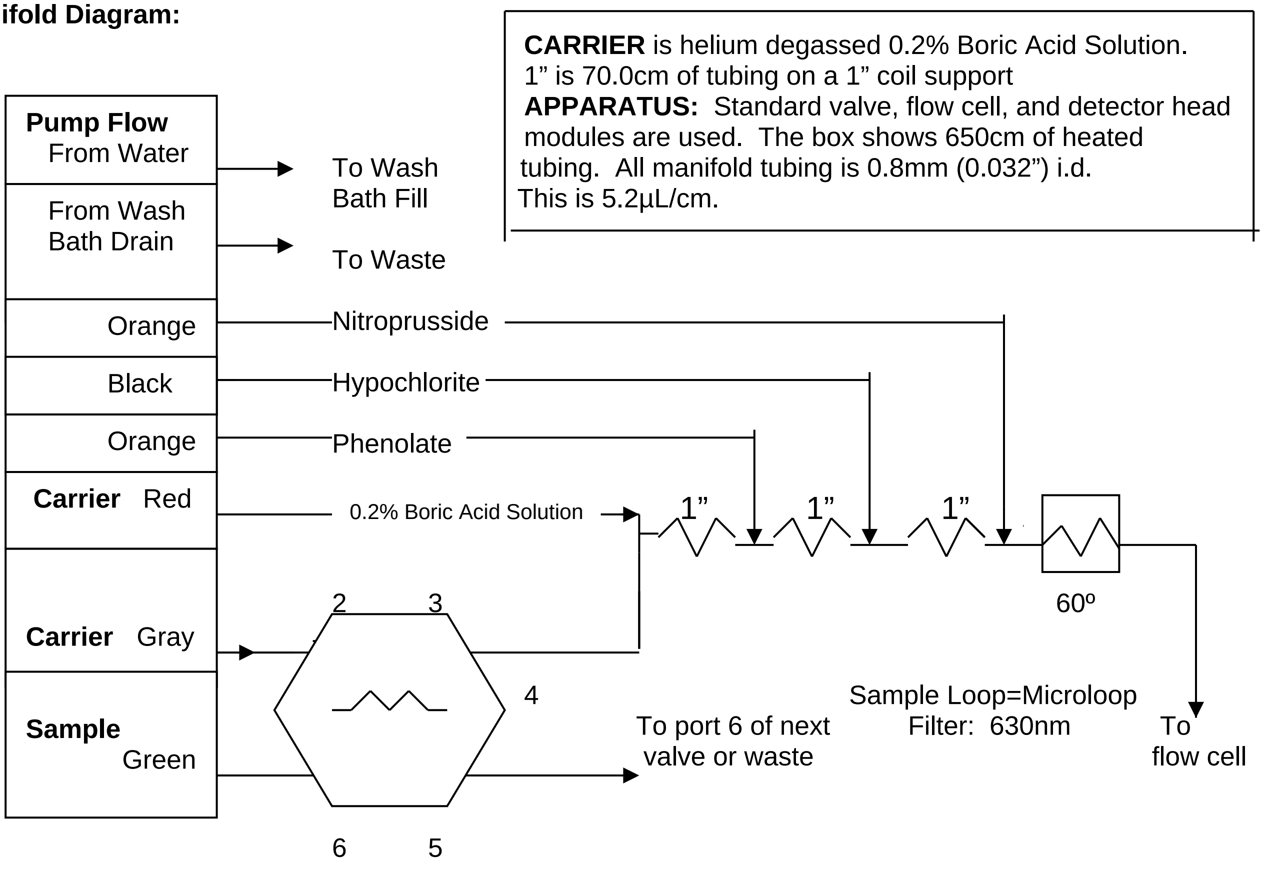
10.3.4.4.3 Place all lines in deionized water and pump until the HCl is thoroughly washed out.

10.3.4.4.4 Resume pumping reagents.

10.3.4.4.5 If sample distillate is colored or turbid, discard the sample distillate and re-distill the original sample at a higher dilution (Sections 10.3.1 and 10.3.2).

If there is not sufficient original sample volume for re-distillation, the colored/turbid distillate may be filtered through a 0.45µm filter and diluted on the Lachat instrument.

Manifold Diagram:



CARRIER is helium degassed 0.2% Boric Acid Solution.
1” is 70.0cm of tubing on a 1” coil support
APPARATUS: Standard valve, flow cell, and detector head modules are used. The box shows 650cm of heated tubing. All manifold tubing is 0.8mm (0.032”) i.d.
This is 5.2µL/cm.

10.4 Continuing Calibration

Analyze the following after every 10 samples and at the completion of analysis:

0.400ppm Low CCV, (Section 8.9.1.2.1)

4.0ppm Hi CCV, (Section 8.9.1.2.2)

Blank, 0.2% Boric Acid Solution. (Section 8.8)

10.5 Preventive Maintenance

10.5.1 All lines are flushed at the end of each run.

10.5.2 All equipment is kept clean.

11. Data Evaluation, Calculations and Reporting

Compute sample NH_3-N concentration by comparing sample peak areas with standard curve, as determined by the Lachat instrument software.

11.1 If the sample has a concentration of less than 2ppm, calculate results by using the low curve generated on Channel 1.

11.2 If the sample concentration is greater than 2ppm but less than 20ppm, calculate results by using the high curve generated on Channel 2.

11.3 If the concentration is greater than 20ppm, dilute the sample and reanalyze.

NOTE: For soil samples Lachat reading is multiplied by extraction volume (150ml) and divided by sample weight (the exact weight is used as recorded in the laboratory notebook). Calculations are done using Lachat software.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If ICV/CCV and/or ICB/CCB fail acceptance criteria, check sections 9.1.2, 9.3.1 and 9.4.1 for correction actions.

If the Method Blank result exceeds the Reporting Limit (RL) for the analyte, the Blank is reanalyzed. If reanalysis will confirm positive Method blank result, data has to be evaluated and all associated samples have be re-distilled and reanalyzed.

If lab doesn’t have samples for reanalysis, then samples with results below RL or more then 10x of reporting limit may be reported with narrative; samples with results between ND and 10x of reporting limit have to be B qualified and reported with narrative

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

If the Matrix Spike acceptance criteria is not met, the spiked sample is reanalyzed (if possible). If failure continues and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

If the RPD for the Duplicate measurements falls outside the Laboratory defined control limits, the sample is reanalyzed (if possible). If failure continues, and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

Note: Method **350.1** required MDL studies to be performed and evaluated every 6 month.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

NOTE: Lachat Phenol waste is hazardous and must be disposed of in "Hazmat" Lachat waste drums

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

None.

Nitrogen, Total Kjeldahl

References: **Method 351.1,** Methods for Chemical Analysis of Water and Waste, EPA-600/4-79-020, U.S. Environmental Protection agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268 (March 1979)

Method SM 4500N_{org}-C, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method SM 4500NH3-H, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-107-06-2-D, Methods for Automated Ion Analyzers, May 20, 1998.

1. Scope and Application

Matrices: Total Kjeldahl nitrogen can be determined in potable, surface, and saline waters as well as domestic and industrial wastewaters.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Tecator and/or Lachat Instrument and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

This method covers the determination of total kjeldahl nitrogen in drinking, surface and saline waters, and domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

In waters and wastewaters the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “Kjeldahl nitrogen”, a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea, and numerous synthetic organic materials. Typical organic nitrogen concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Ammonia is present naturally in surface and wastewaters. Its concentration generally is low in groundwaters because it adsorbs to soil particles and clays and is not leached readily from soils. It is produced largely by deamination of organic nitrogen containing compounds and by hydrolysis of urea. At some water treatment plants ammonia is added to react with chlorine to form a combined chlorine residual.

In the chlorination of wastewater effluents containing ammonia, virtually no free residual chlorine is obtained until the ammonia has been oxidized. Rather, the chlorine reacts with ammonia to form mono- and dichloramines. Ammonia concentrations encountered in water vary from less than 10µg ammonia nitrogen/L in some natural surface and groundwaters to more than 30 mg/L in some wastewaters.

In this discussion, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3–N, nitrite nitrogen as NO_2 - - N, and ammonia nitrogen as NH_3–N.

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

Organic Kjeldahl nitrogen is defined as the difference obtained by subtracting the free-ammonia value from the total Kjeldahl nitrogen value. This may be determined directly by removal of ammonia before digestion.

2. Summary of Method

The organic nitrogen is converted to ammonia via heating in the presence of concentrated sulfuric acid, K_2SO_4, $HgSO_4$, and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and treated and made alkaline with a hydroxide-thiosulfate solution. The digestate is distilled at high pH into a solution of boric acid. The ammonia in the distillate is determined colorimetrically by the phenate method.

The phenate 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 product is measured at 630nm, and is directly proportional to the ammonia concentration in the digestate.

2.1 Method Modifications from Reference

This method has been modified for soil digestion, Section 9.4.1.

3. Detection Limits

The RDL is determined to be 0.3 mg/L for liquid samples and 150 mg/Kg for soil or solid samples.

4. Interferences

4.1 Instrumental

Samples with a high concentration of TKN may carry-over into the next sample and therefore yield false high results in that next sample. If a sample with a low concentration follows a sample with a high concentration, re-analyze the low sample to ensure results are accurate.

4.2 Parameters

High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

NOTE: Both Phenol and Mercury used in this method are hazardous and general laboratory safety practices must be observed. Due to the Mercury used in this procedure, digestion must be done under a hood.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

The most reliable results are obtained on fresh samples. Use plastic or glass containers.

6.2 Sample Preservation

Samples may be preserved by addition of 2mL of concentrated H_2SO_4 per liter if sample cannot be analyzed immediately. Refrigerate at 4ºC.

6.3 Sample Handling

Even when properly preserved, conversion of organic nitrogen to ammonia may occur. Therefore samples should be analyzed as soon as possible.

7. Equipment and Supplies

7.1 **Digestion apparatus:** Kjeldahl Digestion System 20, Model 1015 Digester. Follow the instrument manufacturer’s instructions.

7.2 **Distillation apparatus:** Tecator Instruments Automatic Distillation Unit: Follow the instrument manufacturer’s instructions for proper operation.

7.3 **Automated Ion Analyzer:** Lachat Instruments

7.4 **Disposable polypropylene cups:** 250mL with covers.

7.5 **Glass Tuttlecaps:** For digestion.

7.6 **Glass Pipets:** Various volumes.

7.7 **Auto-pipettor with tips:** For 10mL capability.

7.8 Analytical balances: capable to weigh 0.1000g aliquots.

7.9 Dilu Vials

7.10 Boiling chips

8. Standards and Reagents

8.1 Sodium Phenolate: **CAUTION!** Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin. In a 1L volumetric flask, dissolve 88mL of 88% liquified phenol or 83g crystalline phenol (C_6H_5OH) in approximately 600mL DI water. While stirring, slowly add 32g sodium hydroxide (NaOH). Cool, dilute to the mark, and invert three times. Do **not** degas this reagent. Expires one month from date of preparation.

8.2 Sodium Hypochlorite (approximate 2.6%): In a 500mL volumetric flask, dilute 250mL Regular Chlorine bleach [5.25% sodium hypochlorite (NaOCl)] to the mark with DI water. Invert three times to mix. Expires one month from date of preparation.

8.3 Sodium Nitroprusside (coloring agent): In a 1L volumetric flask, dissolve 3.50g sodium nitroprusside (Sodium Nitroferricyanide [$Na_2Fe(CN)_5NO_2 \cdot H_2O$]) dilute to the mark with DI water. Degas with helium to prevent bubble formation. Use He at 140kPa (20 lb/in^2) through a helium degassing tube. Bubble He vigorously through the solution for one minute. Expires one month from date of preparation.

8.4 Boric 1.5% Boric Acid Solution: To a 1000mL volumetric flask add 15g Boric Acid. Dilute to the mark with DI water. Expires one month from date of preparation.

8.5 Mercuric Sulfate Solution: Dissolve 8g red mercuric oxide (HgO) in 50mL of l:4 sulfuric acid (10.0mL concentrated H_2SO_4 : 40mL distilled water) and dilute to 100mL with distilled water. Expires one month from date of preparation.

8.6 Digestion Solution (Sulfuric Acid-Mercuric Sulfate-Potassium Sulfate): Dissolve 267g K_2SO_4 in 1300mL distilled water and 400mL concentrated H_2SO_4. Add 50mL mercuric sulfate (Section 8.5) solution and dilute to 2L with distilled water. Expires one month from date of preparation.

8.7 Sodium Hydroxide-Sodium Thiosulfate Solution: Dissolve 500g NaOH and 25g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1L. Expires one month from date of preparation.

8.8 0.2% Boric Acid Solution (Carrier Solution): To a 2L volumetric flask, dissolve 4g Boric Acid (H_3BO_3) in DI water. Degas by bubbling vigorously with Helium for one minute. Expires one month from date of preparation.

8.9 Stock Standard, 1000ppm as NH_3 (for calibration solutions): Commercially prepared. Certificate of analysis is required.

8.9.1 Intermediate Calibration Stock Standard, 100ppm as NH_3: To a 100mL volumetric flask, add 10.0mL of Stock Standard (Section 8.9) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Expires one month from date of preparation.

8.9.1.1 Nine Working Calibration Standards: Prepare the following standards in volumetric flasks:

8.9.1.1.1 20.0ppm: 40mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.2 10.0ppm: 20mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.3 8.00ppm: 8mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.4 4.00ppm: 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.5 2.00ppm: 2mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.6 1.00ppm: 1mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.7 0.400ppm: 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.8 0.200ppm: 1mL of 20ppm standard (Section 8.9.1.1.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler. Prepare fresh each day of use.

8.9.1.1.9 0.100ppm: 1mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler. Prepare fresh each day of use.

8.9.1.1.10 0.050ppm: 20mL of 0.100ppm standard (Section 8.9.1.1.9) to 40mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler. Prepare fresh each day of use.

8.9.1.2 Continuing Calibration Standards:

8.9.1.2.1 0.400ppm Low CCV: 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.2.2 4.00ppm Hi CCV: 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.10 Stock Standard, 1000ppm as TKN (for spike): Commercially prepared. Certificate of analysis is required

8.10.1 Intermediate spike Stock Standard, 200ppm as TKN: To a 100mL volumetric flask, add 20.0mL of Stock Standard (Section 8.10) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Expires one month from date of preparation.

8.11 Stock Standard Solution, 1000ppm as NH_3 (for ICV only): Commercially prepared. Certificate of analysis is required. This must be from a different source than that used for Stock Standard (Section 8.9).

8.11.1 Initial Calibration Verification Standards (ICV):

8.11.1.1 Hi ICV, 10ppm: To a 100mL volumetric flask add 1mL of 1000ppm standard (Section 8.11). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.11.1.2 Hi ICV, 8.0ppm: To a 100mL volumetric flask add 0.8mL of 1000ppm standard (Section 8.11). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.11.1.3 Low ICV, 1.0ppm: To a 100mL volumetric flask add 10mL of 10ppm ICV (Section 8.11.1.1). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.12 Stock Standard Solution, 1000ppm as TKN (for LCS): Commercially prepared. Certificate of analysis is required. This must be from a different independent source than that used for Stock Standard (Section 8.10).

8.12.1 LCS solution, 200ppm as TKN: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.12) and dilute to the mark with DI water. Expires one month from date of preparation.

8.13 6N Sodium Hydroxide Solution : Dissolve 240g NaOH in distilled water and dilute to 1L. Expires six month from date of preparation.

9. Procedure

9.1 SET-UP

9.1.1 Clean 250mL Tecator tubes by rinsing twice with approximately 0.5mL of 6N NaOH solution and 100mL RO water. Rinse twice again with DI water.

9.1.2 Rinse glass tuttlecaps under the hood in a 1000mL beaker with approximately 500mL DI and 1.0 mL of 6N NaOH. Allow to sit in this solution until use.

9.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

9.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has 7 calibration points. The correlation coefficient of each curve must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

9.2.1.1 Channel 1 is used to generate a calibration curve on the low range from 0.00 – 2ppm.

9.2.1.2 Channel 2 is used to generate a calibration curve on the high range from 0 – 20ppm.

Alternative method: One board can be used to calibrate the Lachat instrument. Ten point calibration curve will be used with calibration standards 10.0, 8.0, 4.0, 2.0, 1.0, 0.4, 0.2, 0.1, 0.05 mg/l each and blank. The correlation coefficient must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards

Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. % recoveries for the low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995. The standards for the calibration curve are recalculated manually to prove the software calculations are within +/- 10% of the true concentrations.

9.2.2 Initial Calibration Verification (ICV)
Prior to sample analysis, an ICV is analyzed at 1.0ppm (Section 8.10.1.2) to verify the low calibration curve on Channel 1. Another ICV is analyzed at 10ppm (Section 8.10.1.1) to verify the high calibration curve on Channel 2. Both ICVs must yield results ± 10% of their true value, otherwise re-calibration is necessary.

Note: if instrument is calibrated using one board calibration, then both ICV's (Low and High), will be evaluated and high ICV will be 8.0 mg/l ICV standard (Section 8.11.1.2)

9.2.3 Initial Calibration Blank (ICB)
Following the ICV is the analysis of an ICB. The ICB consists of an aliquot of 0.2% Boric Acid (Section 8.8). Results must be less than < 0.05mg/L.

9.3 Standardization (Continuing Calibration Verification)

Analyze the following after every 10 samples and at the completion of analysis:

0.400ppm Low CCV, (Section 8.9.1.2.1)

4.0ppm Hi CCV, (Section 8.9.1.2.2)

Blank, 0.2% Boric Acid Solution. (Section 8.8)

9.4 Equipment Operation and Sample Analysis

9.4.1 Aqueous Sample Digestion: Add 50mL of homogenized sample or a portion diluted to 50mL with DI water, to pre-washed Tecator tubes that are numbered to correspond with the samples.

Soil/Solid Sample Digestion: Weigh 0.1g of homogenized soil/solid sample into dilu vial and record the weight in the laboratory notebook. Transfer to a pre-washed, pre-numbered Tecator tube and add 50mL of DI water.

In a similar manner, for each matrix, prepare the QC samples to be digested with the batch (refer to Sections 10.2.1, 10.3.1, 10.7 and 10.8)

Then add approximately 1g of black boiling chips to each tube. Move to hood before adding 10mL of Digestion Solution (Section 8.6) to each tube with a calibrated pipettor.

Rinse glass tuttlecaps with DI and place one onto the top of each Tecator tube. Place Tecator tube rack onto Tecator digestion block and turn temperature control knob to "4" which represents approximately 250ºC. (Temperature should never exceed 300ºC.) Cook for approximately 2 hours to SO_3 fumes. The remaining mixture will be clear or pale yellow in color. Remove tube from digestion block and allow to cool to ~80 °C before adding DI to the 90mL mark on the tube.

9.4.2 **Distillation:** To minimize contamination, leave distillation apparatus assembled after steaming out and until just before starting sample distillation. Make the digestate alkaline by careful addition of 20mL of sodium hydroxide-thiosulfate solution (Section 8.7) without mixing. Do not mix until the digestion tube has been connected to the distillation apparatus. Connect the Tecator tube to the Tecator distillation unit's stopper as defined in the manual. Distill for 3 minutes 40 seconds as defined in the manual and collect distillate in 20mL boric acid solution (Section 8.4). Distill at maximum rate with the tip of the delivery tube below the surface of boric acid receiving solution. Collect at least 90mL distillate. Dilute to 150mL with DI water. Refrigerate at 0-4ºC if Lachat analysis is delayed.

9.4.3 **Ammonia analysis of distillate:** Follow the manufacturer's instructions for the proper operation of the ion analyzer. The following are specific notes for this analysis.

Sample throughput:	90 samples/hr; 40 sec/sample
Pump speed:	35
Cycle period:	40 s
Inject to start of peak period:	25 s
Inject to end of peak period:	63 s

9.4.4 **System Notes:**

9.4.4.1 Allow 15 minutes for heating unit to warm up to 60ºC.

9.4.4.2 System IV GAIN: 175 x 1.

9.4.4.3 If standards are not distilled, samples should be multiplied by procedure dilution (final volume 150mL) divided by initial volume.

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

9.4.4.4.1 Place all reagent lines in deionized water and pump to clear reagents (2-5 minutes).

9.4.4.4.2 Place reagent lines and carrier in 1M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

9.4.4.4.3 Place all lines in deionized water and pump until the HCl is thoroughly washed out.

9.4.4.4.4 Resume pumping reagents.

9.4.4.5 If samples are colored or are suspected to show a background absorbance, this interference should be subtracted. This can be done by diluting or by the following procedure:

9.4.4.5.1 Calibrate the system in the normal manner.

9.4.4.5.2 Disable the check standard or DQM features and analyze the samples.

9.4.4.5.3 Place reagent and carrier lines in DI water and allow the baseline to stabilize.

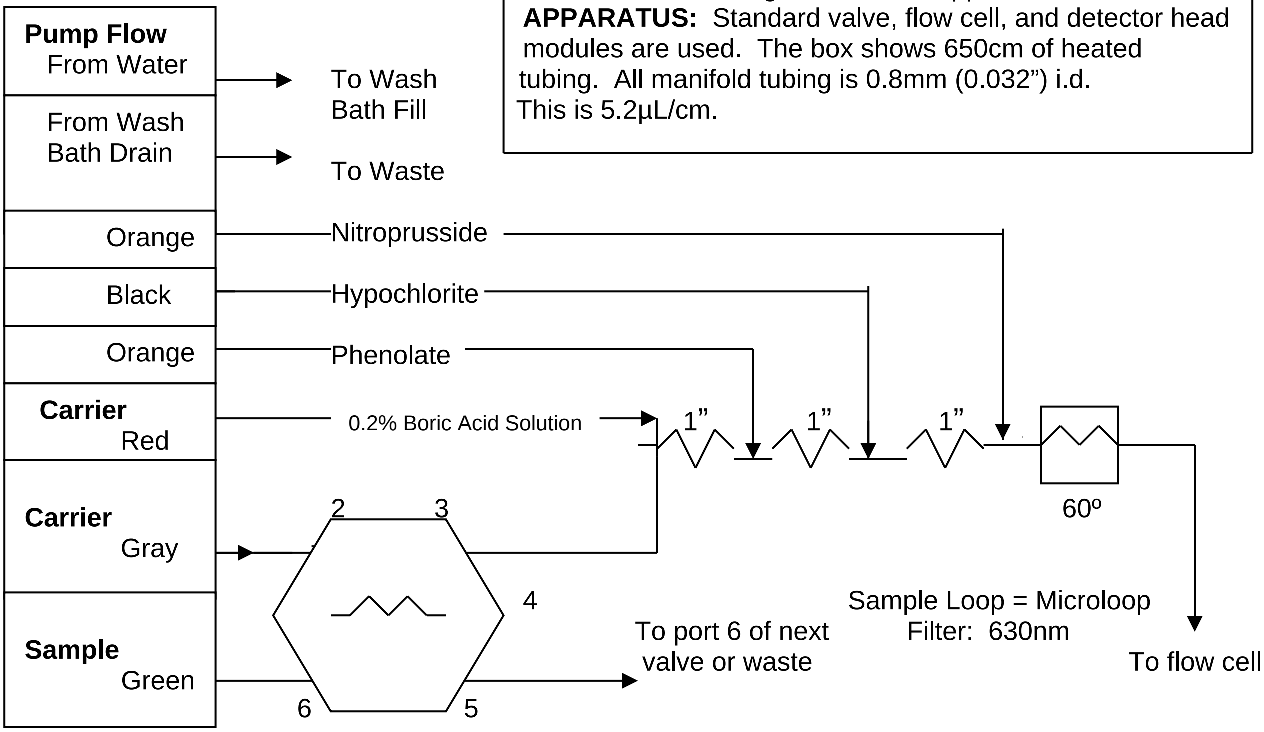
9.4.4.5.4 Inject samples again without recalibrating.

9.4.4.5.5 Subtract the "background" concentration from the original concentration to give the corrected concentration.

Original Concentration – Background Concentration = Corrected Concentration.

Manifold Diagram:

CARRIER is helium degassed 0.2% boric acid solution.
1" is 70.0cm of tubing on a 1" coil support
APPARATUS: Standard valve, flow cell, and detector head modules are used. The box shows 650cm of heated tubing. All manifold tubing is 0.8mm (0.032") i.d.
This is 5.2µL/cm.



9.5 Preventative Maintenance

9.5.1 All lines are flushed at the end of each run.

9.5.2 All equipment is kept clean.

9.6 Calculations

Prepare standard curves by plotting peak areas of standards processed through the manifold against NH_3 -N concentrations in standards. Compute sample NH_3 -N concentration by comparing sample peak areas with standard curve, as determined by the Lachat instrument software.

9.6.1 If the sample has a concentration of less than 2ppm, calculate results by using the low curve generated on Channel 1.

9.6.2 If the sample concentration is greater than 2ppm, calculate results by using the high curve generated on Channel 2.

Alternative method: use only one Channel and calculate all results from one channel.

To compute final results for aqueous samples, multiply the direct reading by the dilution factor based on the initial preparation volume.

$$\text{TKN mg/L} = \text{mg/L direct reading} \times \text{dilution factor}$$

To compute results for soil/solid samples, multiply the direct reading by the extraction final volume (150mL) and then divide by the weight of the sample used for extraction (Section 9.4.1), and multiply by a dilution factor as necessary.

$$\text{TKN mg/Kg} = \left[\frac{\text{(mg/L direct reading)} \times \text{extraction final volume (150mL)}}{\text{Sample weight (g)}} \right] \times \text{dilution factor}$$

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for failed parameters of the method.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test for all parameters listed in the method.

10.2 Blank

10.2.1 The Distillation Blank is 50mL of DI. Distill one per batch of 20 samples or less. Subtract any blank greater than 0.3mg/L from all samples and QC.

10.2.2 The Analytical Blank (ICB) for the Lachat analysis is not distilled and is 0.2% Boric Acid Solution (Section 8.8).

The ICB is run after the initial calibration verification standards (ICV) and another is run after the continuing calibration standards (CCV).

10.3 Laboratory Control Samples (LCS)

10.3.1 **Distillation:** Distill a Low and a Hi LCS with each batch of 20 samples or less. The results from the Hi LCS are reported for the batch. The Low LCS is used to verify the low curve, but is not reported.

10.3.1.1 **Low 4.0ppm LCS:** Add 1mL of 200ppm LCS solution (Section 8.10.2) to 50 mL DI. This is used for the Low 0 - 6 ppm curve.

10.3.1.2 **Hi 40ppm LCS:** Add 10mL of 200ppm LCS solution (Section 8.10.2) to 50 mL DI. This is used for the Hi 0 - 60 ppm curve.

10.4 Initial Calibration Verification Standards

10.4.1 **Lachat Analysis:** The ICVs are not distilled. Analyze the following after calibration of the Lachat instrument. Recoveries must be within 10% of the true value, otherwise recalibration of the instrument is necessary.

10.4.1.1 **Low ICV, 1.0ppm** (Section 8.10.1.2)

10.4.1.2 **Hi ICV, 10ppm** (Section 8.10.1.1); **HI ICV** will be 8.0 ppm (Section 8.10.1.2) in case of one board calibration.

10.5 Continuing Calibration Verification Standards

10.5.1 **Lachat Analysis:** The CCVs are not distilled. Analyze the following after every ten samples and at the completion of analysis. Recoveries must be within 10% of the true value.

If recoveries fall outside of this range, the cause for the failure is determined and corrected, and the instrument is recalibrated. All samples that were analyzed since the last CCV that was within range are reanalyzed.

10.5.1.1 **0.4ppm Low CCV** (Section 8.9.1.2.1)

10.5.1.2 **4.0ppm Hi CCV** (Section 8.9.1.2.2)

10.6 Interference Check Standards

None.

10.7 Matrix Spike

One per batch of 20 samples or less. Prior to distillation, add 2mL of 200ppm Spiking Solution (Section 8.9.2) per 50mL of sample.

10.8 Duplicates

Distill one duplicate sample per batch of 20 samples or less.

10.9 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

Control limits for the method parameters are generated by the QC staff. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

10.10 Analytical Sequences

10.10.1 Distillation Sequence:

3 Rinse tubes
Blank 1
Low LCS
Hi LCS
Rinse
Samples (each sample must be followed by rinse, except between sample and its spike or sample and its duplicate)
Duplicate
Spike
Rinse
Shut-down

10.10.2 Lachat Analytical Sequence:

Instrument Calibration
DQM = Hi 4.0ppm CCV
Low 0.4ppm CCV
CC Blank
Low 1.0ppm ICV
Hi 10ppm ICV or 8.0 ppm ICV
IC Blank
Samples
DQM : Run after every 10 samples and at completion of analysis
Rinse reagent lines with 1M HCl for 5 to 10 minutes
DI water rinse for 5 to 10 minutes
Air rinse 5 to 10 minutes.
Shut-Down.

11. Method Performance

11.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732 These studies performed by the laboratory are maintained on file for review.

11.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

11.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

11.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

12. Corrective Actions

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

See Chemical Hygiene Plan SOP/1728 for waste handling and disposal.

NOTE: TKN Lachat waste contains Mercury and must be deposited into TKN/Lachat waste stream in the Waste Room.

Orthophosphate - Colorimetric, Combined Reagent

Reference: SM 4500P-E, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: Water and wastewater samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), 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 run-off 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.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

Phosphorus analyses embody two general procedural steps: (a) conversion of the phosphorus form of interest to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45-µm-pore-diameter membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45-µm filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed “reactive phosphorus.” While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The ascorbic acid method is used for the determination of orthophosphate in environmental samples. Ammonium molybdate and potassium antimonyl tartrates react in acid medium with orthophosphate to form a heteropoly acid-phosphomolybdic acid that is reduced to intensely colored molybdenum blue by ascorbic acid. Samples are analyzed at 880nm using a spectrophotometer.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The Reporting Limit is 0.005mg/L.

4. Interferences

Correction for Turbidity or Interfering Color: The natural color of water generally does not interfere at the high wavelength used. For highly colored or turbid waters, prepare a blank by adding all reagents except ascorbic acid and potassium antimonyl tartrate to the sample. Subtract the blank absorbance from the absorbance of each sample.

In some cases, the background color or turbidity can be eliminated by a dilution, but this will raise the reporting limit.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 250mL plastic containers.

6.2 Sample Preservation

Samples are not preserved. Do not add either acid or $CHCl_3$ as a preservative.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ±2 °C. Analysis must be performed within 48 hours of collection.

7. Equipment and Supplies

7.1 Spectrophotometer, with infrared phototube for use at 880nm, providing a light path of 2.5cm.

7.2 Acid-washed Glassware: Use acid-washed glassware for determining low concentration of phosphorus. Phosphate contamination is common because of its absorption on glass surfaces. Avoid using commercial detergents containing phosphate. Clean all glassware with 1:1 HCl and rinse well with distilled water. The glassware should only be used for phosphate determination.

7.3 Centrifuge Tubes: 50mL volume. (Must be new and disposable.)

7.4 0.45µm Acrodisc filters with disposable syringes

7.5 Pipets or Pipettor: Various sizes, new and disposable or acid-rinsed glass

8. Reagents and Standards

8.1 Stock Phosphate Standard: 1000 mgP/L This stock solution is certified and purchased commercially prepared. Store at room temperature. Expires upon manufacturer's specified date.

8.2 Intermediate Phosphate Standard: 50 mgP/L Dilute 50.0mL stock phosphate solution to 1000mL with DI water, 1.00mL = 2.50µg P. Store refrigerated at 4 ±2 °C. Expires 6 months from date of preparation.

8.3 Working Standard: 1.0 mgP/L Add 2mL of 50 mgP/L intermediate standard (Section 8.2) to 100mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.4 Calibration Standards: Follow table below for preparation instructions. Prepare fresh on each day of use. Use a calibrated pipettor and bring up to 25mL with DI.

Volume of 1.0 mg/L Working Standard (Section 8.3)	Final Volume (mL)	Calibration Standard Final Concentration (mgP/L)
0	25	0.0
0.125 mL	25	0.005
0.250 mL	25	0.01
1.250 mL	25	0.05
2.50 mL	25	0.10
12.5 mL	25	0.50
25 mL	25	1.00

8.5 **ICV-LCS-CCV Stock Standard: 1000 mgP/L** Second source standard than that used in Section 8.1.

8.6 **ICV-LCS-CCV Intermediate Standard: 25 mgP/L** Add 5mL of 1000 mgP/L stock standard (Section 8.5) to 200mL volumetric flask and dilute to volume with DI water. Store refrigerated at 4 ±2 °C. Expires 6 months from date of preparation.

8.7 **ICV-LCS-CCV Working Standards:** Prepare fresh on each day of use.

8.7.1 **0.5 mgP/L:** Add 0.25mL of 50 mg/L intermediate standard (Section 8.6) to 25mL volumetric flask and dilute to volume with DI water.

8.8 **Matrix Spike Solution:** Intermediate Phosphate Standard (Section 8.2) is utilized for matrix spike solution. 0.25mL of the 50 mgP/L standard added to 25mL of sample will afford a 0.5mgP/l matrix spike concentration.

8.9 **Sulfuric Acid, H2SO4, 5N:** Dilute 70mL concentrated sulfuric acid to 500mL with DI. Store at room temperature. Expires 6 months from date of preparation. Alternatively, 1L of 5N H2SO4 can be made: dilute140 ml of concentrated sulfuric acid to 1000mL with DI. Expires 6 months from date of preparation.

8.10 **Potassium Antimonyl Tartrate Solution:** Dissolve 1.3715g $K(SbO) C_4H_4O_6 \cdot \frac{1}{2}H_2O$ in 400mL DI water in a 500mL volumetric flask and dilute to volume. Store in a glass-stoppered bottle at room temperature. Expires one month from date of preparation.

8.11 **Ammonium Molybdate Solution:** Dissolve 20g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 500mL DI water. Store in a glass-stoppered bottle at room temperature. Expires one month from date of preparation.

8.12 **Ascorbic Acid, 0.1M:** Dissolve 3.52g ascorbic acid in 200mL DI water. The solution is stable for about 1 week at 4 ±2ºC. Alternatively, 100 ml of Ascorbic Acid can be made: Dissolve 1.76g ascorbic acid in 100mL DI water. The solution is stable for 1 week at 4 ±2ºC.

8.13 **Combined Reagent:** Mix the above reagents in the following proportions for 100mL of the combined reagent: 50mL 5N H_2SO_4 (Section 8.9), 5mL potassium antimonyl tartrate solution (Section 8.10), 15mL ammonium molybdate solution (Section 8.11), and 30mL ascorbic acid solution (section 8.12). Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours. Discard reagent if it turns blue or black in color.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 **ICB or Method Blank** - One method blank, which consists of 25mL DI water filtered through a 0.45 micron Acrodisc filter, must be analyzed per batch of 20 samples or less. The method blank result must be less than the Reporting Limit.

9.1.2 **Continuing Calibration Blank** – The ICB should be re-read after every 10 samples (thus becoming the CCB) and at the end of the batch The CCB result must be less than the Reporting Limit. If the CCB is greater than the RL, all samples analyzed since the last passing CCB must be recolored and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCS per batch of 20 samples or less. The calibration curve must be verified by a second source standard prior to performing any sample analysis. The LCS must be recovered at 90 – 110% of the true value.

If the LCS fails, re-analyze. If failure remains, stop analysis, correct problem and perform re-calibration.

9.3 Initial Calibration Verification (ICV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. The ICV must be recovered at 90 – 110% of the true value.

If the ICV fails, re-analyze. If failure remains, stop analysis, correct problem and perform re-calibration.

9.4 Continuing Calibration Verification (CCV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. The CCV must be recovered at 90 – 110% of the true value.

If the CCV fails, re-analyze. If failure remains, stop analysis, correct problem and perform re-calibration.

The CCV must be re-read every 10 samples and at the end of the batch

9.5 Matrix Spike

Analyze one per batch of 20 samples or less. Concentration is 0.5 mg P /L. The matrix spike must be recovered at 80 – 120% of the true value. If the MS recovery is outside of acceptance limits, the sample and its spike are reanalyzed. If the MS failure continues, a narrative is submitted with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate, per batch of 20 samples or less. The RPD must be $\leq$ 20%. If the RPD is outside of acceptance limits, the sample and its duplicate are reanalyzed. If the RPD failure continues, a narrative is submitted with the data for inclusion on the Client report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Calibration curve generation or verification of existing curve.
- Acid-rinsing of glassware
- Sample filtration including blank
- Add sample aliquot to a new centrifuge tube
- Add combined reagent to samples
- Read sample absorbance after 10-30 minutes
- Analyze CCV and CCB after every 10 samples to verify curve
- End sequence with CCV and CCB
- Calculate results

10. Procedure

10.1 Equipment Set-up

10.1.1 **Preparation of calibration curve:** Prepare individual calibration curve from a series of six standards and DI (0.005 mgP/L to 1.0 mgP/L) on a daily basis when samples are to be analyzed. Use a DI water blank to zero the instrument before taking photometric readings for the calibration curve. Plot a curve of absorbance vs. phosphate concentration. The curve is acceptable if the calibration coefficient is ≥ 0.995. The calibration curve is prepared fresh each day of analysis.

All calibration points are back calculated and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn’t exceed 100% and correlation coefficient will not be worse than 0.995.

10.2 Initial Calibration

See Section 9.3

10.3 Equipment Operation and Sample Processing

10.3.1 Filter samples and QC samples through 0.45µm Acrodisc filters. 1 µm Acrodisc filter may be used to prefilter hard-to-filter samples.

10.3.2 Pour 25mL of each clear filtered sample, a duplicate and matrix spike sample into a corresponding new (never used) centrifuge tube.

10.3.3 Add 4.0mL combined reagent to all 25mL samples and QC sample aliquots and mix thoroughly.

10.3.4 After at least 10 minutes but no more than 30 minutes, measure absorbance of each sample at 880nm with a 2.5cm cell. Follow the procedure described in the Wet Chemistry Electronic Notebook Work Instructions (WI/2516).

10.3.5 Compare with a standard curve prepared from a range of standards and carried through this procedure. Sample concentration must fall within the range of the calibration curve.

10.3.6 If the sample concentration is greater than the highest calibration standard concentration, dilute the original sample with DI water and recolor and reanalyze as outlined above (Section 10.3.1 – 10.3.6).

10.4 Continuing Calibration

See Section 9.4.

10.5 Preventive Maintenance

The Spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

mg P/L = mg P (from calibration curve) x dilution

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances or improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

2516 Electronic Laboratory Notebook Work Instructions

16. Attachments

None.

Total Phosphorous

Dissolved Phosphorus

Colorimetric, Combined Reagent

References: **SM 4500P-E,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

SM4500P-B, Section 5 (Persulfate Digestion), Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

AQ2 method**: EPA-119-A** Rev. 7, equivalent to EPA 365.1, version 2(1993) **SM4500-P-B, F(**18-20**)**

1. Scope and Application

Matrices: Water and wastewater samples and soils.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), 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 run-off 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.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

Phosphorus analyses embody two general procedural steps: (a) conversion of the phosphorus form of interest to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45-µm-pore-diameter membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45-µm filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed “reactive phosphorus.” While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms.

Acid hydrolysis at boiling-water temperature converts dissolved and particulate condensed phosphates to dissolved orthophosphates. The hydrolysis unavoidably releases some phosphate from organic compounds, but this may be reduced to a minimum by judicious selection of acid strength and hydrolysis time and temperature. The term “acid-hydrolyzable phosphorus” is preferred over “condensed phosphate” for this fraction.

The phosphate fractions that are converted to orthophosphate only by oxidation destruction of the organic matter present are considered “organic” or “organically bound” phosphorous. The severity of the oxidation required for this conversion depends on the form of, and to some extent on the amount of, the organic phosphorus present. Like reactive phosphorus and acid hydrolyzable phosphorus, organic phosphorus occurs both in the dissolved and suspended fractions.

The total phosphorus as well as the dissolved and suspended phosphorus fractions each may be divided analytically into the three chemical types that have been described: reactive, acid hydrolyzable, and organic phosphorus. Determinations usually are conducted only on the unfiltered and filtered samples. Suspended fractions generally are determined by difference.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Digestion Method: Because phosphorus may occur in combination with organic matter, a digestion method to determine total phosphorus must be able to oxidize organic matter effectively to release phosphorus as orthophosphate. This digestion is performed by using the persulfate oxidation technique.

Colorimetric Method: The ascorbic acid method is used for the determination of orthophosphate in environmental samples. An extraction step is recommended for the lower levels and when interferences must be overcome. Ammonium molybdate and potassium antimonyl tartrates react in acid medium with orthophosphate to form a heteropoly acid-phosphomolybdic acid that is reduced to intensely colored molybdenum blue by ascorbic acid. The absorbance of this complex is measured photometrically at 880nm.

2.1 Method Modifications from Reference

Glassware is acid rinsed with room temperature 1:1 HCl, instead of hot dilute HCl.

Initial testing of samples with phenolphthalein has been eliminated since samples are received already preserved with H_2SO_4 and are pH checked by the Login Department upon receipt.
Soil samples are analyzed using the same digestive procedure.

3. Reporting Limits

The Reported Detection Limit is 0.01mg/L for waters and 5.0mg/kg for soils

4. Interferences

Correction for Turbidity or Interfering Color: The natural color of water generally does not interfere at the high wavelength used. For highly colored or turbid waters, prepare a blank by adding all reagents except ascorbic acid and potassium antimonyl tartrate to the digested sample aliquot. Subtract the blank absorbance from the absorbance of each sample.

Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1mg As/L interfere with the phosphate determination.

Hexavalent chromium and NO_2 interfere to give results about 3% low at concentrations of 1mg/L and 10 to 15% low at 10mg/L.

Sulfide (Na_2S) and silicate do not interfere at concentrations of 1.0 and 10mg/L.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Water samples are collected in 500mL plastic bottles, soil samples may be collected in plastic or glass jars.

6.2 Sample Preservation

If samples are for Dissolved Phosphorus analysis, filtration must take place prior to preservation with H_2SO_4 to a pH < 2.

All samples are preserved with H_2SO_4.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2 °C. Analysis must be performed within 28 days of collection. All samples should be analyzed as soon as possible after digestion. If a prolonged period passes in between, sample extracts are refrigerated at 4 ± 2 °C.

7. Equipment and Supplies

7.1 Spectrophotometer, with infrared phototube for use at 880nm, providing a light path of 2.5cm.

7.2 Acid-washed Glassware: Use acid-washed glassware for determining low concentration of phosphorus. Phosphate contamination is common because of its absorption on glass surfaces. Avoid using commercial detergents containing phosphate. Clean all glassware with 1:1 HCl two times followed by two DI water rinses. Preferably, reserve the glassware only for phosphate determination. Only disposable syringes and filters are to be used for filtering samples for Dissolved Phosphorus analysis.

7.3 Centrifuge Tubes: 50mL volume. (Must be new and disposable.)

7.4 Hot Plate: A 30cm x 50cm heating surface is adequate.

7.5 Scoop, 0.5gm To hold required amounts of persulfate crystals.

7.6 Erlenmeyer Flasks: 125mL volume.

7.7 0.45µm membrane filters: For Dissolved Phosphorus sample preparation.

7.8 Borosilicate Glass beads

7.9 SEAL AQ2 Discrete Analyzer, with all associated reagent wedges, sample tubes, and reaction segments. The SEAL has a light and filter capable of maintaining a 880nm wavelength.

7.10 Boiling Chips ultra-pure, non-reactive.

7.11 Syringes to use with membrane filters.

7.12 Pipettes Class A glass or automated.

8. Reagents and Standards

8.1 Calibration Curve and Spike, Stock Complex Phosphate Standard: 1000 mgP/L This stock solution is certified and purchased commercially prepared. Stored at room temperature per manufacturer's specifications. Expires upon manufacturer's specified date.

8.2 Calibration Curve and Spike, Intermediate Complex Phosphate Standard: 50 mgP/L Dilute 5.0mL stock complex phosphate solution to 100mL with DI water. Store at 4 ± 2°C. Expires 6 months after date of preparation.

8.3 Calibration Curve, Working Standard: 1.0 mgP/L Add 2mL of 50 mgP/L intermediate standard (Section 8.2) to 100mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.4 Calibration Standards: Follow table below. Prepare fresh on each day of use.

Volume of 1.0 mg/L Working Standard (Section 8.3)	Final Volume (mL)	Calibration Standard Final Concentration (mgP/L)
0 mL	50	0
0.5 mL	50	0.010
2 mL	50	0.040
5 mL	50	0.100
25mL	50	0.500
50mL	50	1.000

8.5 ICV-LCS-CCV Stock Complex Phosphate Standard: 1000 mgP/L Second, independent, source standard. Stored at room temperature per manufacturer's specifications. Expires upon manufacturer's specified date.

8.6 ICV-LCS-CCV Intermediate Complex Phosphate Standard: 50 mgP/L Add 5mL of 1000 mgP/L stock standard (Section 8.5) to 100mL volumetric flask and dilute to volume with DI water. Store at 4 ± 2˚C. Expires 6 months after date of preparation.

8.7 ICV-LCS-CCV Working Standard: Prepare fresh each day of use.

8.7.1 0.5 mgP/L: Add 0.5mL of 50 mg/L intermediate standard (Section 8.6) to 50mL centrifuge tube and dilute to the 50mL mark with DI water.

8.8 Matrix Spike: Intermediate Phosphate Standard (Section 8.2) is utilized for matrix spike solution. Pipet 0.5mL of the 50 mgP/L standard into 50mL of sample to result in a 0.5mg/L spike concentration.

8.9 Sulfuric Acid, H_2SO_4, 5N: Dilute 140mL concentrated sulfuric acid to 1L with DI. Store at room temperature. Expires 6 months from date of preparation.

8.10 Potassium Antimonyl Tartrate Solution: Dissolve 1.3715g $K(SbO)\ C_4H_4O_6 \cdot \frac{1}{2}H_2O$ in 400mL DI water in a 500mL volumetric flask and dilute to volume. Store at 4 ± 2°C. Expires one month from date of preparation.

8.11 Ammonium Molybdate Solution: Dissolve 10g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 250mL distilled water. Store at 4 ± 2°C. Expires one month from date of preparation.

8.12 Ascorbic Acid, 0.1M: Dissolve 3.52g ascorbic acid in 200mL DI water. The solution is stable for about 1 week at 4 ± 2°C.

8.13 Orthophosphate 1000ppm solution Independent, source standard. Store at 4 ± 2˚C. Expires upon manufacturer's specified date.

8.14 Orthophosphate 25ppm spike solution Add 2.5mLs 1000ppm Orthophosphate solution to a clean, glass 100mL volumetric and dilute to volume with DI. Store at 4 ± 2°C. Expires 6 months from date of preparation.

8.15 SEAL Working Ascorbic Acid, 15g/L (with orthophosphate spike): Dissolve 1.5g of ascorbic acid in about 80mL DI water. Spike with .15mL 25ppm Orthophosphate standard to produce a spike level of .025mg P/L. Dilute to 100mL and mix well. The solution is stable for one week if stored at 4 ± 2°C. Discard if the solution becomes yellowed.

8.16 Combined Reagent: Mix 8.9, 8.10, 8.11, and 8.12 in the following proportions for 100mL of the combined reagent: 50mL 5N H_2SO_4 (Section 8.9), 5mL potassium antimonyl tartrate solution (Section 8.10), 15mL ammonium molybdate solution (Section 8.11), and 30mL ascorbic acid solution (section 8.12). Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours. Discard reagent if it turns blue or black in color.

8.17 SEAL Working Coloring Reagent: To a clean 100mL volumetric flask, add 40mL sulfuric acid (8.9), followed by 6.5 mL antimony potassium tartrate (8.10) and swirl to mix. Then, add 20 mL ammonium molybdate (8.11). Swirl the contents, fill the flask up to the mark with DI water and mix well. Expires three weeks from day of preparation if stored at 4 ± 2°C. Discard if the reagent turns blue or becomes turbid.

8.18 Sodium Hydroxide, 6N: Dissolve 240 grams of NaOH pellets in 1000mL of DI water. Store at room temperature. Expires one month from date of preparation.

8.19 Phenolphthalein Indicator: Aqueous solution, commercially available. Store at room temperature. Expires upon manufacturer's specified date.

8.20 11N Sulfuric Acid Solution: Dilute 308mL concentrated sulfuric acid to 1000mL with DI. Store at room temperature. Expires 6 months from date of preparation.

8.21 Potassium Persulfate ($K_2S_2O_8$): Commercially available. Store at room temperature. Expires upon manufacturer's specified date.

8.22 Deionized Water

8.23 Soil LCS/SRM ERA Standard Reference Material for Nutrients in soil, catalog no. 542

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Method Blank/Calibration Blank - One method blank, which consists of DI water brought through the entire method, must be analyzed per batch of 20 samples or less. The CCB is analyzed after every 10 samples and at the end of the sequence.

Results of the Blanks must be less than the reporting limit. Otherwise the entire batch of samples must be re-prepared and reanalyzed. Exceptions are samples with results of more than 10 times the positive blank value.

Soil blanks are made with 0.1gm boiling chips and 50mLs of DI water and are analyzed like water blanks.

9.2 Laboratory Control Sample (LCS)

Analyze one per batch of 20 samples or less. The calibration curve must be verified by a second source standard prior to performing any sample analysis. For Total and Dissolved Phosphorus, the LCS is the ICV.

The ICV/LCS must be recovered within 90-110% of the true value. If the ICV/LCS fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

Soil LCS's are made with approximately 0.15g of Standard Reference Material (SRM) brought up to 50mL with DI water.

9.3 Initial Calibration Verification (ICV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. For Total and Dissolved Phosphorus, the ICV is the LCS.

The ICV/LCS must be recovered within 90-110% of the true value for water samples and be within vendor criteria for SRM. If the ICV/LCS fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

9.4 Continuing Calibration Verification (CCV)

The calibration curve must be verified by a second source standard. The CCV is analyzed after every 10 samples and at the end of the sequence to verify the curve.

The CCV must be recovered within 90-110% of the true value. If the CCV fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

9.5 Matrix Spike

Analyze one per batch of 20 samples or less. Concentration is 0.5 mgP/L. The matrix spike must be recovered within 75 – 125% of the true value. If the matrix spike recovery is outside acceptance criteria, and the LCS is acceptable, a narrative is submitted with the data for inclusion on the client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of 20 samples or less. The RPD must be ≤ 20%. If this criterion is not met, a narrative is submitted with the data for inclusion on the client report.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

9.8.1 Using spectrophotometer:

- Acid-rinse all glassware
- Calibration curve generation.
- Filter samples if analysis is for Dissolved Phosphorus, then preserve with H_2SO_4.
- Add 50mL of water sample or 0.1g of soil sample and 50mL DI water to an Erlenmeyer flask.
- Add 1mL H_2SO_4 solution and scoop solid $K_2S_2O_8$ and glass beads
- Boil down to 10mL or less.
- Cool and dilute to 30mL with DI.
- Add 1 drop phenolphthalein indicator solution.
- Neutralize to faint pink color with NaOH.
- Add sample aliquot to a new centrifuge tube and bring up to 50mL with DI.
- Add 4 mL combined reagent to a 25mL aliquot of sample.
- Read sample absorbance after 10-30 minutes.
- Analyze CCV and CCB after every 10 samples to verify curve.
- End sequence with CCV and CCB.
- Calculate results.

9.8.2 Using SEAL AQ2 analyzer:

- Acid-rinse all glassware
- Filter samples if analysis is for Dissolved Phosphorus, then preserve with H_2SO_4.
- Add 50mL of water sample (soils are not done on the SEAL) to an Erlenmeyer flask, along with a 1.0ppm calibration standard for the calibration curve.
- Add 1mL H_2SO_4 solution and scoop solid $K_2S_2O_8$ and glass beads
- Boil down to 10mL or less.
- Cool and dilute to 50mL with DI.
- Turn on the SEAL AQ2 analyzer.
- Fill out a run sequence and save it.
- Fill cups, including a cup of 1.0ppm digested standard for the curve
- Start the analysis.
- Change names of blank and LCS to be what the LIMS will recognize (ie, what is on the batch sheet).
- Export the data to LIMS by dropping it into the “SEAL on bowzer” folder.

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation for Dissolved Phosphorus Analysis

Prior to preservation, samples to be analyzed for dissolved phosphorus are filtered using new disposable syringes and new 0.45um filter discs. 100mL of sample is filtered, poured into two new centrifuge tubes and preserved with H_2SO_4.

10.2 Initial Calibration

10.2.1:

Preparation of calibration curve, with spectrophotometer: Prepare individual calibration curve from a series of six digested standards (0 mgP/L to 1.0 mgP/L) on each day of analysis. The curve must be digested. Use DI water without the combined reagent to zero the Spectrophotometer. Plot absorbance vs. phosphate concentration to give a straight line. The correlation coefficient must be 0.995 or greater for the curve to be considered valid. Analyze at least one phosphate standard with each batch of 20 samples or less.
All calibration points are back calculated (on excel) and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

10.2.2:

Preparation of calibration curve, with SEAL AQ2 analyzer: Prepare and digest a 1.0 mgP/L standard and put it into the first slot in the auto-sampler. When prompted, click on "Auto-calibrate" to start calibration. Once the curve is finished, it may be checked in the "calibration" section. The correlation coefficient must be 0.995 or greater for the curve to be considered valid.
. All calibration points are back calculated by SEAL software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

10.3 Equipment Operation and Sample Processing, with spectrophotometer

10.3.1 Add 50mL or a suitable portion of thoroughly mixed sample to a prepared (Section 7.2) 125mL Erlenmeyer flask. Use 0.1g of soil sample with 50 ml of DI for soil samples.

10.3.2 Add 1mL H_2SO_4 solution, one scoop solid $K_2S_2O_8$ and 3 to 5 glass beads.

10.3.3 Boil gently on a preheated hot plate until a final volume of 10mL or less is reached. Organophosphorus compounds such as AMP may require as much as 1-1/2 to 2 hours for complete digestion.

10.3.4 Cool, dilute to 30mL with DI water.

10.3.5 Add 0.05mL (1 drop) phenolphthalein indicator solution.

10.3.6 Neutralize to a faint pink color with NaOH.

10.3.7 Pour pink liquid sample into a new (unused) centrifuge tube and bring volume to 50mL with DI. Pour back into a 125mL Erlenmeyer flask.

10.3.8 Swirl the sample to mix and pour off 25mL digested sample into centrifuge tube.

10.3.9 Add 4.0mL combined reagent to all 25mL sample and QC sample aliquots and mix thoroughly.

10.3.10 After at least 10 minutes but no more than 30 minutes, use DI as the reference solution to zero the spectrophotometer at 880nm. Measure absorbance of each sample and record in the electronic laboratory notebook. If samples seem to have high background color before the addition of the coloring reagent, a background color may be checked for (see section 4).

If the sample concentration is greater than the highest concentration of the calibration curve (1.0mg/L), the digested sample is diluted with DI water to a concentration within the range of the calibration curve.

10.4 Equipment Operation and Sample Processing, with SEAL AQ2 Analyzer

10.4.1 Acid rinse all glassware twice with 1:1 hydrochloric acid and then twice with DI water.

10.4.2 Pour out 50mLs of mixed samples and QC samples, including a 1.0ppm calibration standard.

10.4.3 Add 1mL H_2SO_4 solution, one scoop solid $K_2S_2O_8$ and 3 to 5 glass beads.

10.4.4 Boil gently on a preheated hot plate until a final volume of 10mL or less is reached. Organophosphorus compounds such as AMP may require as much as 1-1/2 to 2 hours for complete digestion.

10.4.5 Cool and dilute to 50mLs.

10.4.6 Turn on SEAL AQ2 analyzer by flipping first the small, and then the large switch in the back.

10.4.7 Give the instrument at least half an hour to warm up.

10.4.8 If it has not yet been done that day, go through daily start-up, check voltages, and test aspiration.

10.4.9 Double click on scheduling to open the schedule form, and insert samples, Method Blanks, LCSs, Duplicates, and Matrix Spikes (the CCVs and CCBs populate automatically) NB: leave the first spot for the calibration curve.

10.4.10 Pour a small (approximately 1 mL) aliquot into small tubes and put them into the instrument. Put the 1.0ppm standard into the first spot.

10.4.11 Check to see that all the reagent wedges are in the correct spots and that there are sufficient reaction segments.

10.4.12 Save the sequence and double click on “run”; check the boxes for curve analysis.

10.4.13 Once the instrument is done analyzing the run, check and approve the results in the Data Review section. Then, change the Blank and LCS names to be whatever they are on the batch sheet (ex: WG123456-1) and save the run in the “out” folder.

10.4.14 Open the “SEAL on bowzer” folder and drop the run into it from the “out” folder. This saves the data to LIMS.

10.5 Continuing Calibration

The method blank and LCS are used as the CCB/CCV and should be read after every ten samples and at the end of the batch. Recovery for the CCV must be between 85-115% of the true value. Recovery for the CCB must be between the RL and its negative, (i.e: within -.01mg/L and .01mg/L for waters).

10.5 Preventative Maintenance

The Spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

Calculate the concentration value of the sample directly from the standard curve. (Section 10.2).

$$\text{mg } P_{Total}/L = \frac{\text{absorbance} - \text{y-intercept}}{\text{slope}} \times \text{Dilution factor}$$

If samples were filtered prior to preservation, report as mg $P_{\text{Dissolved}}$ / L.

For soil samples, convert results to mg/kg, by multiplying result in mg/l by extraction volume and dividing by weight. All results must by reported based on dry weight.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances or improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

SOP CHANGE FORM

SOP Title: NITROGEN, Total Kjedahl R	
SOP Code: GEN - 351.2	
SOP Revision No.: 6.	
SOP Date: 6/1/14.	
SOP Section(s) Affected by Change: 10.3.3.	

Description of Change: standard 0.05ppm removed from analysis on Lachat 8500.

Change(s) Submitted by: Gabriela Nita - Jouppi	Date: 12/17/19.

Approvals:

Technical Reviewer Signature:	Date: 12/17/19
QA Signature:	Date: 12/19/19
Laboratory Director Signature:	Date: 12/30/19

Change(s) Effective Date: 12/17/19

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title:	All SOPs for Inorganics which have a Matrix Spike
SOP Code:	
SOP Revision No.:	
SOP Date:	
SOP Section(s) Affected by Change:	

Description of Change:

Add to section 3. Matrix Spike Duplicate (MSD or DMS) – A duplicate of the Matrix Spike. The RPD between the MS and the MSD are evaluated for precision.

Section 10 - Add MSD to the MS preparation.

Section 16 - Add that the lab is using MSD instead of a Sample Duplicate as standard practice for matrix QC.

Reason(s) for Change(s):
More clients requesting MSD for inorganics. Change for consistency.

Change(s) Submitted by: Vicky Collom	Date: 12/14/16

Approvals:

Technical Reviewer Signature:	Date:
QA Signature:	Date: 12/14/16
Laboratory Director Signature:	Date: 12/15/16

Change(s) Effective Date: 12/16/16

SOP user acknowledgement:

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Nitrogen, Total Kjeldahl Block Digestion and Analysis by Flow Injection

SOP Code: GEN-351.2

SOP Revision No.: 6

SOP Date: 6/1/14

SOP Section(s) Affected by Change: 12.1, 16.11

Description of Change:

12.1 - add - in the comments section of the Digest sheet, document any observations which may impact the validity of the results (for example, but not limited to: boil-over, boiling stones missing, low final digest volume, unusual color).

16.11 - add - if a prep batch is discarded due to failing QC, or other evidence of contamination, evaluate the potential impact on any other batch digested in the same block at the same time. Re-digest both batches if gross contamination is evident (MB>2xMRL, LCS>150%, ~~RPD>40~~), or if boil-over or popping/spattering is observed in either batches. CK6/6/16 6/7/16

Reason(s) for Change(s):
RC0337.

Change(s) Submitted by: Vicky Collom	Date: 6/6/16

Approvals:

Technical Reviewer Signature:	Date: 6/6/16
QA Signature:	Date: 6/6/16
Operations Manager Signature:	Date: 6/7/16

Change(s) Effective Date: 6/6/16

SOP user acknowledgement:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Nitrogen, Total Kjeldahl Block Digestion and Analysis by Flow Injection

SOP Code: GEN-351.2

SOP Revision No.: 6

SOP Date: 6/1/14

SOP Section(s) Affected by Change: 16.2, 12.1.10, 16.10, 21

Description of Change:

12.1.10 Remove requirement to place digestates in cooler.

16.2 and 16.10 MB - Add DOD limit of <1/2LOQ.

21 – Add to References – Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013

Reason(s) for Change(s):
12.1.10 Digestates occasionally crystallize. Refrigeration may contribute to the problem.
16, 21 Addition to DOD scope.

Change(s) Submitted by: Vicky Collom	Date: 10/14/15

Approvals:

Technical Reviewer Signature:	Date: 10/15/15
QA Signature:	Date: 10/15/15
Operations Manager Signature:	Date: 10/15/15

Change(s) Effective Date: 10/14/15

SOP user acknowledgement:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Nitrogen, Total Kjedahl Block Digestion and Analysis by Flow Injection
SOP Code: GEN-351.2
SOP Revision No.: 6
SOP Date: 6/1/14
SOP Section(s) Affected by Change: 11.3

Description of Change:
Add new section after 11.3 – Verify the linear calibration range by refitting the high curve point to the curve. If the point is outside of 90-110 % of its true value, linearity must be reestablished. For the 8000, rerun the high standard for a LRC (Linear Range Check).

Reason(s) for Change(s):

NYSDOH audit RC0236.

Change(s) Submitted by: Vicky Collom	Date: 5/15/15

Approvals:

Technical Reviewer Signature:	Date: 5/15/15
QA Signature:	Date: 5/15/15
Laboratory Director Signature: Peter Frank	Date:5/15/15

Change(s) Effective Date: 4/13/15

SOP user acknowledgement:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Nitrogen, Total Kjedahl Block Digestion and Analysis by Flow Injection
SOP Code: GEN-351.2
SOP Revision No.: 6
SOP Date: 6/1/14
SOP Section(s) Affected by Change: 12.1.5, 12.1.6, 12.1.7

Description of Change:

12.1.5, 12.1.6, 12.1.7 Add temperature routine for different methods:

	351.2	ASTM D3590	SM4500NorgD
Preheat (°C)	160	200	200
Digest at Preheat temp	60 min		
Ramp, hold at 380 for:	30 min	90 min	60 min

Reason(s) for Change(s):

Did not change over to Standard Methods as planned. Need options in SOP until switch is made.

Change(s) Submitted by: Vicky Collom	Date: 6/6/14

Approvals:

Technical Reviewer Signature:	Date: 6/6/14
QA Signature:	Date: 6/12/14
Laboratory Director Signature:	Date: 6/19/14

Change(s) Effective Date:6/1/14

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

ALS Standard Operating Procedure

DOCUMENT TITLE:	NITROGEN, TOTAL KJELDAHL BLOCK DIGESTION AND ANALYSIS BY FLOW INJECTION
REFERENCED METHOD:	EPA 351.2, SM 4500 NORG D, ASTM D3590(B)-02
SOP ID:	GEN-351.2
REV. NUMBER:	6
EFFECTIVE DATE:	6/1/14

ALS Environmental

NITROGEN, TOTAL KJELDAHL BLOCK DIGESTION AND ANALYSIS BY FLOW INJECTION

EPA 351.2, SM 4500 NORG D, ASTM D3590(B)-02

SOPID: GEN-351.2 Rev. Number: 6 Effective Date: 6/1/14

Approved By: Department Supervisor – Christine Kutzer Date: 5/9/14

Approved By: QA Manager – Lisa Reyes Date: 5/12/14

Approved By: Laboratory Director – Larry Lewis Date: 5/12/14

Archival Date: ________ Doc Control ID#: 14-GEN-01 5/13/14 Editor: ________

TABLE OF CONTENTS

NITROGEN, TOTAL KJELDAHL BLOCK DIGESTION AND ANALYSIS BY FLOW INJECTION

1) Scope and Applicability

1.1 This SOP is for the block digestion and colorimetric determination of total Kjeldahl nitrogen using EPA method 351.2, SM 4500-Norg D – 1997(11), or ASTM D3590(B)-02 in drinking, ground, and surface waters, domestic and industrial wastes, and soils. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbozones and some refractory tertiary amines.

1.2 The applicable range is 0.05 to 10.0 mg/L TKN. The range may be extended with sample dilution. For waters, samples requested for low level have a PQL of 0.10 mg/L TKN while samples requested for regular level have a PQL of 0.20 mg/L TKN. Soils and waste samples have a PQL of 20 mg/kg.

2) Summary of Procedure

2.1 The sample is heated in the presence of sulfuric acid (H2SO4) in a block digestor. Copper sulfate is used as a catalyst, and potassium sulfate is added to raise the boiling temperature of the digestion mixture. The residue is cooled, diluted to original volume, and analyzed by flow injection (Lachat).

2.2 Total Kjeldahl nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate ((NH4)2SO4) during the digestion procedure.

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

3) Definitions

3.1 Independent Calibration Verification (ICV) - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.2 Matrix Spike (MS) - In the matrix spike analysis, a predetermined quantity of a standard solution of the analyte is added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recovery is calculated for the analyte detected.

3.3 Duplicate Sample (DUP) - A laboratory duplicate. The duplicate sample is a separate sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.4 Method Blank (MB) - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.5 Continuing Calibration Verification Standard (CCV) - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.6 Instrument Blank (ICB or CCB) - The instrument blank (also called initial or continuing calibration blank) is a volume of blank reagent of composition identical to the digestates that has not been digested. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

3.7 Laboratory Control Sample (LCS) - An aliquot of reagent water to which a known quantity of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.8 Organic Blank Spike - a predetermined quantity of an organic standard solution of the analyte is added to a blank prior to sample digestion and analysis. This standard monitors the breakdown of organically bound Nitrogen to ammonia. Percent recoveries are calculated for the analyte detected.

3.9 Batch – A group of no more than 20 samples that are analyzed together on the same day. See ADM-BATCH for more information.

3.10 Limit of Quantitation (LOQ) / Reporting Limit – The minimum levels, concentrations, or quantities of a target that can be reported with a specified degree of confidence. For DOD, the lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard.

3.11 Limit of Detection (LOD) – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory – dependent. For DOD, the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

4) Health and Safety Warnings

4.1 All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

4.2 Chemicals, reagents and standards must be handled as described in the company safety policies, approved methods and in MSDSs where available. Refer to the Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method. Sodium Nitroprusside ($Na2Fe(CN)5NO\cdot 2H2O$) has the potential to be highly toxic or hazardous.

4.3 Sulfuric Acid is used in this method. This acid is extremely corrosive and care must be taken while handling it. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these.

4.4 Refer to the Safety Manual for further discussion of general safety procedures and information.

4.5 Waste Management and Pollution Prevention

4.5.1 It is the laboratory’s practice to minimize the amount of acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when disposed of properly.

4.5.2 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the EH&S Manual.

4.5.3 Samples are disposed according to SMO-SPLDIS.

5) Cautions

5.1 Be sure that all digestion tubes are thoroughly cleaned and rinsed.

5.2 Block digestors should be inspected and cleaned whenever necessary.

5.3 Nearly all of the components of the Lachat can be easily cleaned or replaced. The exception is the valve. When the valve becomes clogged it is necessary to have the unit sent out to be serviced. To avoid this expense and inconvenience, be sure sample cups, and dispo cups are free of particulates by rinsing thoroughly with D.I. water and drying. Visual inspection of this equipment is also recommended before analysis. Turbid samples should also be filtered to prevent valve clogs.

5.4 Be sure to change pump tubes regularly to ensure optimal performance.

5.5 All changes in tubing, hardware, or programming are to be noted in the maintenance logbook. Record the first acceptable run in the logbook after major maintenance.

5.6 Keep the instrument and the bench area clean. Wipe down counters before and / or after use.

5.7 When analyzing on the Lachat, be sure to introduce the buffer solution and 0.8M NaOH solution into the manifold before the other reagents to prevent the formation of clogs due to reagent precipitate. Also be sure to rinse all reagent lines prior to rinsing the buffer line.

5.8 Be sure all glassware is clean. See GEN-GC.

6) Interferences

6.1 High nitrate concentrations (>10 times the TKN level) result in low TKN values. If interference is suspected, samples should be diluted and redigested and analyzed.

6.2 Method interferences may be caused by contaminates in the reagent water, reagents, glassware, and any other sample processing apparatus that bias analyte response. Airborne ammonia can be rapidly absorbed by the digestion solution.

6.3 Samples high in organics may consume the sulfuric acid during digestion, resulting in a matrix effect on the pH-dependent color reaction.

7) Personnel Qualifications and Responsibilities

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

Training - Follow policies in the SOP for Training (CE-QA003).

8) Sample Collection, Containers, Preservation, and Storage

8.1 Samples should be collected in plastic or glass bottles. All bottles should be new and clean. Volumes collected should be sufficient to insure a representative sample, allow for replicate analysis (if necessary), and minimize waste disposal. Bottles supplied by the lab are typically certified clean 250 mL HDPE bottles.

8.2 Samples must be preserved with H2SO4 to a pH < 2 and cooled to 0-6°C at the time of collection.

8.3 Preserved samples are maintained at 0-6°C and may be held up to 28 days. For ASP, preserved samples may be held for up to 26 days from VTSR (Verified Time of Sample Receipt)

8.4 See SMO-GEN and SMO-ICOC for further sample handling, storage, and custody procedures.

9) Equipment and Supplies

9.1 Block digestor(s) with adequate number of digestion tubes.

9.2 Glassware - Class A volumetric flasks and pipets as required.

9.3 Micropipet, adjustable 100-1000μL. See ADM-PCAL for calibrating.

9.4 Rack for holding and cooling of digestion tubes.

9.5 Vortex mixer.

9.6 Balance - Analytical, capable of accurately weighing to the nearest 0.0001g. Calibrated according to ADM-DALYCK.

9.7 Calibrated repipetor (20.0 and 8.0 mL capacity).

9.8 Disposable 25 mL pipets.

9.9 Disposable Beakers (dispo-cups).

9.10 Sample Containers (B-cups).

9.11 Teflon Boiling Chips.

9.12 Instrument:

Instrument ID	Instrument Configuration	Manufacturer Part	Serial Number	Year Acquired
Lachat 8000 (R-FIA-01)	Flow Injection System	Lachat 8000		1999
	Colorimeter	Lachat	A83000-1286	
	Pump	Lachat	A82000-525	
	Autosampler	Lachat	A81010-168	
	Computer Workstation	Gateway GP6-233	9767124	
	Analytical Software	Omnion FIA v.2	-	
Lachat 8500 (R-FIA-05)	Flow Injection System	Lachat 8500		2011
	Colorimeter		110100001295	
	Cell	BASi CC-3D	11314	
	Pump	14951	0595996-2	
	Autosampler	ASX-260	021109A260	
	Computer Workstation	Dell Optiplex 780		
	Analytical Software	Omnion FIA v.3.0	-	

10) Standards and Reagents

10.1 Standards Preparation General Information and Disclaimers

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 ppm standard may be made by adding 1 mL of 200 ppm to 10 mLs or may be made by adding 4 mL of 50 ppm to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

The initial calibration curves given are typical, but also subject to variation due to detection levels needed. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

All Standards must be traceable using the laboratory lot system (CE-QA007)

10.2 Purchased reagents and neat standards: Store at room temperature unless otherwise indicated below. Expire per Expiration Policy (CE-QA012) if no other indication is given.

- Ammonium Chloride (NH4Cl) –Pre-dry at 103-105 °C for 2 hours. Allow to cool and store in a desiccator at room temperature.
- Potassium Sulfate (K2SO4)
- Copper Sulfate (CuSO4)
- Sulfuric Acid (H2SO4) – Instra-analyzed
- Sodium Phosphate Dibasic Heptahydrate (Na2HPO4·7H2O)
- Disodium EDTA (C10H14N2Na2O8·2H2O)
- Sodium Hydroxide (NaOH) – pellets
- Sodium Salicylate (HOC6H4COONa)
- Sodium Nitroprusside ((Na2Fe(CN)5NO·2H2O)
- Sodium Hydroxide flakes (NaOH) – purchased commercially with special attention to ammonia content
- L-Glutamic Acid – pre-dry for one hour at 103-105˚C. Allow to cool and store in a desiccator at room temperature.
- Hydrochloric Acid (HCl) – Instra-Analyzed

10.3 Prepared Standards and Reagents – expire per Expiration Policy (CE-QA012) if no other indication is given.

10.3.1 Nitrogen Calibration Standard Stock Solution (1000 mg N/L): In a 1-L volumetric flask, dissolve 3.819 g dried Ammonium Chloride in about 800 mL reagent grade water (DI). Dilute to volume with DI water and invert several times until thoroughly mixed. Store at 0-6°C in amber glass. **Note: This is the same standard as is prepared for ammonia analysis. The same stock may be used for both analyses.

10.3.2 Working Stock Solution (10.0 mg N/L): In a 100 mL volumetric flask, add 1.00 mL of 1000 mg/L standard stock solution and dilute to volume with PDMM and invert several times to mix. Prepare fresh for each run.

10.3.3 Calibration Standards: Working standards are prepared directly into PDMM and analyzed without digestion. Prepare standards in disposable beakers immediately prior to the analysis as follows:

Concentration (ppm)	PDMM (mL)	Working Stock 10 ppm (mL)
10.0	0	10.0
5.00	5.00	5.00
2.00	8.00	2.00
1.00	9.00	1.00
0.50	1/10 dilution of 5.0 std	
0.20	1/10 dilution of 2.0 std.	
0.10	1/10 dilution of 1.0 std.	
0.05	1/10 dilution of 0.5 std.	
0.00	10.0	0.00

10.3.4 Nitrogen Calibration Reference Stock Solution (400 mg/L): In a 1-L volumetric flask dissolve 1.5276g dried Ammonium Chloride from a separate source as was used for the standard stock solution in about 800 mL reagent grade water (DI). Dilute to volume with DI water and invert several times until thoroughly mixed. Store at 0-6°C in amber glass.

10.3.5 ICV/CCV (4.0 ppm): In a disposable beaker, add 0.10 mL 400 ppm Reference Stock Solution to 9.90 mL PDMM. Prepare fresh each run.

10.3.6 Method blank: Analyze DI as a sample.

10.3.7 LCS / Matrix Spike (2.5 mg/L): Pipet 20 mL of DI water /sample into a digestion tube. With an adjustable micropipet, add 0.050 mL of 1000 ppm Calibration Standard Stock. Digest along with the other samples. Prepare fresh each run.

10.3.8 Organic Nitrogen Spiking Solution (500 mg/L): In a 1 Liter volumetric flask, dissolve 5.252 g pre-dried L-Glutamic acid in DI. Bring to volume with DI and mix by inversion.

10.3.9 Organic Blank Spike – (2.50 mg/L) – Add 0.100 mL of 500 mg/L Organic Nitrogen Spiking Solution to 20 mL DI. Digest as a sample.

10.3.10 Digest Solution: To a 2-L volumetric flask, dissolve 268 g Potassium Sulfate and 14.6 g Copper Sulfate in about 1600 mL of DI water. While stirring, slowly add 268 mL Sulfuric Acid. Stir until cool. Bring to volume with DI water and invert several times until thoroughly mixed. Store at room temperature in amber glass. Expires in one month.

10.3.11 Post Digestion Matrix Match (PDMM): To a 2-L volumetric flask, add 800 mL of digest solution and bring to volume with DI water. Invert several times until thoroughly mixed. Decant 100 mLs, then bring to volume again with DI. Store in amber glass and at room temperature. Expires in one month.

10.3.12 Buffer Solution: To a 1-L volumetric flask add about 900 mL DI water. Next, add 35.0 g $Na2HPO4 \cdot 7H2O$, 20.0 g Disodium EDTA and 50.0 g NaOH pellets. Stir until dissolved. Dilute to volume with DI water and invert several times until thoroughly mixed. Store at room temperature in amber glass. Discard after one month.

10.3.13 Salicylate Nitroprusside Color Reagent: To a 1-L volumetric flask dissolve 150.0 g Sodium Salicylate and 1.00 g Sodium Nitroprusside in about 800 mL DI water. Stir until dissolved. Dilute to volume with DI water and invert several times until thoroughly mixed. Store at room temperature in amber glass. Discard after one month.

10.3.14 0.8 M Sodium Hydroxide: In a 1-L volumetric flask, dissolve 32.0 g Sodium Hydroxide flakes in about 800 mL DI water. Stir until dissolved. Dilute to volume with DI water and invert several times until thoroughly mixed. Store at room temperature in a plastic bottle.

10.3.15 Hypochlorite Solution: In a 250-mL volumetric flask dilute 15.0 mL 4-6% sodium hypochlorite to the mark with DI water and invert to mix. Pour into a plastic bottle. Prepare fresh for each run. Do not use Chlorox Bleach.

10.3.16 50/50 HCl: To a clean 2.5-L acid jar add 1.25 L of DI water. While wearing a face shield, carefully add 1.25 L of concentrated Instra-Analyzed Hydrochloric Acid (HCl).

11) Method Calibration

11.1 Follow ADM-ICAL unless otherwise specified in this SOP.

11.2 After the last standard has been analyzed, review the calibration data. Prepare a linear regression using the equation y=mx+b. Use 1/x weighting as per the manufacturer.

11.2.1 The correlation coefficient must be 0.997 or better for the analysis to continue. If a linear curve is not achieved, stop the analysis, correct the problem, and recalibrate.

11.2.2 Due to limitations of the software, once a calibration is complete, standards may not be reanalyzed. Since there are legitimate times (bad integration, air spike in peak) when it is necessary to repeat a standard, include two injections of the zero and the two lowest standards. The software is selected to average the replicates of the injections. Points may only be dropped due to assignable cause as detailed in ADM-ICAL and the cause must be documented in the raw data.

11.3 After an acceptable calibration is achieved, verify the calibration with the ICV and ICB. If these are acceptable, continue with sample analysis. Each sample must be “bracketed” by an acceptable CCV/CCB (or ICV/ICB) set. If a CCV or CCB fails, all samples back to the last good set must be reanalyzed after the problem is corrected and a good set is obtained.

11.4 For all samples in which the analyte value has exceeded the high standard, proper dilutions must be made of the sample to bring it within the range of the calibration standards.

11.5 When finished analyzing samples and QC items, place feedlines in water except the buffer and NaOH lines and rinse for a couple of minutes. Place the remaining lines in DI and rinse for 5 minutes. Remove all lines from DI and pump to dry for about 5 minutes. Turn off the pump and release tension on pump tubes.

11.6 Print the results and the calibration information.

12) Sample Preparation and Analysis

12.1 DIGESTION PROCEDURE:

**Note: Preheat the digestion blocks to 200°C before proceeding.

12.1.1 Thoroughly wash digestion tubes in hot soapy water and rinse 3 times with hot tap water. Rinse once with 50/50 HCl and 4 or 5 times with DI water. Place clean tubes in rack and invert to dry tubes.

12.1.2 Once tubes are dry, add 20.0 mL of sample, or an aliquot diluted to 20.0 mL, to the digestion tube. For soil samples, place 0.20 g of soil into digestion tube and bring to 20.0 g with DI water. Water samples are to be shaken unless they are from monitoring wells.

12.1.3 Add 8.0 mL of digest solution to all digestion tubes. In order to prevent cross-contamination, do not touch the tip of the pipet to the tube. Hold the pipet tip above the tube.

12.1.4 Add a scoop of Teflon boiling chips (about 4 chips per tube) to all tubes.

12.1.5 Put the end plates on the TKN tube rack. Transfer the loaded rack to the preheated block digestor for 1 hour at 200°C.

12.1.6 Adjust the block digestor control to 380°C and continue to monitor temperature. Once 380˚C is reached, digest the samples for an additional 60 minutes.

*Note: To adjust the block temperature with the Omega controller, press the temperature control button on the front of the temperature regulator in while turning it until the desired target temperature is displayed. Once the button is no longer pushed in, the LCD will display the actual block temperature.

12.1.7 After digestion time is complete, reset block digestors back to 200°C. Transfer the rack of tubes from the block digestors and set onto a second empty rack. Carefully remove the end plates and allow the tubes to cool for approximately 15 minutes.

**Warning: Exercise caution since all tubes contain hot sulfuric acid!!!

12.1.8 Carefully and slowly add 20.0 mL DI water using a calibrated repipetor (dispense down the inside of the tube rather than directly into the digest solution to prevent possible splattering and sample loss). Do not touch end of repipetor against inside of tube to prevent the possibility of cross contamination.

12.1.9 Mix on a Vortex mixer until all residue has been redissolved. If necessary, tubes may be reheated to help redissolve stubborn residue.

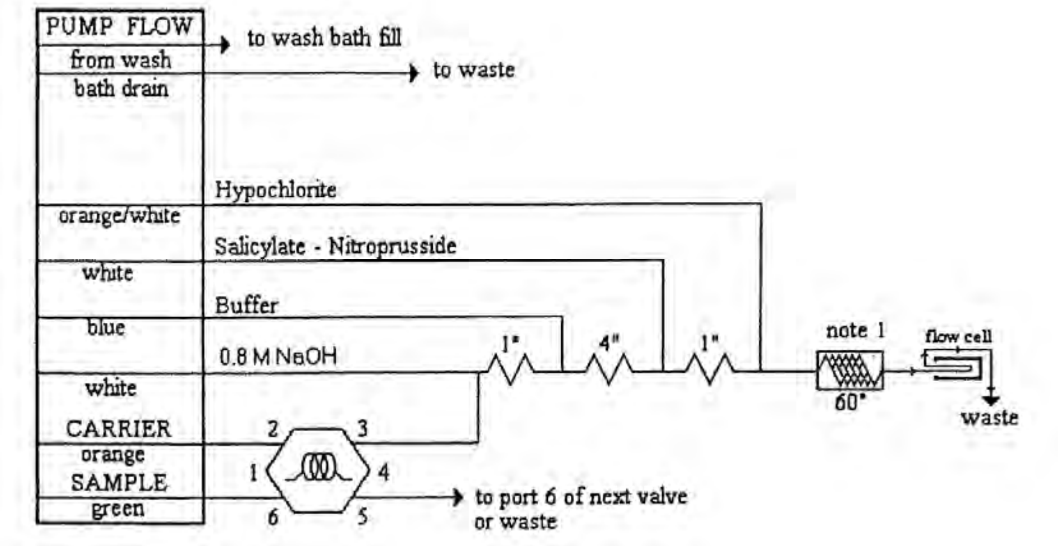
12.1.10 Transfer samples into labeled B-cups (High Level) or labeled VOA vials (Low Level) and cap tightly. Proceed with colorimetric analysis. Samples are stable at this point and may be stored overnight at room temperature before analysis is necessary. If analysis will not be done by the next day, place samples in 0-6°C cooler until analysis is completed.

12.2 COLORIMETRIC ANALYSIS:

12.2.1 Prepare standards and reagents as described in the Standards and Reagents Section.

12.2.2 Turn on the computer and Lachat components, including the heating block (60°C).

12.2.3 Connect the manifold to the instrument.



Carrier is Post Digestion Matrix Match

1” is 70.0 cm of tubing on a 1 inch coil support
4” is 255 cm of tubing on a 4 inch coil support

All manifold tubing is 0.8mm (0.032 in.)i.d. This is 5.2 uL/cm.

Note 1: 650 cm of Teflon tubing is wrapped on the heater block.

12.2.4 Inspect the manifold for proper connections, appropriate sample loop (30.5 cm), wavelength filter (660 nm), and heating coil length (650 cm). Place pump tubes loosely in their holders.

12.2.5 Load the appropriate method into the software and enter sample labels for the first tray in accordance with the analytical sequence described below. The method should be optimized from the suggested operating parameters in the Lachat instrument manual. The Lachat software will calculate all dilutions provided the dilution information is entered.

12.2.6 Analytical Sequence: Set up in accordance with ADM-BATCH and the QC Section in this SOP.

Analyze CRDLs on the Lachat 8000 to see the response at the low end of the curve since the software does not quantitate the standards in the calibration.

The sequence must end with a CCV/CCB set. Consult the QC section for further QC sample frequency requirements.

12.2.7 Load the calibration standards, and the samples into the autosampler as entered into the software with standards in order of decreasing concentration. Make sample dilutions in compliance with ADM-DIL.

12.2.8 For turbid samples or samples with particulates, filter through a syringe filter which has been shown to be free of TKN and shown not to remove TKN. Demonstrate these properties by analyzing a filtered blank and a filtered LCS.

12.2.9 Place all reagent lines in DI to rinse. Be sure reagent waste lines go to a collection vessel for proper disposal. Other lines may go to the sink.

12.2.10 Attach lines to pump collars. Turn on pump. Lock in collars. Rinse for a couple of minutes. Inspect for leaks. While still rinsing, allow heating coil temp to come back up to at least 59°C before placing lines in reagents. This cuts down on drift during the run.

12.2.11 With pump still engaged, place the buffer and NaOH feed lines in their appropriate reagent bottles. Pump for a couple of minutes. Add the rest of the feed lines to the appropriate reagents.

13) Troubleshooting

- Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by laboratory staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

14) Data Acquisition

14.1 Print a Responsibility Report from LIMS to determine what samples are available to be run. Prioritize samples to be run based on holding time and due dates. Scan the samples to be analyzed out of storage according to SMO-ICOC.

14.2 The data acquired is transferred via Omnion FIA software to LIMS electronically.

14.3 Specifics pertaining to data review are contained in ADM-DREV.

14.4 Data reporting is handled by the LIMS. More information is in ADM-RG

15) Calculation, and Data Reduction

15.1 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted according to ADM-DIL and reanalyzed.

15.2 Calculations: Sample concentration is calculated from the regression equation. Lachat software will calculate all results, including those with dilutions, provided that the correct dilution information was entered.

Result from curve x Digestion Factor x Dilution Factor

$$\text{Digestion Factor} = \frac{\text{Final Volume (mL) after digestion and dilution}}{\text{Initial weight (g)}}$$

15.3 Report results to three significant figures in mg N/L.

15.4 Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV.

15.5 If Total Organic Nitrogen (TON) is requested, subtract the ammonia result (GEN-350.1) from the TKN result.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 Ongoing verification of the LOD and LOQ are required. See CE-QA011 for requirements.

16.2 Acceptance Criteria:

	EPA 351.2	SM 4500 N-org D	ASTM D3590(B)-02
ICV	90-110%	90-110%	90-110%
CCV	90-110%	90-110%	90-110%
ICB/CCB	<1/2MRL	<1/2MRL	<1/2 MRL
LCS	90-110%	DQO (calculated)	85-115%
Organic blank spike	70-130% (lab limit)	70-130% (lab limit)	85-115%
Duplicate	20% RPD water 30%RPD soil	20% RPD water 30%RPD soil	20% RPD water 30%RPD soil
Matrix Spike	90-110%	DQO (calculated)	DQO (calculated)
MB	<RL	<RL	<1/2MRL

16.3 ICV – Analyze immediately after the calibration standards. The result of the ICV must be 90-110 % of the true value. If it is not, fix the problem and recalibrate if necessary.

16.4 CCV – Analyze every 10 samples or fewer. Recovery must be 90-110% of the true value, or the bracketed samples must be repeated.

16.5 ICB/CCB - Analyze one for every 10 or fewer samples. They must be less than the ½ reporting limit. If they are not, correct the problem and re-analyze the affected samples or raise the reporting limit.

16.6 LCS - Prepare one LCS for every 20 or fewer field samples. LCS recovery must be within the limits in the above Table. If the LCS is outside of these limits, redigest and reanalyze the affected samples or flag the affected data.

16.7 Organic Blank Spike - Prepare one per 20 samples digested or one per batch, whichever is more frequent. If the organic blank spike is outside the limits of the above table, correct the problem, redigest, and re-analyze the batch or flag the data. Samples less than the reporting limit may be reported with a high Organic Blank Spike.

16.8 Duplicates – One duplicate must be run for every 10 or fewer field samples. The relative percent difference (RPD) between matrix duplicates should be 20 or less for waters and 30 or less for soils. If the RPD of the duplicates is out of limits, repeat the sample and duplicate unless there is assignable matrix interference, historical failures, or lack of volume. If an out of control duplicate is not repeated, note the reason on the data quality checklist. If, at the time when the problem is discovered, the sample exceeds twice the holding time, discuss with supervisor or Project Manager prior to repeating the samples. Report all of the replicates and explain in the checklist for the case narrative.

16.9 Matrix Spike – One MS must be run for every 10 or fewer field samples. Matrix spike recovery must be within the limits in the above Table. If it is not, reanalyze the MS or flag the data.

16.10 Method Blank - Prepare a blank for digestion and analysis for every 20 or fewer field samples. The result of the MB must be less than the reporting limit or the sample hits need to be more than 10 x the MB hit. If it is not, redigest and reanalyze the affected samples, flag the data, or raise the reporting limit.

17) Data Records Management

Records are maintained according to CE-GEN003 and ADM-ARCH.

18) Contingencies for Handling Out-of-Control or Unacceptable Data

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, flag and narrate appropriately.

19) Method Performance

Detection and Quantitation limits are determined according to the requirements in CE-QA011.

Demonstration of Capability is performed according to CE-QA003. The documentation of this method performance is retained by the Quality Assurance office.

Interlaboratory Precision and Accuracy Data is available in the EPA method.

20) Summary of Changes

- Changed the digestion times and temperatures to match SM4500-Norg D.
- Updated Appendix to most current digest sheet

21) References and Related Documents

- ASTM D3590(B)-02
- Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA, EPA/600/R-93/100 August 1993.
- Lachat QuikChem Method [10-107-06-2-H].
- *Standard Methods for the Examination of Water and Wastewater*. Method 4500-Norg D - 1997 with 2011 editorial revisions.

22) Attachments

For example only – controlled separately on the Rochester Intranet:

- Digest Benchsheet
- Standards Prep Job Aid

ALS Environmental

Analyte: TKN Digest

Low Level / I

Analyst: ____________________ Date:

Pipet ID: ____________________ Spk Witness:

Balance ID: NA

#	Misc.	Order #	Sample Amt (mL/g)	Dilution	Spk amount	Comment
1		PB 1 LL	20	1		
2		LCS 1 INORG LL	20	1	0.050	1000 ppm
3		LCS 1 ORG LL	20	1	0.100	500 ppm
4			20	1		
5			20	1		
6			20	1		
7			20	1		
8			20	1		
9			20	1		
10			20	1		
11			20	1		
12			20	1		
13			20	1		
14			20	1		
15			20	1		
16			20	1		
17			20	1		
18			20	1		
19			20	1		
20			20	1		
21		PB 1 RL	20	1		
22		LCS 1 INORG RL	20	1	0.050	1000 ppm
23		LCS 1 ORG RL	20	1	0.100	500 ppm
24			20	1		
25			20	1		
26			20	1		
27			20	1		
28			20	1		
29			20	1		
30			20	1		
31			20	1		
32			20	1		
33			20	1		
34			20	1		
35			20	1		
36			20	1		
37			20	1		
38			20	1		
39			20	1		
40			20	1		
41			20	1		
42			20	1		
43			20	1		
44			20	1		
45			20	1		
46			20	1		
47			20	1		
48			20	1		
49			20	1		
50			20	1		

Template-TKNDigest-r1 12/9/13

TKN (10.00 - 0.000 (pql=0.05))

A.) 10 ppm WORKING STOCK:
1.00 ml of 1000 ppm NH_3 / TKN Standard Stock to 99.0 ml Post Digestion Matrix Match (PDMM) in a 100 ml volumetric flask.

B.) STANDARDS:

conc. (mg/l)	mls 10 ppm	mls PDMM
a.) 10.00	10.00	0
b.) 5.00	5.00	5.00
c.) 2.00	2.00	8.00
d.) 1.00	1.00	9.00
e.) 0.50	1/10 dil'n of b.) 5.00	
f.) 0.20	1/10 dil'n of c.) 2.00	
g.) 0.10	1/10 dil'n of d.) 1.00	
h.) 0.05	1/10 dil'n of e.) 0.50	
i.) 0.00	10 ml PDMM	

C.) ICV / CCV: (True Value = 4.00 mg/l)
9.90 mls PDMM + 0.10 mls 400.0 ppm TKN Reference Stock.

D.) LCS / Matrix Spike: (True Value = 2.5 mg/l)
digested with samples. 0.050 ml 1000 ppm TKN Spike added to 20.0 ml DI or sample

P:\INTRANET\QAQC\Forms Controlled\WC Stds TKN r1.doc 7/14/14

SOP CHANGE FORM

SOP Title: Ammonia by Colorimetry
SOP Code: GEN-350.1
SOP Revision No.: 6
SOP Date: 07/15/13
SOP Section(s) Affected by Change: 10.4.2

Description of Change: Liquid Sodium Phenolate now purchased from supplier as opposed to being prepared in the lab by the analyst in lieu of recent issues with reagent consistency.

Change(s) Submitted by: Michael Rogerson	Date: 05/07/2020

Approvals:

Technical Reviewer Signature:	Date: 5/7/2020
QA Signature:	Date: 5/7/20
Laboratory Director Signature:	Date: 5/7/2020

Change(s) Effective Date:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Ammonia by Colorimetry	
SOP Code: GEN-350.1	
SOP Revision No.: 6	
SOP Date: 7/15/2013	
SOP Section(s) Affected by Change: multi	

Description of Change:

	Instrument ID	Manufacturer	Serial Number	Year Acquired
9.5	Gallery (R-Discrete-02)	Thermo Fisher	861000110585	2017

10.2.4 Calibration Range:	Standard 1	0.0	
(Instrument Auto Dilute)	Standard 2	0.020ppm	(100x of Standard 8)
	Standard 3	0.050ppm	(40x of Standard 8)
	Standard 4	0.100ppm	(20x of Standard 8)
	Standard 5	0.200ppm	(10x of Standard 8)
	Standard 6	0.500ppm	(4x of Standard 8)
	Standard 7	1.00ppm	(2x of Standard 8)
	Standard 8	2.00ppm	

10.2.5 LCS / Matrix Spike / Matrix Spike Dup: 0.500ppm

10.2.7 ICV / CCV: 1.00ppm

12.5.5 Instrument Set-Up for Thermo Gallery
- 12.5.5.1 The test parameters are attached in the appendix
- 12.5.5.2 Switch on the Thermo Gallery
- 12.5.5.3 Switch on the workstation
- 12.5.5.4 Enter the software by clicking on the Thermo Gallery icon
- 12.5.5.5 Log into the software
- 12.5.5.6 Wait for the main screen
- 12.5.5.7 Click Startup
- 12.5.5.8 Run three extra rinses
- 12.5.5.9 Run water blank
- 12.5.5.10 Check water blank

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

12.5.5.11 Check and update the reagent rack according to procedure
12.5.5.12 Add new samples and select method in menu beneath sample ID. Set up the batch in accordance with ADM-Batch.
12.5.5.13 Fill cuvette loader when prompted

Change(s) Submitted by:	Date: 2/25/2020

Approvals:

Technical Reviewer Signature:	Date: 2/26/2020
QA Signature:	Date: 2/28/2020
Laboratory Director Signature:	Date:

Change(s) Effective Date:

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

Test parameters

350.1 NH3N Version number 1 . 7

Gallery 01

Date	2/25/2020	**User**	Lab
Time	5:12:33PM	**Software version: 6.0.1**	

Dilution

	Dilution with	Diluent
	Primary dilution 1 +	0
Neat sample		
	Dispense with	Extra
	Volume (µl)	20
Diluent		
	Diluent ID	0.04N H2SO4
	Dispense with	Extra
	Volume (µl)	20

Limits

	Measuring range (mg/l) Min	Max		Next dilution ratio (1+) Low	High
Primary dilution	*	2.0000000		*	4.0
2nd dilution	*	*		*	*
3rd dilution	*	*		*	*
4th dilution	*	*		*	*
Test limit	0.0000000	11.0000000	mg/l		
Critical limit	*	*	mg/l		
Init. abs.	*	2.5	A		

Calibration

Calibration type	Linear
Repeat time (days)	1
Points/calibrator	Single
Acceptance	Manual

Nbr	Calibrator	Current lot	Concentration	Dilution 1 +
1	NH3-0	Default	0.0000000	0
2	NH3-High	Default	2.0000000	99
3	NH3-High	Default	2.0000000	39
4	NH3-High	Default	2.0000000	19
5	NH3-High	Default	2.0000000	9
6	NH3-High	Default	2.0000000	3
7	NH3-High	Default	2.0000000	1
8	NH3-High	Default	2.0000000	0

Abs. error (A)	*
Rel. error (%)	*
Factor limit min.	*
Factor limit max.	*
Bias limit min.	*
Bias limit max.	*
Concentration axis	Linear
Response axis	Linear

QC

Test parameters

350.1 NH3N Version number 1.7 Gallery 01

Date	2/25/2020	**User**	Lab
Time	5:12:32PM	**Software version: 6.0.1**	

Procedure	NH3 Cal	QC profile	
Interval type		In use	Yes
Requests		Acceptance	Manual
Time (hh:mm)	0:00	Trigger	Calibration

Procedure	NH3 Run	QC profile	
Interval type	Requests	In use	Yes
Requests	10	Acceptance	Manual
Time (hh:mm)	0:00	Trigger	Interval

Procedure	Control	Current Lot	Conc.	SD	Req. count
NH3 Cal	NH3 ICV	Default	1.000000	0.1000000	1
NH3 Cal	NH3-ICVB	Default	0.000000	0.0500000	1
NH3 Run	NH3-CCV	Default	1.000000	0.1000000	1
NH3 Run	NH3-CCVB	Default	0.000000	0.0500000	1

Procedure	Nbr of controls	SD multiplier
NH3 Cal	1	1
NH3 Run	1	1

Wash definition

Preceding reagent	Following reagent	Wash type	Wash reagent	Tag
NH3-BUFF	All	Water	None	
NH3-PHEN	All	Water	None	

SOP CHANGE FORM

SOP Title: All SOPs for Inorganics which have a Matrix Spike

SOP Code:

SOP Revision No.:

SOP Date:

SOP Section(s) Affected by Change:

Description of Change:

Add to section 3. Matrix Spike Duplicate (MSD or DMS) – A duplicate of the Matrix Spike. The RPD between the MS and the MSD are evaluated for precision.

Section 10 - Add MSD to the MS preparation.

Section 16 - Add that the lab is using MSD instead of a Sample Duplicate as standard practice for matrix QC.

Reason(s) for Change(s):
More clients requesting MSD for inorganics. Change for consistency.

Change(s) Submitted by: Vicky Collom	Date: 12/14/16

Approvals:

Technical Reviewer Signature:	Date:
QA Signature:	Date: 12/14/16
Laboratory Director Signature:	Date: 12/15/16

Change(s) Effective Date: 12/16/16

SOP user acknowledgement:

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Ammonia by Colorimetry

SOP Code: GEN-350.1

SOP Revision No.: 6

SOP Date: 7/15/13

SOP Section(s) Affected by Change: 10.2.7, 10.2.5, 10.2.4

10.2.7 Description of Change: ICV/CCV 0.90mg/L to 0.50mg/L. Using a purchased 1000ppm Reference Standard make 2 1/10 dilutions using carrier diluent to make a 10ppm Reference working stock. Add 0.5ml of Reference working stock to 9.5ml carrier diluent.
More on back.

Reason(s) for Change(s): Using purchased 1000ppm Reference + Standard stocks. Linear range of 0-2.00 not possible on Lachat 8500.

Change(s) Submitted by: N. Mansen Nicol	Date: 1/7/16

Approvals:

Technical Reviewer Signature:	Date: 2/3/16
QA Signature:	Date: 2/3/16
Laboratory Director Signature:	Date: 2-5-16

Change(s) Effective Date: 1/13/16

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

LCS/Matrix Spike 0.500mg/L to 0.25mg/L. To 10ml of carrier or sample, add 0.25ml of 10X mg/L working stock solution. (10.2.5)

No 2.0ppm standard in curve. Range is 0.01–1.00ppm (10.2.4)

↑
Remove

SOP CHANGE FORM

SOP Title: Ammonia by Colorimetry

SOP Code: GEN-350.1

SOP Revision No.: 6

SOP Date: 7/15/13

SOP Section(s) Affected by Change: 11.1.4

Description of Change:
11.1.4 (new) – Verify the linear calibration range by refitting the high curve point to the curve. If the point is outside of 90-110 % of its true value, linearity must be reestablished. For the 8000, rerun the high standard for a LRC (Linear Range Check).

Reason(s) for Change(s):

NYSDOH audit RC0236.

Change(s) Submitted by: Vicky Collom	Date: 5/15/15

Approvals:

Technical Reviewer Signature:	Date: 5/15/15
QA Signature:	Date: 5/15/15
Laboratory Director Signature: Peter Frank	Date:5/15/15

Change(s) Effective Date: 4/13/15

SOP user acknowledgement:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: AMMONIA BY COLORIMETRY

SOP Code: GEN-350.1

SOP Revision No.: 6

SOP Date: 7/15/13

SOP Section(s) Affected by Change: 10.2.4,10.2.7,12.5.2

Description of Change:Only for Lachat 8500:
10.2.4 Linear range 0-1.00 ppm instead 0-2.00 ppm
10.2.7 ICV/CCV TV=0.45mg/L instead TV=0.9mg/L
12.5.2 back pressure loop40.5cm instead 200cm

Reason(s) for Change(s):
Using 0-2.00 ppm linear range 0.01mg/L standard (MRL) not detected.

Change(s) Submitted by: GNita	Date: 09/05/2014

Approvals:

Technical Reviewer Signature:	Date: 9/5/14
QA Signature:	Date: 9/5/14
Laboratory Director Signature:	Date: 9/8/14

Change(s) Effective Date: 9/2/14

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

ALS Standard Operating Procedure

DOCUMENT TITLE:	AMMONIA BY COLORIMETRY
REFERENCED METHOD:	EPA 350.1
SOP ID:	GEN-350.1
REV. NUMBER:	6
EFFECTIVE DATE:	7/15/13

AMMONIA BY COLORIMETRY

EPA 350.1

SOPID: GEN-350.1 Rev. Number: 6 Effective Date: 7/15/13

Approved By: Date: 6/17/13
Department Supervisor – Christine Kutzer

Approved By: Date: 6/18/13
QA Manager – Lisa Reyes

Approved By: Date: 6/18/13
Laboratory Director – Michael Perry

Date: 5/16/19
QA Manager – Vicky Collar

Archival Date: ____________ Doc Control ID#: ____________ Editor: ____________

19-GEN-01
5/16/19

TABLE OF CONTENTS

AMMONIA BY COLORIMETRY

1) Scope and Applicability

1.1 This SOP uses EPA method 350.1 to determine the concentration of Ammonia in drinking, ground, surface and saline waters, domestic and industrial wastes, and soils. The method is based on reactions that are specific for the ammonium (NH4) ion.

1.2 The reporting limit is 0.05 mg/L for regular level water analysis and 0.01 mg/L for low level water analysis. The reporting limit is 5.00 mg/Kg for soils and non-aqueous liquids.

2) Summary of Procedure

This method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside is added to enhance sensitivity. The absorbance of the reaction product is measured at 630nm or 660nm, and is directly proportional to the original ammonia concentration in the sample. This method is performed by flow injection. The laboratory does not distill samples.

3) Definitions

3.1 Initial Calibration - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the system.

3.2 QA/QC Samples - Samples added to a sample preparation batch, or an analytical batch to provide quality assurance checks on the analysis.

3.3 Laboratory Control Sample (LCS) - An artificial sample to which known quantities of the analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.4 Matrix Spike (MS) - In the matrix spike analysis, a predetermined quantity of standard solutions of the analyte is added to a sample matrix prior to sample analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for the analyte detected.

3.5 Duplicate Sample (DUP) - A laboratory duplicate. The duplicate sample is a separate aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.6 Independent Calibration Verification (ICV) – The ICV solution is made from a stock solution which is independent from the stock used to prepare calibration standards and is used to verify the validity of the calibration.

3.7 Continuing Calibration Verification Standard (CCV) - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.8 Instrument Blank (ICB/CCB) - The instrument blank (also called initial or continuing calibration blank) is a volume of blank reagent of composition identical to the samples. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis. This blank may also be known as the Method Blank since there is no separate preparation step.

3.9 Relative Percent Difference (RPD) – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.10 Batch - Samples processed together as a unit, not to exceed 20 samples. See ADM-BATCH for further discussion.

3.11 Method Detection Limit (MDL): a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.12 Limit of Detection (LOD) - An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory – dependent. For DOD, the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

3.13 Limit of Quantitation (LOQ) / Reporting Limit/MRL - The minimum levels, concentrations, or quantities of a target that can be reported with a specified degree of confidence. For DOD, the lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard.

3.14 Method Blank (MB) – The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure. Because samples are not digested, the instrument blank may serve as the method blank.

4) Health and Safety Warnings

4.1 All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

4.2 Chemicals, reagents and standards must be handled as described in the company safety policies, approved methods and in MSDSs where available. Refer to the Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

4.3 Sodium Hydroxide (NaOH) is a strong caustic and a severe health and contact hazard. Use nitrile or latex gloves while handling pellets or preparing solutions.

4.4 Sulfuric Acid is used in this method. This acid is extremely corrosive and care must be taken while handling it. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions

4.5 Refer to the Safety Manual for further discussion of general safety procedures and information.

4.6 The following chemicals have the potential to be highly toxic or hazardous. Consult specific MSDS.

- Sodium Nitroprusside (Na2Fe(CN)5NO·2H2O)
- Phenol, Liquefied (C6H5OH)

4.7 Waste Management And Pollution Prevention

- It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when disposed of properly.
- The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the EH&S Manual. Reagents are prepared upon an as-needed basis in small quantities. Minimum sample volumes are used during analysis.
- All used reagent is collected in a container and disposed of into an inorganic waste carboy for proper disposal.
- Samples are disposed according to SMO-SPLDIS.

5) Cautions

5.1 Nearly all of the components of the Lachat can be easily cleaned or replaced. The exception is the valve. When the valve becomes clogged it is necessary to have the unit sent out to be serviced. To avoid this expense and inconvenience, be sure sample cups, and dispo cups are free of particulates by rinsing thoroughly with D.I. water and drying. Visual inspection of this equipment is also recommended before analysis. Turbid samples should also be filtered to prevent valve clogs.

5.2 Be sure to change pump tubes regularly to ensure optimal performance.

5.3 Prevent contamination - Keep the instrument and the bench area clean. Wipe down counters before and/or after use. Clean all glassware according to GEN-GC.

6) Interferences

6.1 Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA or sodium tartrate is added to the sample in-line in order to prevent this problem.

6.2 Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration through a 0.45µm filter. Sample color may be corrected for by running the samples through the manifold without color formation.

6.3 Samples with extreme pH will affect color development and result in a negative response. Samples and standards should contain approximately the same amount of chemical preservative.

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

6.5 Contamination by carryover can occur when high level samples immediately precede samples containing significantly lower levels of contamination. Pay close attention to samples which follow high level samples. Re-analyze if contamination is suspected.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

7.2 Training – see CE-QA003.

8) Sample Containers, Collection, Preservations, and Storage

8.1 Samples should be collected in purchased, certified clean plastic or glass bottles or jars. Volumes collected should be sufficient to insure a representative sample, allow for replicate analysis (if necessary), and minimize waste disposal. For bottle sets sent to the field, the lab typically uses 250 mL plastic bottles and adds H_2SO_4 preservative.

8.2 Water samples must be preserved with H_2SO_4 to a $pH < 2$. Water and soil samples are cooled to 0-6°C at the time of collection.

8.3 Samples are maintained at 0-6°C and must be analyzed within 28 days from collection.

8.4 For further sample handling, storage, and custody procedures, see SMO-GEN and SMO-ICOC.

9) Equipment and Supplies

9.1 Balance - Analytical, capable of accurately weighing to the nearest 0.0001g. Calibrated according to ADM-DALYCK.

9.2 Micropipet, adjustable 100-1000µL or fixed at desired volume. Calibrated according to ADM-PCAL.

9.3 Repipetor (20.0 mL capacity).Calibrated according to ADM-PCAL.

9.4 Disposable beakers (dispo-cups)

9.5 Instrument:

Instrument ID	Instrument Configuration	Manufacturer Part	Serial Number	Year Acquired
Lachat 8000 (R-FIA-01)	Flow Injection System	Lachat 8000		1999
	Colorimeter	Lachat	A83000-1286	
	Pump	Lachat	A82000-525	
	Autosampler	Lachat	A81010-168	
	Computer Workstation	Gateway GP6-233	9767124	
	Analytical Software	Omnion FIA v.2	-	
Lachat 8500 (R-FIA-05)	Flow Injection System	Lachat 8500		2011
	Colorimeter		110100001295	
	Cell	BASi CC-3D	11314	
	Pump	14951	0595996-2	
	Autosampler	ASX-260	021109A260	
	Computer Workstation	Dell Optiplex 780		
	Analytical Software	Omnion FIA v.3.0	-	

10) Standards and Reagents

10.1 Standards Preparation General Information and Disclaimers

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 ppm standard may be made by adding 1 mL of 200 ppm to 10 mL or may be made by adding 4 mL of 50 ppm to 10 mL. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

The initial calibration curves given are typical, but also subject to variation due to detection levels needed. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for analyte concentration.

All Standards must be traceable using the laboratory lot system (CE-GEN007).

Standards and Reagents expire per the Expiration Policy (CE-QA012) unless otherwise specified in this SOP.

10.2 Standards –

10.2.1 Ammonium Chloride (NH4Cl) – purchased commercially. Dry for two hours at 103-105°C. Cool to room temperature and store in a desiccator.

10.2.2 Nitrogen Calibration Standard Stock Solution (1000 mg N/L): In a 1-L volumetric flask, dissolve 3.819 g dried Ammonium Chloride (NH4Cl) in about 800 mL reagent grade water (DI). Dilute to volume with DI water and invert several times until thoroughly mixed. Store at 0-6°C in amber glass. **Note: This is the same standard as is prepared for total kjeldahl nitrogen analysis. The same stock may be used for both analyses.

10.2.3 Working Stock Solution (100.0 mg and 10.0 mg N/L): Dispense 9.00 mL of carrier into each of two dispo-cups using an adjustable repipettor or graduated pipet. Add 1.00 mL of 1000 mg/L standard stock solution to the first dispo-cup using an adjustable pipettor (This is 100.0 mg N/L). Mix and then transfer 1.00 mL of the 100 mg/L to the second dispo-cup and mix (This is 10.0 mg N/L). Prepare fresh each run.

10.2.4 Initial Calibration Standards: Calibration standards are prepared directly into the carrier solution and analyzed. The EPA method requires at least 3 standards covering the range and a blank for calibration. Prepare standards in disposable beakers immediately prior to the analysis as follows:

Concentration (ppm)	Carrier (mL)	Working Stock 10 ppm (mL)
2.000	8.00	2.00
1.000	9.00	1.00
0.500	9.50	0.50
0.200	9.80	0.20
0.100	9.00	1.00 mL of 1.000 Std.
0.050	9.00	1.00 mL of 0.500 Std.
0.020	9.00	1.00 mL of 0.200 Std.
0.010	9.00	1.00 mL of 0.100 Std.
0.000	10.0	0.00

10.2.5 LCS / Matrix Spike (0.500 mg N/L): To 10.0 mL of carrier or sample, add 0.050 mL of 100.0 mg N/L working stock solution. Prepare fresh each run.

10.2.6 Nitrogen Calibration Reference Stock Solution (180 mg/L): In a 1-L volumetric flask dissolve 0.687 g dried Ammonium Chloride (NH4Cl), second source, in about 800 mL reagent grade water (DI). Dilute to volume with DI water and invert several times until thoroughly mixed. Store at 0-6°C in amber glass.

10.2.7 ICV/CCV (0.90 mg/L): Dispense 9.00 mL of carrier into each of two dispo-cups using an adjustable repipettor. Add an additional 0.5 mL of carrier to the second cup. To the first cup, add 1.00 mL of reference stock solution (to make 18.0 mg N/L). Mix and then transfer 0.50 mL of the 18.0 mg/L to the second dispo-cup and mix. This is the working reference standard and will be used for the ICV and all CCVs. Prepare fresh each run.

10.3 Stock Reagents – Store at room temperature.

10.3.1 Sulfuric Acid - (H2SO4) – Instra-Analyzed.

10.3.2 Liquefied Phenol (C6H5OH) 88% - Store in a flammable cabinet. Discard sooner than expiration if color becomes darker than straw yellow.

*CAUTION: Phenol causes severe burns and is an extreme health hazard through skin absorption. Wear proper protective laboratory clothing including gloves and rinse any exposed skin IMMEDIATELY with copious amounts of water.

10.3.3 Sodium Hydroxide flakes (NaOH) – purchase with special attention to ammonia content.

10.3.4 Sodium Hypochlorite (6%)

10.3.5 Disodium EDTA (C10H14N2Na2O8·2H2O).

10.3.6 Sodium Nitroprusside ((Na2Fe(CN)5NO·2H2O) – also called nitroferricyanide

10.4 Working Reagents

10.4.1 Carrier and Diluent: In a 1-L plastic bottle add 998 g Millipore DI water and 3.68 g Instra-Analyzed Sulfuric Acid. Invert several times until thoroughly mixed. Prepare fresh for each run.

10.4.2 Sodium Phenolate: To a tared 1 Liter amber glass jar add 888 g Millipore DI, 94.2 g of 88% liquefied Phenol (C6H5OH) and 32 g of Sodium Hydroxide flakes (NaOH). Stir until dissolved. Store at 0-6°C in an amber glass jar. *PREPARE IN HOOD!!

10.4.3 Sodium Hypochlorite working solution: In a 1L amber bottle add 300 mL 6% sodium hypochlorite and 300 mLs Millipore DI. Invert to mix. Prepare fresh for each run.

10.4.4 Buffer Solution: In a 1-L glass amber jar, dissolve 50.0 g Disodium EDTA and 9.0 g NaOH in 965 g Millipore DI water. Stir until dissolved. Store in an amber glass jar at room temperature.

10.4.5 Sodium Nitroprusside Color Reagent: In a 1-L amber glass jar dissolve 3.50 g Sodium Nitroprusside in 1000 g Millipore DI water. Stir until dissolved. Store in an amber glass jar at room temperature.

10.5 MB – Analyze carrier/diluent as a sample.

11) Method Calibration

11.1 Initial Calibration – follow policies in ADM-ICAL.

11.1.1 The data system will prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation (see below). The ICAL must have at least 3 points which cover the calibration range and a blank. The lab routinely uses all of the points described in the Standards and Reagents Section. The correlation coefficient must be ≥0.997. If it is not, correct the problem and recalibrate.

11.1.2 Calibration Curve, Linear Regression: This method of quantitation uses the equation of a line ($y=mx+b$). The curve must not be forced through zero. Upon recommendation of the instrument manufacturer, a 1/x weighting method is used for this calibration.

11.1.3 Due to the limitations of the Lachat software, once a calibration is complete, standards may not be reanalyzed. Since there are legitimate times (bad integration, air spike in peak) when it is necessary to repeat a standard, include two injections of the zero and the two lowest standards. The software is selected to average the replicates of the injections. Points may only be dropped due to assignable cause as detailed in ADM-ICAL and the cause must be documented in the raw data.

12) Sample Preparation and Analysis

12.1 Print a Responsibility Report from LIMS to determine what samples are available to be run. Prioritize samples to be run based on holding time and due dates. Scan the samples to be analyzed out of storage according to SMO-ICOC.

12.2 Sample Preparation

12.2.1 If a sample is not preserved, it must be acidified to pH<2 with sulfuric acid prior to analysis.

12.2.2 Soil samples – Weigh 1.0 g of sample into a clean, tared B-cup. Bring the total weight to 100 g with carrier/diluent. Cap the cup and shake vigorously for 5 minutes.

12.2.3 Turbid water samples and prepared soil samples - filter through a syringe filter directly into the autosampler cup or into a dispo cup if dilutions are to be performed. If any samples are filtered, also filter a blank and an LCS. See GEN-FILTER for more information.

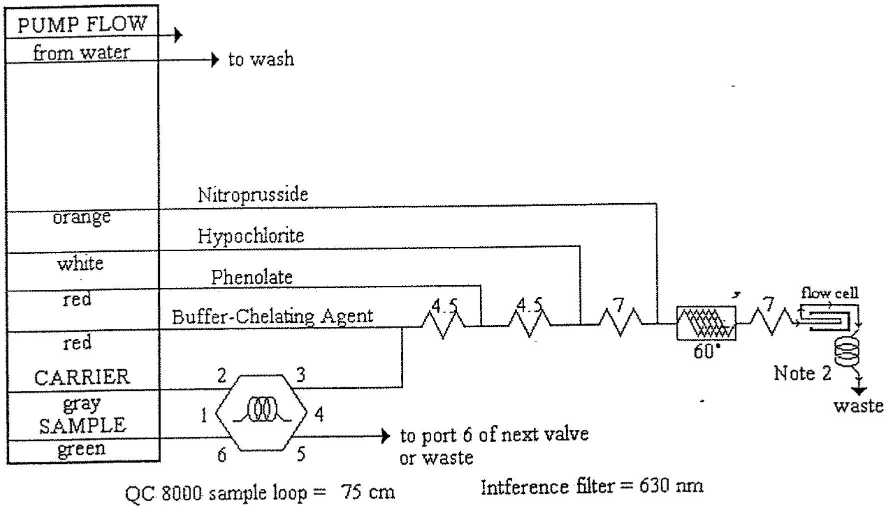
12.3 Determine which instrument is to be used (based on workload). The ID of the instrument must be documented on the raw data – this is typically printed in the header of the instrument's sample results report.

12.4 Prepare standards and reagents - as described in the Standards and Reagents Section for the appropriate instrument. Prepare the entire set of initial calibration standards even when low level analysis is not required.

12.5 Instrument Setup

12.5.1 Adjust heating coil setting to 60°C. Turn on and allow to preheat.

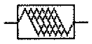
12.5.2 Connect the manifold to the instrument - heating coil length (650 cm). Place pump tubes loosely in their holders.



CARRIER is Reagent 5.

Manifold tubing is **0.8 mm (0.032 in) i.d.** This is **5.2 uL/cm.**

4.5	is	**70**	cm of tubing on a 4.5 cm coil support
7	is	**135**	cm of tubing on a 7 cm coil support

APPARATUS: The  indicates 650 cm of tubing wrapped around the heater block at the specified temperature.

Note 1: TYGON PUMP TUBES MUST BE USED FOR THIS METHOD

Note 2: 200 cm x 0.022" i.d. backpressure loop.

12.5.3 Once the heating coil has reached 60°C, place reagent feed lines in DI. Turn the pump on, adjust the tension of the pump tubes and inspect the system for leaks.

12.5.4 Load the method into the software and enter sample labels for the first tray. Set up the batch in accordance with ADM-BATCH and the frequency requirements of this SOP. Analyze CRDLs to see the response at the low end of the curve since the software does not convert the raw area counts of the standards into concentration in the calibration. The method should be optimized from the suggested operating. The Lachat software will calculate all dilutions provided the dilution information is entered.

The Lachat Suggested Operating Parameters for the QuikChem 8000

Pump Speed:	35
Cycle Period:	60

Analyte Data:

Concentration Units:	mg N/L
Peak Base Width:	27.0 s
% Width Tolerance:	100
Threshold:	10000
Inject to Peak Start:	41.8s
Chemistry:	direct

Calibration Data:

Calibration Fit Type:	1st Order Polynomial
Calibration Rep. Handling:	Average
Weighting Method:	None
Concentration Scaling:	None
Force through Zero:	No

Sampler Timing:

Min. Probe in Wash Period:	5.0s
Probe in Sample Period:	24s

Valve Timing:

Load Time:	0.0s
Load Period:	15s
Inject Period:	45s

12.6 Instrument Analysis

12.6.1 Load the calibration standards and the samples into the autosampler as entered into the software with standards in order of decreasing concentration. Make sample dilutions with the diluent/carrier in compliance with ADM-DIL.

12.6.2 Place the feed lines in the appropriate reagents and allow to pump for about 5 minutes.

12.6.3 Run the standards, references, and samples. Be sure all ICAL and QC criteria is met and samples are within linear range. Repeat any samples at a dilution which are over the linear range (see ADM-DIL).

12.6.4 Repeat any samples which may be influenced by carryover from the previous sample (response has not returned to baseline). Repeat any samples which have airpeaks which may influence the results. Repeat any samples which were incorrectly integrated by the software.

12.6.5 Close with an acceptable CCV/CCB set.

12.6.6 Print the results and the calibration information immediately after the run.

12.6.6.1 If any other runs are done before this information is printed, special care must be taken to ensure the correct calibration is applied to the results.

12.6.7 Shut Down

When finished, place all lines in DI and rinse for about 5 minutes. Remove lines from DI and pump to dry for about 5 minutes. Turn off the pump and heating coil, and release tension on pump tubes.

12.6.8 Scan and return the samples to storage.

12.6.9 Create the run in LIMS and upload the data to LIMS – see ADM-DREV for details.

13) Troubleshooting

Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by laboratory staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

14) Data Acquisition

Data is uploaded electronically from the instrument to LIMS.

15) Calculations and Data Reduction Requirements

15.1 Calculations: Instrument software will calculate all results from the regression equation, including those with dilutions, provided that the correct dilution information was entered. Soil samples are entered with a 100 fold dilution in the software to account for the 1g to 100 mL extraction, but the dilution is entered as straight in the LIMS since this is the lowest possible dilution. The MRL in LIMs is based on this assumption.

15.2 Review the data to the required reporting limit. Both low level and regular level samples are analyzed on the same initial calibration curve, but the regular level samples do not need to meet the low level criteria.

15.3 Water samples are reported in mg N/L. Soil samples are reported in mg N/Kg.

15.4 Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV.

15.5 If requested, the LIMS calculates a Total Organic Nitrogen (TON) result by subtracting the ammonia result from the TKN result (GEN-351.2).

15.6 Report instrument blanks associated with the samples as the Method Blank in the final report.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 Initial calibration requirements are given in the Method Calibration Section.

16.2 ICV/CCV –

- Frequency - Analyze an ICV immediately after the initial calibration. Analyze a CCV after every 10 or fewer samples.
- Acceptance - Recovery must be 90-110% of the true value
- Corrective Action - Recalibrate if ICV fails. If a CCV fails, and acceptable CCV must be achieved before continuing. Samples bound by the unacceptable CCV must be reanalyzed. Samples with results less than the reporting limit may be reported with a CCV with high recovery.

16.3 ICB/CCB/Method Blank –

- Frequency -Analyze immediately after each ICV and CCV – every 10 samples.
- Acceptance - The result must be less than the reporting limit.
- Corrective Action - If the result is above the reporting limit, correct the problem and re-run the samples bound by the unacceptable CCB, or raise the reporting limit. If samples are greater than 10 times the contamination in the blank, the samples may be reported.

16.4 LCS –

- Frequency - Prepare one for every 20 samples.
- Acceptance - LCS recovery must be 90-110% of the true value.
- Corrective Action - If an LCS is out of control, correct the problem and reanalyze the affected samples or flag the data. Samples with results less than the reporting limit may be reported with an LCS with high recovery

16.5 Duplicates –

- Frequency - Analyze one duplicate for every 10 or fewer samples. Do not choose field blanks as samples for the analysis of duplicates.
- Acceptance - The relative percent difference (RPD) between matrix duplicates should be 20 or less. If one or both of sample or duplicate is < 5X RL, the control limit is ± RL. (The RL is the client reporting limit-typically the LOQ or MRL, but may be lower)
- Corrective Action - If they are not, repeat the original and the duplicate or flag the affected data.

16.6 Matrix Spike –

- Frequency - Analyze one matrix spike for every 10 or fewer samples. Do not choose field blanks as samples for the analysis of the MS.
- Acceptance - Matrix spike recovery must be within the limits in the Data Quality Objectives Table.
- Corrective action - if recovery of the MS fails on a sample, the batch is considered acceptable as long as the LCS meets acceptance criteria. It is recommended that the MS/MSD be reprepared and reanalyzed to confirm the outliers, however it is not required due to probable matrix interferences. Flag the outlier.

17) Data Records Management

See CE-GEN003 and ADM-ARCH

18) Contingencies for Handling Out Of Control Data

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, flag and narrate appropriately.

19) Method Performance

Detection and Quantitation limits are determined according to the requirements in CE-QA011.

Demonstration of Capability is performed according to CE-QA003.

"In a single laboratory (EMSL-Cincinnati), using surface water samples at concentrations of 1.41, 0.77, 0.59, and 0.43 mg NH3-N/L, the standard deviation was ±0.005.

In a single laboratory (EMSL-Cincinnati), using surface water samples at concentrations of 0.16 and 1.44 mg NH3-N/L, recoveries were 107% and 99%, respectively.

Interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in mg NH3-N/L."

20) Summary of Changes

- Incorporated Change Forms for diluting soils with carrier/diluent instead of DI, and for averaging replicates of low standards.
- Added that unpreserved samples must be preserved prior to analysis
- Updated to ALS format – removed CAS throughout.
- Removed Konelab throughout – no longer in use

21) References and Related Documents

- Methods for Chemical Analyses of Water and Wastewater, USEPA, EPA-600/4-79-020, March 1983. Method 350.1
- Methods for the Determination of Inorganic Substances in Environmental Samples, USEPA, EPA/600/R-93/100 August 1993. Method 350.1
- Lachat QuikChem Method [10-107-06-1-B].

22) Appendix

- Calibration Standard Preparation - example only – controlled separately on the Controlled Forms section of the Rochester Intranet.
- Table 1 – Interlaboratory precision and Accuracy Data.

AMMONIA (2.000 - 0.000 (pql=0.010mg/L))

A.) 10.0 ppm WORKING STOCK:
do two (2) 1/10 serial dilutions of the 1000 ppm NH_3 / TKN Standard Stock. Prepare these dilutions with Carrier-Diluent, as well as all standards, references, and sample dilutions.

B.) STANDARDS:

conc. (mg/l)	mls 10.0 ppm	mls Carrier-Diluent
a.) 2.000	2.00	8.00
b.) 1.000	1.00	9.00
c.) 0.500	0.50	9.50
d.) 0.200	0.20	9.80
e.) 0.100	1/10 dil'n of b.) 1.000	
f.) 0.050	1/10 dil'n of c.) 0.500	
g.) 0.020	1/10 dil'n of d.) 0.200	
h.) 0.010	1/10 dil'n of e.) 0.100	
i.) 0.000	10 mLs of Carrier-Diluent	

C.) ICV / CCV: (True Value = 0.90 mg/l)
do one (1) 1/10 serial dilutions of 180 ppm NH_3 Reference Stock Solution. Add 0.50 mL of 18 ppm Ref stock to 9.5 mL DI.

D.) LCS / Matrix Spike: (True Value = 0.500 mg/l)
10.0 mls Carrier-Diluent / sample + 0.050mls 100 ppm Working Stock.

P:\INTRANET\QAQC\Forms Controlled\WC Stds Ammonia r0.doc 4/12/13

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE 1. INTERLABORATORY PRECISION AND ACCURACY DATA

NUMBER OF VALUES REPORTED	TRUE VALUE (T)	MEAN (X)	RESIDUAL FOR X	STANDARD DEVIATION (S)	RESIDUAL FOR S
134	0.270	0.2670	-0.0011	0.0342	0.0015
157	0.692	0.6972	0.0059	0.0476	-0.0070
136	1.20	1.2008	0.0001	0.0698	-0.0112
195	1.60	1.6095	0.0076	0.1023	0.0006
142	3.00	3.0128	0.0069	0.1677	-0.0067
159	3.50	3.4991	-0.0083	0.2168	0.0165
156	3.60	3.5955	-0.0122	0.1821	-0.0234
200	4.20	4.2271	0.0177	0.2855	0.0488
196	8.76	8.7257	-0.0568	0.4606	-0.0127
156	11.0	11.0747	0.0457	0.5401	-0.0495
142	13.0	12.9883	-0.0465	0.6961	0.0027
199	18.0	17.9727	-0.0765	1.1635	0.2106

REGRESSIONS: $X = 1.003T - 0.003$, $S = 0.052T + 0.019$

SOP CHANGE FORM

SOP Title: Nitrate / Nitrite by Colorimetry
SOP Code: GEN-353.2
SOP Revision No.: 5
SOP Date: 11/6/17
SOP Section(s) Affected by Change: 10.6.2, 10.6.3, 10.6.4

Description of Change: The LCS and samples are now being spiked at 0.50 mg/L. The ICV/CCV was raised to 1.0 mg/L for consistency. The cadmium column check (for both NO2 and NO3) was also raised to 1.0 mg/L to be consistent with ICV/CCV concentrations.

Change(s) Submitted by: Michael Rogerson	Date: 10/25/18

Approvals:

Technical Reviewer Signature:	Date: 10/25/18
QA Signature:	Date: 10/26/18
Laboratory Director Signature:	Date: 10/26/1

Change(s) Effective Date: Current practice

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

NITRATE/NITRITE BY COLORIMETRY

DOCUMENT I.D. GEN-353.2

Approved By: ______________________ Date: 10/31/17
WetChemistry Supervisor, Chris Woods

Approved By: ______________________ Date: 10/31/17
Inorganics Manager, Christine Kutzer

Approved By: ______________________ Date: 10/31/17
Operations Manager, Michael Cymbal

Approved By: ______________________ Date: 10/31/17
Quality Assurance Manager, Vicky Collom

Approved By: ______________________ Date: 10/31/17
Laboratory Director, Carlton Beechler

Doc Control ID:	17-GEN-01	Archived Date:	________________

cm 11/1/17

TABLE OF CONTENTS

1) Scope and Applicability

1.1 This SOP uses EPA 353.2 for the determination of nitrite, nitrate+nitrite, and nitrate (by calculation) in drinking, ground and surface waters, domestic and industrial wastes, and soils.

1.2 Linear Range and PQL

1.2.1 Combined NO3+NO2 - The applicable linear range for NO3+NO2 using a 150 cm sample loop is 0 to 2.0 mg/L. Dilutions may be made as necessary. The PQL is 0.002 mg/L for low level and 0.05 mg/L for regular level.

1.2.2 The applicable linear range for NO2 is 0 to 1.0 mg /L. The PQL is 0.01 mg/L.

2) Summary of Procedure

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N (1-naphthyl) ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite alone is determined by removing the cadmium column. The procedure is automated and analyzed by flow injection (Lachat Analyzer) or discrete analyzer (Thermo Gallery). The Thermo Gallery Discrete Analyzer is only used for nitrite since it does not have a cadmium column. To calculate nitrate alone, analyze separately for nitrite and for (nitrite + nitrate) and subtract the nitrite.

3) Definitions

3.1 Nitrate+Nitrite (NO3+NO2) – the sum of the indigenous nitrite plus the nitrate which is converted to nitrite in the cadmium column.

3.2 Independent Calibration Verification (ICV) - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.3 Laboratory Control Sample (LCS) - An aliquot of reagent water to which a known quantity of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.4 Matrix Spike (MS) - In the matrix spike analysis, a predetermined quantity of standard solution of the analyte is added to a sample matrix prior to sample analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recovery is calculated for the analytes detected.

3.5 Matrix Spike Duplicate (MSD or DMS) – a duplicate of the Matrix Spike. The RPD between the MS and the MSD are evaluated for precision.

3.6 Duplicate Sample (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.7 Continuing Calibration Verification Standard (CCV) - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.8 Instrument Blank (ICB/CCB/MB) - The blank (also called initial or continuing calibration blank) is a volume of DI water which is analyzed exactly like the sample. Since there is no preparation required, this blank also serves as a method blank. The purpose of the blank is to determine the levels of contamination associated with the analysis.

3.9 Relative Percent Difference (RPD) – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.10 Batch - A group of no more than 20 samples that are analyzed together on the same day. For more information see ADM-BATCH.

3.11 Method Detection Limit (MDL): a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.12 Limit of Detection (LOD) - An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory – dependent. For DOD, the smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

3.13 Limit of Quantitation (LOQ) / Reporting Limit/MRL - The minimum levels, concentrations, or quantities of a target that can be reported with a specified degree of confidence. For DOD, the lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard.

4) Health and Safety Warnings

4.1 All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as safety glasses, lab coat and the correct gloves.

4.2 Chemicals, reagents and standards must be handled as described in the company safety policies, approved methods and in MSDSs where available. Refer to the Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

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

- Cadmium
- Ammonium hydroxide
- Sodium hydroxide
- Phosphoric acid

- Sulfanilamide

4.4 Refer to the Safety Manual for further discussion of general safety procedures and information.

4.5 Waste Management and Pollution Prevention

- It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when disposed of properly.
- The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the EH&S Manual. Reagents are prepared upon an as-needed basis in small quantities. Minimum sample volumes are used during analysis.
- Standards and reagents should be purchased or prepared on an as-needed basis. Waste lines for these analyses should be run into the sink. Samples are disposed according to SMO-SPLDIS.

5) Cautions

5.1 Nearly all of the components of the Lachat can be easily cleaned or replaced. The exception is the valve. When the valve becomes clogged it is necessary to have the unit sent out to be serviced. To avoid this expense and inconvenience, be sure sample cups, and dispo cups are free of particulates by rinsing thoroughly with D.I. water and drying. Visual inspection of this equipment is also recommended before analysis. Turbid samples should also be filtered to prevent valve clogs.

5.2 Be sure to change pump tubes regularly to ensure optimal performance.

5.3 Daily, weekly, and monthly maintenance is detailed in the help/maintenance section on the Discrete.

5.4 Prevent contamination - Keep the instrument and the bench area clean. Wipe down counters before and/or after use. Clean all glassware according to GEN-GC.

6) Interferences

6.1 EDTA is added to the buffer to reduce interference caused by high concentrations of iron, copper, or other metals in the sample

6.2 Grease and oils will foul the cadmium column; these may be pre-extracted with organic solvents.

6.3 Residual chlorine can interfere by oxidizing the column.

6.4 A color blank should be analyzed for highly colored samples, or samples which are still turbid after filtering. The color blank is subtracted from the sample result.

6.5 Contamination by carryover can occur when high level samples immediately precede samples containing significantly lower levels of contamination. Pay close attention to samples which follow high level samples. Re-analyze if contamination is suspected

7) Personnel Qualifications and Responsibilities

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

8) Sample Collection, Containers, Preservation, and Storage

8.1 Samples should be collected in certified clean plastic or glass bottles. The lab typically provides certified clean 250 mL HDPE bottles.

8.2 NO3+NO2 analyses require preservation of the sample with H2SO4 to pH < 2. Samples to be analyzed for NO2 alone should not have a chemical preservative added. Separate bottles are required for analysis for calculation of NO3.

8.3 Samples are stored at 0-6 °C until analysis.

8.4 Holding time for preserved NO3+NO2 = 28 days. For NO2, holding time is 48 hours.

8.5 For further sample handling, storage, and custody procedures, see SMO-GEN and SMO-ICOC.

9) Equipment and Supplies

9.1 Instrument

Instrument ID	Instrument Configuration	Manufacturer Part	Serial Number	Year Acquired
Lachat 8000 (R-FIA-01)	Flow Injection System	Lachat 8000		1999
	Colorimeter	Lachat	A83000-1286	
	Pump	Lachat	A82000-525	
	Autosampler	Lachat	A81010-168	
	Computer Workstation	Gateway GP6-233	9767124	
	Analytical Software	Omnion FIA v.2	-	
Lachat 8500 (R-FIA-05)	Flow Injection System	Lachat 8500		2011
	Colorimeter		110100001295	
	Cell	BASi CC-3D	11314	
	Pump	14951	0595996-2	
	Autosampler	ASX-260	021109A260	
	Computer Workstation	Dell Optiplex 780		
	Analytical Software	Omnion FIA v.3.0	-	

Gallery (R-Discrete-02)	Instrument	Thermo Fisher Gallery	**861000110585**	2017
	Computer Workstation	**Dell Optiplex 7040**		

9.2 Cadmium columns - purchased commercially from Lachat Instruments. Spent columns are discarded. Columns may be prepared in-house if necessary. Consult the Lachat method for details.

9.3 Lachat Suggested Operating Parameters:

Pump speed: 35
Cycle period: 65 sec.

Analyte Data:
Peak Base Width: 25 sec.
% Width Tolerance: 100
Threshold: 5000
Inject to Peak Start: 22 sec.
Chemistry: Direct

Calibration Data:
Calibration Fit Type: 1st Order Polynomial
Calibration Rep. Handling: Average
Weighting Method: None
Concentration Scaling: None
Force Through Zero: No

Sampler Timing:
Min. probe in wash period: 12s
Probe in sample period: 32s

Valve Timing:
Load Time: 0.0 sec.
Load Period: 28 sec.

Inject Period: 37 sec

9.4 1000 mL volumetric flasks

9.5 1000 uL adjustable pipette and tips- calibrated according to ADM-PCAL

9.6 Disposable plastic 10 mL pipettes

9.7 10 mL disposable plastic beakers (dispo cup).

9.8 2 mL sample cups

9.9 Top loading balance - calibrated according to ADM-DALYCK.

9.10 Culture Tubes – 13x100mm

10) Standards and Reagents

10.1 Standards Preparation General Information and Disclaimers

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 mg/L standard may be made by adding 1 mL of 200 mg/L to 10 mL or may be made by adding 4 mL of 50 mg/L to 10 mL. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

The initial calibration curves given are typical, but also subject to variation due to detection levels needed. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for analyte concentration.

All Standards must be traceable using the laboratory lot system (CE-QA007).

All standards and reagents expire per the Expiration Policy unless otherwise indicated in this SOP

10.2 Purchased reagents and standards: Store at room temperature.

- Hydrochloric Acid (HCl) – concentrated
- Ammonium Hydroxide (NH4OH) – concentrated
- Disodium ethylenediamine tetracetic acid dihydrate (Na2EDTA 2H20)
- Sodium Hydroxide (NaOH) – 15M
- Phosphoric Acid (H3PO4) 85%
- Sulfanilamide
- N-1-napthylethylenediamine dihydrochloride (NED)
- 1000 mg/L Nitrite Standard Stock – Environmental Express (P/N IC-NO2-M), or equivalent.
- 1000 mg/L Nitrite Reference stock – Ultra Scientific (P/N ICC-007A), or equivalent.

10.3 Prepared Reagents -

10.3.1 Ammonium Chloride Buffer, pH = 8.5

Under a hood: To a 1 Liter amber glass jar, add 778.5 g of D.I. water, 113.4 g of concentrated HCL, 76.5 g of ammonium hydroxide and 0.90 g Na2EDTA. Stir until dissolved. Adjust the pH up to 8.5 with concentrated NaOH or HCl. Store in an amber glass container at room temperature.

10.3.2 Sulfanilamide Color Reagent:

To a tared 1 Liter amber glass jar, add 788 g of water, 153 g 85% phosphoric acid, 36.0 g sulfanilamide and 0.90 g NED. Stir until dissolved. Store in an amber glass bottle at room temperature. Expires in one month or when discolored, whichever is sooner.

10.4 Nitrite Standards

10.4.1 Nitrite intermediate standard (10 mg N/L): Perform two 1/10 serial dilutions of the 1000 mg/L stock. Prepare fresh daily.

10.4.2 Nitrite intermediate reference (10mg N/L if using a 1000mg/L Standard: Perform two 1/10 serial dilutions of the secondary 1000mg/L stock. Prepare fresh daily.

10.4.3 Lachat Nitrite calibration standards –
Prepare fresh daily in dispo cups as follows:

Conc. (mg/L)	mL of 10 mg/L NO2 standard	mL DI water
1.00	1.00	9.0
0.50	0.50	9.5
0.20	0.20	9.8
0.10	1.0 mL of 1.00 mg/L standard	9.0
0.05	1.0 mL of 0.50 mg/L standard	9.0
0.02	1.0 mL of 0.20 mg/L standard	9.0
0.01	1.0 mL of 0.10 mg/L standard	9.0
0.00	0.0	10.0

Pour back and forth between two dispo cups to mix.

10.4.4 Discrete - A manual calibration can be performed by the analyst by preparing the standards as listed above for the Lachat or the analyst can use the Thermo Gallery's automated calibration dilution feature. This is a feature that is programed into the method and uses a 1mg/L Standard Solution prepared from the 1000mg/L Standard Stock. The instrument is programmed to dilute the 1mg/L Standard into DI Water to prepare the following calibration levels.

Conc. (mg/L)	Dilution Performed by Instrument
0.00	0
0.01	n+99
0.02	n+49
0.05	n+19
0.10	n+9

0.20	n+4
0.50	n+1
1.00	n+0

Instrument uses the dilution convention where all dilutions are assumed to have 1 part calibration standard used plus “n” parts of DI Water. For example, in the above table, a dilution of n+99 would represent 1 part of standard plus 99 parts DI Water (a 1/100 dilution). Using a calibrant of 1mg/L, this would result in a standard of 0.010mg/L.

10.4.5 Nitrite LCS, Matrix Spikes/MSD (0.25 mg/L): Into a dispo cup, measure 10 mL DI (LCS) or sample (MS and MSD). Add 0.25 mL of 10 mg/L working stock. Pour back and forth between two dispo cups to mix. Prepare fresh per run.

10.4.6 Nitrite Reference ICV/ CCV (0.50 mg/L): Using a purchased 1000 mg/L stock, make (2) 1/10 dilutions for a 10 mg/L reference stock. Add 0.5 mL of 10 mg/L reference stock to 9.5 mL of UPDI water. Mix by pouring the solution back and forth between two dispo cups. Prepare fresh per run.

10.5 Nitrate Stock and Intermediate Standards

10.5.1 Nitrate Intermediate standards (100 mg/L and 10 mg/L) – Prepare two 10X serial dilutions of the 1000 mg/L Nitrate. Prepare fresh daily.

10.6 Nitrate Working Standards – Prepare fresh daily

10.6.1 Nitrate ICAL standards - Prepare in dispo cups as follows:

Conc. (mg/L)	mL of 10 mg/L standard	mL DI water
2.00	2.00	8.0
1.00	1.00	9.0
0.50	0.50	9.5
0.20	0.20	9.8
0.10	1.0 mL of 1.00 mg/L standard	9.0
0.05	1.0 mL of 0.50 mg/L standard	9.0
0.01	1.0 mL of 0.10 mg/L standard	9.0
0.005	1.0 mL of 0.05 mg/L standard	9.0
0.002	(2) 1/10 serial dilutions of the 0.20 mg/L standard	
0.00	0.0	10.0

Pour back and forth between two dispo cups to mix.

10.6.2 N03+NO2 LCS, Matrix Spikes/MSD (0.20 mg/L): Into a dispo cup, measure 10 mL DI (LCS)or sample (MS and MSD) using a 10 mL disposable pipette. Add 0.020 mL of 100 mg/L nitrate working stock. Pour back and forth between two dispo cups to mix.

10.6.3 NO3+NO2 Reference ICV/ CCV (0.50 mg/L): Using 1000 mg/L Reference stock, make (2) 1/10 dilutions to create a 10 mg/L reference working stock. Add 0.5mL of 10 mg/L reference working stock to 9.5 mL of UPDI. Mix by pouring the solution back and forth between two dispo cups.

10.6.4 Cadmium column efficiency check solutions: Prepare fresh daily

10.6.4.1 0.5 mg/L NO2 - Perform two 1/10 serial dilutions of the 1000 mg/L NO2 stock. Add 0.50 mL of the resulting 10 mg/L working stock to 9.5 mL DI (or, use the 0.50 mg/L std prepared for nitrite analysis).

10.6.4.2 0.5 mg/L NO3 - Use the 0.50 mg/L standard prepared for nitrate analysis.

10.7 Method Blank / ICB/CCB – DI water

11) Method Calibration

11.1 Initial Calibration – follow policies in ADM-ICAL unless otherwise detailed in this SOP.

11.1.1 Frequency - daily

11.1.2 The data system will prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation (see below). The ICAL must have at least 3 points which cover the calibration range and a blank. The lab routinely uses all of the points described in the Standards Section. The correlation coefficient must be 0.997 or higher. If the correlation coefficient limit is not achieved, stop the analysis, correct the problem, and recalibrate.

11.1.3 Calibration Curve, Linear Regression : This method of quantitation uses the equation of a line (y=mx+b).

11.1.4 Due to the limitations of the Lachat software, once a calibration is complete, standards may not be reanalyzed. Since there are legitimate times (bad integration, air spike in peak) when it is necessary to repeat a standard, include two injections of the zero and the two lowest standards. The software is selected to average the replicates of the injections. Points may only be dropped due to assignable cause as detailed in ADM-ICAL and the cause must be documented in the raw data.

11.1.5 Verify the linear calibration range by refitting the high curve point to the curve. If the point is outside of 90-110% of its true value, linearity must be reestablished. For the 8000, rerun the high standard for a LRC (Linear Range Check).

11.1.6 To calibrate for Nitrite on the Thermo Gallery, first make sure that all start up procedures have been followed (See section12.3). Once start up is performed, call up the nitrite method. The software will prompt the analyst to pour up a fresh 1mg/L standard, zero standard (DI), ICV and ICB and add this rack to the instrument. Once the start button is pressed, the automatic dilution system will perform the calibration based on the standards listed in section 10.

12) Sample Preparation and Analysis

12.1 Sample Preparation

12.1.1 Soil samples – Homogenize and subsample according to ADM-SPLPREP. Weigh 1.0 g of sample into a clean, tared B-cup. Document the ID of the balance used to weigh the sample. Bring the total weight to 100 g with DI. Cap the cup and shake vigorously for 5 minutes.

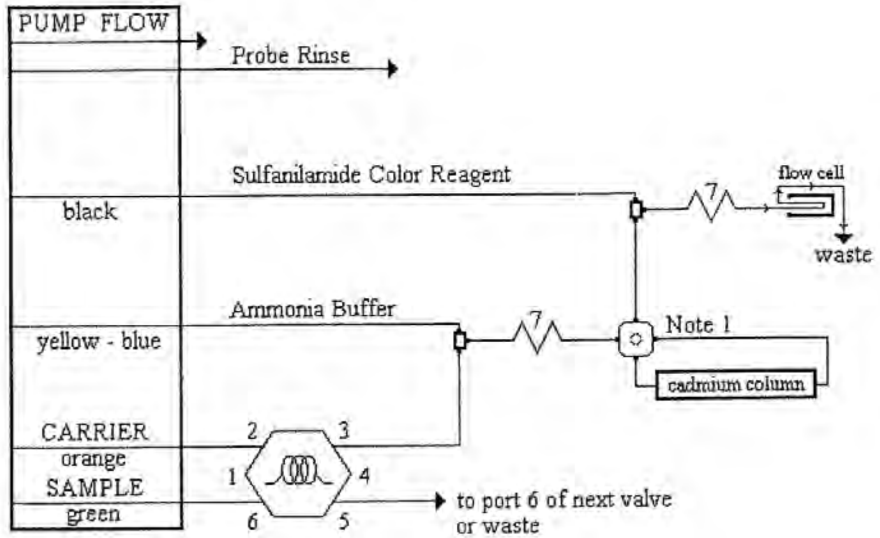
12.1.2 Turbid water samples and prepared soil samples - filter through a syringe filter directly into the autosampler cup or into a dispo cup if dilutions are to be performed. If any samples are filtered, also filter a blank and an LCS. See GEN-FILTER for more information.

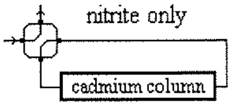
12.2 Instrument Set-up - Lachat

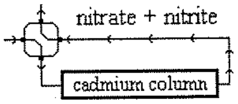
12.2.1 Turn on the computer and Lachat components.

12.2.2 Connect the manifold to the instrument. Inspect the manifold for proper connections and appropriate sample loop and wavelength filter. Place pump tubes loosely in their holders.

12.2.3 Load the appropriate method into the software and enter sample labels for the first tray. Set up the batch in accordance with ADM-BATCH and the frequency requirements in this SOP. Analyze CRDLs on the Lachat 8000 to see the response at the low end of the curve since the software does not convert the raw area counts of the standards into concentration in the calibration. The Lachat software will calculate all dilutions provided the dilution information is entered.







Sample loop = 150 cm x 0.8mm

Interference Filter = 520 nm

Carrier is DI Water

Manifold tubing is 0.8 mm (0.032 in) i.d. This is 5.2 uL/cm

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

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

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

12.3 Instrument Set-up for Thermo Gallery

12.3.1 The test parameters are attached in the appendix.

12.3.2 Switch on the Thermo Gallery if not already on

12.3.3 Switch on the Workstation

12.3.4 Enter the software by clicking on the Thermo Gallery Icon

12.3.5 Log into the software

12.3.6 Wait for the main screen

12.3.7 Run three extra rinses

12.3.8 Run water blank

12.3.9 Check water blank

12.3.10 Check and update reagent rack according to the procedure

12.3.11 Add new samples and select method in menu beneath sample ID. Set up the batch in accordance with ADM-BATCH and Section 16.

12.3.12 Fill cuvette loader when prompted.

12.4 Instrumental Analysis -

12.4.1 Load the column check standards, calibration standards, and the samples into the autosampler as entered into the software with standards in order of decreasing concentration if manually preparing calibration standards. If you are utilizing the automatic calibration dilution feature, prepare a 1mg/L standard. Prepare any sample dilutions in compliance with ADM-DIL.

12.4.2 Lachat only: Place all reagent lines in DI to rinse. Be sure all waste lines go to sink. Attach lines to pump collars. Turn on pump. Lock in collars. Rinse for a couple of minutes. Inspect for leaks. Place the color reagent, buffer, carrier (DI) and rinse feed lines in their appropriate reagent bottles. For NO3+NO2, when all bubbles are out of the lines, put column in-line. Pump until a stable baseline is established.

12.4.3 For NO3+NO2 analyses, calculate the cadmium column efficiency prior to analysis of samples. Calculate the column efficiency as follows:

Reduction efficiency = ([NO3 std] / [NO2 std]) x 100%

Record the efficiency in the Cd column Efficiency Log. If the column efficiency is <90%, stop the analysis, insert a new column, restart the analytical sequence including a new column proficiency check, and recalibrate. Discard the expired column.

12.4.4 For best results, a new column should be “shocked” prior to use. To “shock” the column, run four injections of 10 mg/L Nitrate followed by four injections of DI.

12.4.5 Run the standards, references, and samples. Be sure all ICAL and QC criteria is met and samples are within linear range. Repeat any samples at a dilution which are over the linear range (see ADM-DIL).

12.4.6 Repeat any samples which may be influenced by carryover from the previous sample (response has not returned to baseline). Repeat any samples which have airpeaks which may influence the results. Repeat any samples which were incorrectly integrated by the software.

12.4.7 Close with an acceptable CCV/CCB set.

12.4.8 Run color blanks for highly colored or turbid samples. Remove the sulfanilamide line from the reagent and place it in a phosphoric acid solution (170 g of 85% H_3PO_4/L). Analyze the desired samples. Subtract the color blank value from the sample result.

12.4.9 Print the results and the calibration information immediately after the run.

Lachat: If any other runs are done before this information is printed, special care must be taken to ensure the correct calibration is applied to the results.

Thermo Gallery: Print results and curve. Transfer file must be created at the end of the day. All of the files/test data will be included in one file. The discrete transfer template will organize by test. Daily files are cleared from the instrument at the end of the day. The data on the instrument can be accessed through the archive after this point.

12.5 Shut Down:

Lachat: When all samples are analyzed, take the cadmium column off-line, if appropriate, before moving lines to rinse. The column is stored infused with reagents. To keep air from entering the column, place the reagent lines in DI to rinse for 5 minutes. Remove all lines from DI and pump dry. Turn off pump and release pump collars.

Thermo Gallery: Perform the daily shut down maintenance (see below) and shut the instrument down.

Load wash solution. Push Standby. This will run the standby procedure. Empty and rinse waste container and cuvette container. Refill DI container.

13) Troubleshooting

13.1 See instrument manual or maintenance log for help in solving specific analytical or instrument problems.

13.2 Daily, weekly, and monthly maintenance is detailed in the help/maintenance section on the Discrete.

13.3 Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by laboratory staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

14) Data Acquisition

Data is uploaded from the instrument to LIMS.

15) Calculation and Data Reduction Requirements

15.1 Calculations: Instrument software will calculate all results from the regression equation, including those with dilutions, provided that the correct dilution information was entered. Soil samples are entered with a 100 fold dilution in the software to account for the 1g to 100 mL extraction, but the dilution is entered as straight in the LIMS since this is the lowest possible dilution. The MRL in LIMs is based on this assumption.

15.2 Report results in mg N/L or mg N/kg.

15.3 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard must be diluted and reanalyzed. See ADM-DIL.

15.4 Soils: mg/kg N = Conc. measured x mL DI used for the extraction / g sample used.

15.5 Colored samples: Conc. Reported = Conc. measured - Color Blank

15.6 Nitrate calculation: mg/L or mg/kg as NO3 as N = [NO3+NO2] - [NO2]. This calculation is done in the LIMS system.

15.7 Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV.

15.8 The results are based on the weight of nitrogen or “as N” or “–N.” The concentration of the individual standards is based on the weight of the nitrogen in the sodium or potassium nitrite or potassium nitrate – not the weight of the NO2 nor NO3. Therefore the sample concentration is the concentration of the Nitrogen from the desired form – not the weight per volume concentration of the NO2 nor NO3. This is the industry convention for this analysis.

16) Quality Control, Acceptance Criteria and Corrective Action

16.1 ICV/CCV –

- Frequency - Analyze at the beginning of the run, every 10 samples or fewer, and at the end of the run.
- Limits - Recovery must be 90-110 % of the true value. Samples less than the reporting limit that are associated with a CCV that is above limits may be reported without qualification.
- Corrective Action - Recalibrate if the ICV fails. If a CCV fails, an acceptable CCV must be achieved before continuing. All samples bound by the unacceptable CCV must be reanalyzed. Samples with results less than the reporting limit may be reported with a CCV with high recovery.

16.2 MB/ICB/CCB –

- Frequency - Analyze immediately after each ICV and CCV – every 10 samples.
- Limits - The result must be less than the reporting limit. For DOD samples, the blanks must be less than ½ the LOQ. Samples with a result less than the reporting limit or greater than 10 times the contamination may be reported without qualification.
- Corrective Action – If the result is above the reporting limit, correct the problem and re-run the samples bound by the unacceptable CCB or raise the reporting limit.

16.3 LCS –

- Frequency - Prepare one for every 20 samples.
- Limits - LCS recovery must be 90-110% of the true value. Samples less than the reporting limit that are associated with a LCS that is above limits may be reported without qualification.
- Corrective Action - If the LCS does not meet limits, correct the problem and reanalyze the affected samples or flag the data.

16.4 Matrix Spike/ Matrix Spike Duplicate –

- Frequency - Analyze one matrix spike/MSD pair for every 10 samples. Do not choose field blanks as samples for the analysis of matrix spikes.
- Limits - Matrix spike recovery must be within the limits in the Data Quality Objectives Table. The RPD must be within 20%.
- Corrective Action - if recovery of the MS fails on a sample, the batch is considered acceptable as long as the LCS meets acceptance criteria. It is recommended that the MS/MSD be reprepared and reanalyzed to confirm the outliers, however it is not required due to probable matrix interferences. Flag the outlier. If the RPD is out of limits, repeat the MS/MSD unless there is assignable matrix interference, historical failures, or lack of volume. If an out of control MS/MSD is not repeated, note the reason on the data quality checklist. If, at the time when the problem is discovered, the sample exceeds twice the holding time, discuss with supervisor or Project Manager prior to repeating the samples. Report all of the replicates and explain in the checklist for the case narrative

17) Data Records Management

Records are maintained according to CE-GEN003 and ADM-ARCH.

18) Contingencies for Handling Out-of-Control Data

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls cannot be met, flag or narrate appropriately.

19) Method Performance

Detection and Quantitation limits are based upon an MDL study performed according to CE-QA011.

Demonstration of Capability is performed according to CE-QA003. The documentation of this method performance is retained by the Quality Assurance office.

Accuracy and Precision data may be found in the EPA method. The method states that single laboratory precision data can be estimated at 50% to 75% of the Interlaboratory precision estimates.

20) Summary of Changes

- Added Gallery Discrete analyzer throughout
- Incorporated Change Forms for Matrix Spike Duplicate (12/14/16), DOD MB limit of <1/2LOQ (1/7/16), sample loop length (8/20/15), and linear range verification (5/15/15).
- Combined low level and regular level NO2+NO3 curves and QC throughout.

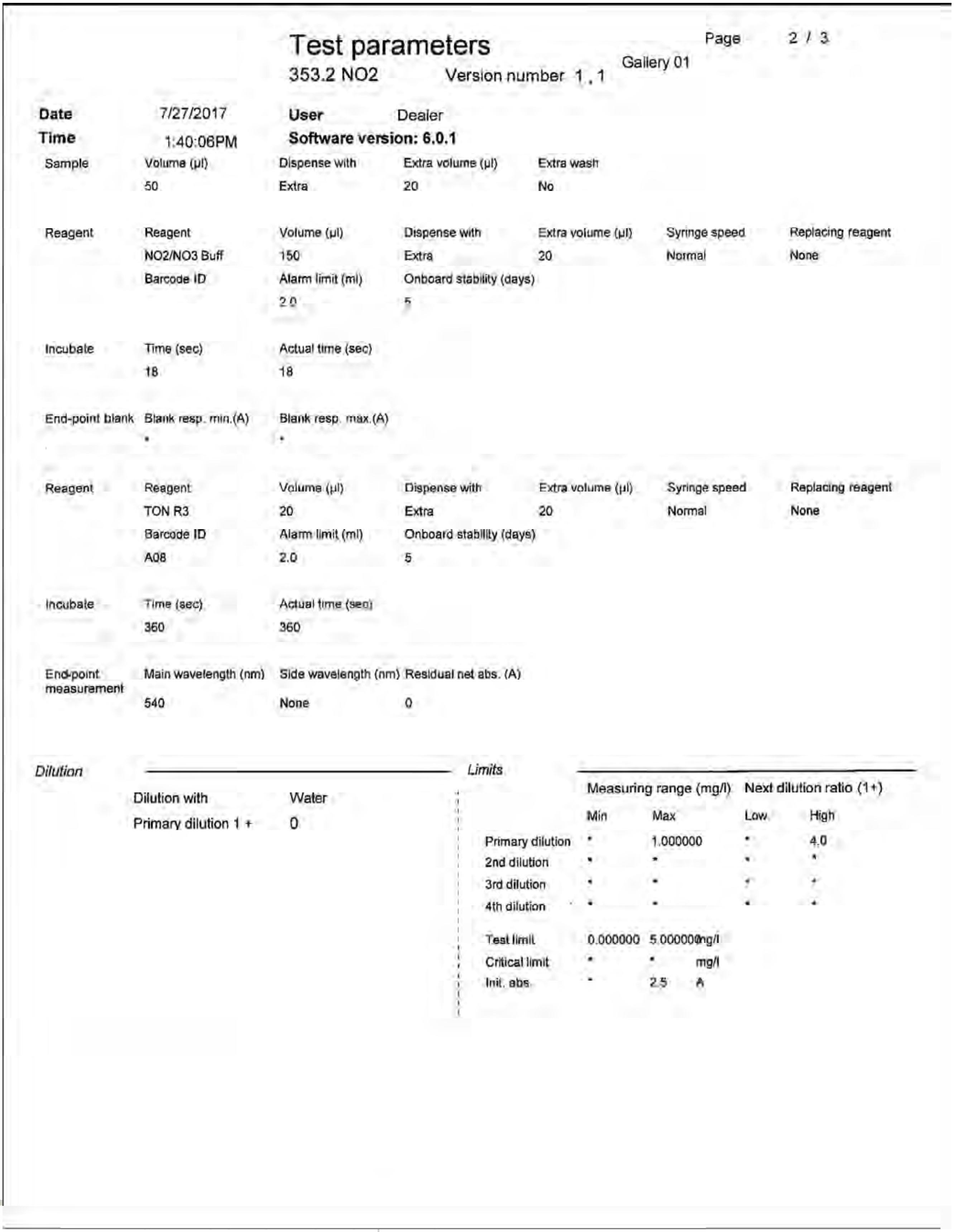
21) References and Related Documents

- Standard Methods for Chemical Analyses of Water and Wastewater, 18th ed. (1992), APHA-AWWA-WPCF, Part 4500, pp. 4-85, 4-87, 4-91, 4-74.
- Lachat QuikChem Method [10-107-04-1-C].
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW-846, September 1986.
- Methods for the Determination of Inorganic Substances in Environmental Samples. EPA/600/R-93/100. August 1993.
- Department of Defense (DOD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013.
- Department of Defense (DOD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1, 2017.
- Thermo Fisher Scientific Product Bulletin, Nitrite Application for Thermo Fisher Gallery Discrete Analyzer, version 1.
- EST Analytical Technical Bulletin, Nitrite, v112707.

22) Appendix

Note: Attachments are included here for illustration only. These attachments are controlled separately on the Forms sections of the Rochester intranet

- Gallery Test Parameters – NO2
- Calibration Standard Preparation – job aids for Nitrite and for Nitrite+Nitrate



Page 2 / 3

Test parameters

353.2 NO2 Version number 1 , 1

Gallery 01

Date	7/27/2017	**User**	Dealer
Time	1:40:06PM	**Software version: 6.0.1**	

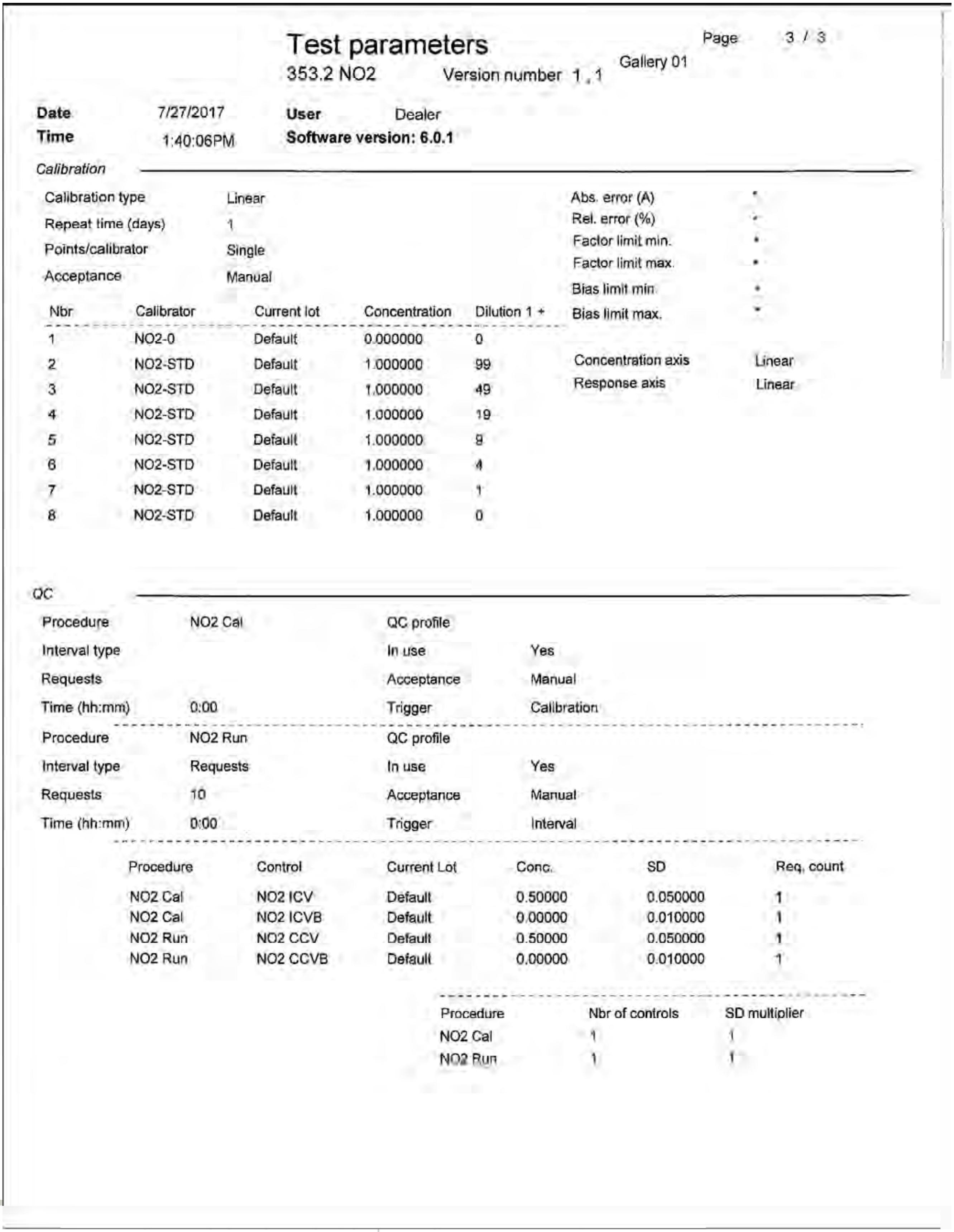
Sample	Volume (µl)	Dispense with	Extra volume (µl)	Extra wash		
	50	Extra	20	No		
Reagent	Reagent	Volume (µl)	Dispense with	Extra volume (µl)	Syringe speed	Replacing reagent
	NO2/NO3 Buff	150	Extra	20	Normal	None
	Barcode ID	Alarm limit (ml)	Onboard stability (days)			
		2.0	5			
Incubate	Time (sec)	Actual time (sec)				
	18	18				
End-point blank	Blank resp. min.(A)	Blank resp. max.(A)				
	*	*				
Reagent	Reagent	Volume (µl)	Dispense with	Extra volume (µl)	Syringe speed	Replacing reagent
	TON R3	20	Extra	20	Normal	None
	Barcode ID	Alarm limit (ml)	Onboard stability (days)			
	A08	2.0	5			
Incubate	Time (sec)	Actual time (sec)				
	360	360				
End-point measurement	Main wavelength (nm)	Side wavelength (nm)	Residual net abs. (A)			
	540	None	0			

Dilution

Dilution with	Water
Primary dilution 1 +	0

Limits

	Measuring range (mg/l)			Next dilution ratio (1+)	
	Min	Max		Low	High
Primary dilution	*	1.000000		*	4.0
2nd dilution	*	*		*	*
3rd dilution	*	*		*	*
4th dilution	*	*		*	*
Test limit	0.000000	5.000000	mg/l		
Critical limit	*	*	mg/l		
Init. abs	*	2.5	A		



Page 3 / 3

Test parameters

353.2 NO2 Version number 1 . 1

Gallery 01

Date	7/27/2017	**User**	Dealer
Time	1:40:06PM	**Software version: 6.0.1**	

Calibration

Calibration type	Linear	Abs. error (A)	*
Repeat time (days)	1	Rel. error (%)	*
Points/calibrator	Single	Factor limit min.	*
Acceptance	Manual	Factor limit max.	*
		Bias limit min.	*
		Bias limit max.	*

Nbr	Calibrator	Current lot	Concentration	Dilution 1 +
1	NO2-0	Default	0.000000	0
2	NO2-STD	Default	1.000000	99
3	NO2-STD	Default	1.000000	49
4	NO2-STD	Default	1.000000	19
5	NO2-STD	Default	1.000000	9
6	NO2-STD	Default	1.000000	4
7	NO2-STD	Default	1.000000	1
8	NO2-STD	Default	1.000000	0

Concentration axis	Linear
Response axis	Linear

QC

Procedure	NO2 Cal	QC profile	
Interval type		In use	Yes
Requests		Acceptance	Manual
Time (hh:mm)	0:00	Trigger	Calibration

Procedure	NO2 Run	QC profile	
Interval type	Requests	In use	Yes
Requests	10	Acceptance	Manual
Time (hh:mm)	0:00	Trigger	Interval

Procedure	Control	Current Lot	Conc.	SD	Req. count
NO2 Cal	NO2 ICV	Default	0.50000	0.050000	1
NO2 Cal	NO2 ICVB	Default	0.00000	0.010000	1
NO2 Run	NO2 CCV	Default	0.50000	0.050000	1
NO2 Run	NO2 CCVB	Default	0.00000	0.010000	1

Procedure	Nbr of controls	SD multiplier
NO2 Cal	1	1
NO2 Run	1	1

NITRITE [NO_2] (1.000 - 0.000 (pql=0.010))

A.) 10 mg/L WORKING STOCK:
do two (2) 1/10 serial dilutions of the 1000 mg/L NO_2 (Nitrite) Standard Stock.

B.) STANDARDS:

conc. (mg/L)	mL 10 mg/L	mL DI
a.) 1.000	1.00	9.00
b.) 0.500	0.50	9.50
c.) 0.200	0.20	9.80
d.) 0.100	1/10 dil'n of a.) 1.000	
e.) 0.050	1/10 dil'n of b.) 0.500	
f.) 0.020	1/10 dil'n of c.) 0.200	
g.) 0.010	1/10 dil'n of d.) 0.100	
h.) 0.000	10 mL DI water	

C.) ICV / CCV: (True Value = 0.50 mg/L)
9.5 mL DI + 0.5 mL 10.0 mg/L Reference Stock (two (2) 1/10 serial dilution of 1000 mg/L NO_2 (Nitrite) Ref. Stock.)

D.) LCS / Matrix Spike: (True Value = 0.250 mg/L)
10.0 mL DI / sample + 0.250mL 10 mg/L (Nitrite) Working Stock.

TOTN [$NO_3 + NO_2$] (2.000 - 0.000 (pql=0.002 mg/L))

A.) 10.0 mg/L WORKING STOCK:
do two (2) 1/10 serial dilutions of the 1000 mg/L NO_3 (Nitrate) Standard Stock.

B.) STANDARDS:

conc. (mg/L)	mL 10.0 mg/L	mL DI Water
a.) 2.000	2.00	8.00
b.) 1.000	1.00	9.00
c.) 0.500	0.50	9.50
d.) 0.200	0.20	9.80
e.) 0.100	1/10 dil'n of b.) 1.000	
f.) 0.050	1/10 dil'n of c.) 0.500	
g.) 0.010	1/10 dil'n of e.) 0.100	
h.) 0.005	1/10 dil'n of f.) 0.050	
i.) 0.002	(2x) 1/10 dil'n of d.) 0.200	
j.) 0.000	10 mL of DI Water	

C.) ICV / CCV: (True Value = 0.50 mg/L)
do two (2) 1/10 serial dilutions of 1000 mg/L NO_3 (Nitrate) Reference Stock Solution. Add 0.50 mL of 10 mg/L Ref. stock to 9.0 mL DI.

D.) LCS / Matrix Spike: (True Value = 0.200 mg/L)
10.0 mL DI / sample + 0.020 mL 100 mg/L (Nitrate) Working Stock.

E.) COLUMN CHECK:

- 0.5 mg/L NO_3 = 0.500 cal. std. c
- 0.5 mg/L NO_2 = (2) two 1/10 serial dilutions of 1000 mg/L NO_2 (Nitrite) Standard Stock. Add 0.5 mL of the 10 mg/L to 9.5 mL DI.

SOP CHANGE FORM

SOP Title: Total Suspended Solids
SOP Code: GEN-2540D
SOP Revision No.: 2
SOP Date: 8/31/15
SOP Section(s) Affected by Change: 11.12

Description of Change:
Remove need to record twice daily temperatures.

Reason(s) for Change(s):
Changes to daily temp protocols – see new ADM-DALYCK.

Change(s) Submitted by: Vicky Collom	Date: 11/12/19

Approvals:

Technical Reviewer Signature:	Date: 11/12/19
QA Signature:	Date: 11/12/19
Laboratory Director Signature:	Date: 11/12/19

Change(s) Effective Date: 11/4/19

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title: Total Suspended Solids

SOP Code: GEN-2540D

SOP Revision No.: 2

SOP Date: 8/31/15

SOP Section(s) Affected by Change: 9, 12.6

Description of Change:

9. Label desiccators with the start date and expiration of the desiccant. The desiccant expires in 3 months or sooner if no longer blue.

12.8 When finished placing hot dishes in the desiccator, place the lid on the desiccator, leaving a 1-2 cm gap. Start a timer for 10 minutes. Close the desiccator completely when the timer alarms.

12.3+12.6 The first measured volume must be <1/2 the total volume received, unless historical data for this sample indicates that the entire volume will pass. If the entire first measured volume does not pass the filter (the filter clogs), discard the filter. Cap and shake the bottle and add a smaller measured volume to a new filter. Do not add any volume remaining in the graduated cylinder back to the sample bottle. Do not add portions of the measured volume and calculate the volume that passed by difference.

If the first measured volume passes the filter and it appears that more volume is needed to be added to meet the reporting limit, estimate the volume expected to pass, cap and shake the sample bottle, measure and filter the aliquot through the same filter. Use small enough aliquots that it is unlikely they will clog the filter. If, however, the estimation was incorrect, and there will be insufficient sample to repeat with a new filter, measure by difference and note the problem on the benchsheet.

Reason(s) for Change(s):
RC0549, RC0550

Change(s) Submitted by: Vicky Collom	Date: 2/24/17

Approvals:

Technical Reviewer Signature:	Date: 2/24/17
QA Signature:	Date: 2/24/17
Laboratory Director Signature:	Date: 2/28/17

Change(s) Effective Date:

SOP user acknowledgement:

Distribution: Original filed with original SOP Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title:	Total Suspended Solids (TSS)
SOP Code: GEN-2540D	
SOP Revision No.: 2	
SOP Date: 8/31/15	
SOP Section(s) Affected by Change: 12.1	

Description of Change:
Add that for in-house tared TSS filters, remove the dried filter from the tin and place the filter directly on the balance to obtain the tare weight.

Reason(s) for Change(s):
RC0319 For procedural consistency when weighing final weights that include both in-house and Proweigh filters.

Change(s) Submitted by: Vicky Collom	Date: 3/7/16

Approvals:

Technical Reviewer Signature:	Date: 3/7/16
QA Signature:	Date: 3/7/16
Operations Manager Signature:	Date: 3/11/16

Change(s) Effective Date:

SOP user acknowledgement:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

SOP CHANGE FORM

SOP Title:	Total Suspended Solids (TSS)
SOP Code: GEN-2540D	
SOP Revision No.: 2	
SOP Date: 8/31/15	
SOP Section(s) Affected by Change: 16.3, 21	

Description of Change:

16.3 MB - Add DOD limit of <1/2LOQ.

21 – Add to References – Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0, July 2013

Reason(s) for Change(s):
Addition of TSS to DOD scope.

Change(s) Submitted by: Vicky Collom	Date: 10/14/15

Approvals:

Technical Reviewer Signature:	Date: 10/15/15
QA Signature:	Date: 10/15/15
Operations Manager Signature:	Date: 10/15/15

Change(s) Effective Date: 10/14/15

SOP user acknowledgement:

Distribution: Original filed with original SOP   Photocopy attached to each controlled copy

ALS Standard Operating Procedure

DOCUMENT TITLE: TOTAL SUSPENDED SOLIDS (TSS)
REFERENCED METHOD: SM 2540D-1997(2011)
SOP ID: GEN-2540D
REV. NUMBER: 2
EFFECTIVE DATE: 8/31/15

ALS Environmental

TOTAL SUSPENDED SOLIDS (TSS)

SM 2540D-1997(2011)

SOPID:	GEN-2540D	Rev. Number:	2	Effective Date:	8/31/15

Approved By: Date: 9/2/15
Department Supervisor - Christine Kutzer

Approved By: Date: 9/2/15
QA Manager – Vicky Collom

Approved By: Date: 9/2/15
Operations Manager – Jason Badman

Archival Date:		Doc Control ID#:	15-GEN-01 On 9/2/15	Editor:	

TABLE OF CONTENTS

TOTAL SUSPENDED SOLIDS (TSS)

1) Scope and Applicability

1.1 This SOP uses Standard Methods 2540D (1997-2011) for the measurement of total suspended solids (non-filterable residue) in drinking, surface, and saline waters, domestic and industrial wastes.

1.2 Range of determination is 1 mg/L to 20,000 mg/L.

1.3 MRL is 1 mg/L when 1000 mL of sample are filtered.

2) Summary of Procedure

A well-mixed sample is filtered through a standard glass-fiber filter and the residue retained on the filter is dried to a constant weight at 103-105°C. The increased weight of the filter represents the total suspended solids.

3) Definitions

3.1 Duplicate Sample (DUP) - A laboratory duplicate. The duplicate sample is a separate sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.2 Method Blank (MB)- The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.3 Laboratory Control Sample (LCS) – A solution with a known value is prepared and analyzed as a sample. Percent recoveries are calculated for the analyte detected.

3.4 Batch – a group of no more than 20 samples analyzed together. See ADM-BATCH for more detail.

3.5 Relative Percent Difference (RPD) – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

4) Health and Safety Warnings

4.1 All appropriate safety precautions for handling samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

4.2 Refer to the Environmental, Health and Safety Manual for other general safety information.

4.3 Use a fume hood on the 104 oven.

4.4 Waste Management and Pollution Prevention

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the EH&S Manual. Samples and sample residue may be washed down the drain. See SMO-SPLDIS for further discussion on waste management.

5) Cautions

5.1 Replace desiccant in desiccators as needed (when desiccant is no longer blue). Desiccant may be dried and reused

5.2 Keep balance clean and dry. See ADM-DALYCK for balance information.

5.3 Add oil to the vacuum pump as needed.

5.4 Be sure dishes are cleaned according to GEN-GC.

6) Interferences

6.1 Nonrepresentative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded if it is determined that their inclusion is not desired in the final result.

6.2 Excessive residue on the filter may form a water-entrapping crust. Limit the sample size to that yielding no more than 200 mg residue.

6.3 For samples high in total dissolved solids, thoroughly wash the filter to ensure removal of dissolved material.

6.4 Prolonged filtration times resulting from filter clogging may produce high results because of increased colloidal materials captured on the clogged filter.

6.5 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because they can affect the results.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

7.2 Training – see CE-QA003.

8) Sample Collection, Containers, and Storage

8.1 Samples should be collected in purchased, certified clean plastic or glass bottles or jars. Volumes collected should be sufficient to insure a representative sample, allow for replicate analysis (if necessary), and minimize waste disposal. Typically, a 1000 mL HDPE bottle is provided with bottle sets.

8.2 Samples are nonpreserved and kept at 0-6°C until analysis to minimize decomposition of solids.

8.3 Holding time is 7 days. Holding time for work under ASP is 5 days from verified time of sample receipt.

8.4 Sample handling, storage, and custody procedures are discussed in SOP SMO-GEN and SMO-ICOC.

9) Equipment and Supplies

- Glass fiber filter discs, 4.7cm, Whatman GF/C or
- Environmental Express Proweigh 47mm
- Filtration Apparatus
- Aluminum Drying Dishes
- Drying Oven capable of maintaining 103-105 °C.
- Desiccator with color indication desiccant
- Analytical Balance, capable of weigh to 0.1 mg. with U-Electrode Ionizer- calibrated according to ADM-DALYCK.
- Forceps
- Graduated Cylinder – Class A or verified within 3% of nominal volume.

10) Standards and Reagents

10.1 Kaolin powder – purchased commercially. Dry at 104 °C for at least 1 hour and cool to room temperature prior to being weighed. Dried Kaolin powder is stored at room temperature in a glass beaker covered with aluminum foil in a desiccator. Expires per Expiration Policy (CE-QA012).

10.2 Reference/LCS: To a tared plastic bottle, add 0.20 -0.30 g of dried Kaolin powder. Record actual weight used. Add DI water to 1000 g. The true value will be between 200 and 300 mg/L. Use as if it were a client sample. Store at 0-6 °C for up to one year.

10.3 MB – Use DI as if it were a client sample.

11) Method Calibration

Balance Calibration – The balance calibration is checked daily prior to use according to ADM-DALYCK. ADM-DALYCK also addresses balance use, limits, corrective action, documentation, and annual check of weights.

12) Sample Preparation and Analysis

Note: Handle filter with forceps at all times.

12.1 For Volatile Suspended Solids, use Whatman glass fiber filters and prepare as follows. For Total Suspended Solids, if using Environmental Express Proweigh Filters and skip the following filter preparation steps.

12.1.1 Place the disc on membrane filter apparatus. While vacuum is on, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings. Remove filter from the apparatus and place in a numbered crucible. Record the lot number of the filters on the benchsheet. Dry the filters at 550°C for at least 20 minutes. Remove to a desiccator and cool for at least 30 minutes.

12.1.2 Weighing of filters – Weigh the 20.0000g Class 1 weight and record on the tare sheet. Transfer crucible containing filter to the analytical balance and record the ID number. Weigh dish twice; successive weights must be within 0.5 mg of each other. Record the balance ID.

12.2 Prepare a sequence of samples and QC in compliance with the frequency requirements in ADM-BATCH and the QC section of this SOP. Prioritize samples according to holding times and rush dates. Use the benchsheet appropriate to the test (TSS or VSS). An example TSS benchsheet is attached to this SOP. An example VSS benchsheet is attached to GEN-160.4.

12.3 Selection of sample volume - The target residue on the filter is 10-200 mg. For clean samples, choose a sample volume up to 1000 mL. Smaller sample volumes will be needed for dirty samples. If the entire measured volume will not pass through the filter, record, by difference, the actual volume filtered. If filtration time exceeds 10 minutes, discard the clogged filter and repeat with a new filter set and a smaller sample volume.

12.4 Transfer the tared filter to the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to fit it to fritted support.

12.5 Shake the sample vigorously and transfer the predetermined sample volume selected to the filter using a graduated cylinder. Type the volume into the spreadsheet with the corresponding sample and dish ID. Verify the volume and mark the verification on the spreadsheet.

12.6 Filter the sample through the glass fiber filter. Record volume used on benchsheet (attached).

12.7 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after all the water has passed through. A 3 minutes dry time is mentioned in the method, but less time is typically used because the vacuum will start to pull the filter through the apparatus before 3 minutes.

12.8 Carefully remove the filter and place into the aluminum dish or crucible. Dry at least one hour at 103-105°C. Record the ID of the oven used. Cool in a desiccator. For TSS, remove the filter from the dish and weigh the filter. For VSS, weigh the entire filter and crucible. Record the balance ID.

12.9 Repeat the drying cycle until a constant weight is obtained (difference in weight should not be greater than 0.0005 g or 4% of previous weight). Record the second weighing on the bench sheet.

12.10 If the samples are to undergo volatile solids analysis, retain the dish and filter in a desiccator and proceed with GEN-160.4/SM2540E.

12.11 The temperature of the oven is recorded twice daily in a designated log book to maintain a compliant operating temperature. The time and temperature is also recorded in the "In/Out" log book whenever samples are put in or taken from the oven.

13) Troubleshooting

Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by laboratory staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. This applies to the oven log used for daily temperature checks and balance log used for calibration and daily checks.

The temperature of the oven is recorded twice daily in a designated log book to maintain a compliant operating temperature. The time and temperature is also recorded in the "In/Out" log book whenever samples are put in and once oven is at temperature.

14) Data Acquisition

Data is recorded manually into a controlled Excel spreadsheet. Tare weights from the pre-weighed filters are downloaded from the manufacturer website and applied to the Excel benchsheet electronically. Data is uploaded from the spreadsheet to LIMS.

15) Calculation and Data Reduction Requirements

$$\text{mg total suspended solids / L} = \frac{(A - B) \times 1000{,}000}{\text{sample volume, mL}}$$

Where: A= weight of dried residue + dish (g)
B= weight of dish (g)

Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV. Run creation, data uploading, and run approval procedures are discussed in ADM-DREV.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 Run one duplicate per batch of ten samples. The DUP should have an RPD < 10%. If the sample is low level (≤5 times the PQL), the RPD may exceed 10%, but the difference between results must be less than ≤ the PQL. If the RPD of the duplicates is >20%, repeat the sample and duplicate unless there is assignable matrix interference, historical failures, or lack of volume. If an out of control duplicate is not repeated, note the reason on the data quality checklist. If, at the time when the problem is discovered, the sample exceeds twice the holding time, discuss with supervisor or Project Manager prior to repeating the samples. Report all of the replicates and explain in the checklist for the case narrative.

16.2 Run a Laboratory Control Sample per batch of 20 samples. The LCS recovery should be within the limits set in the Wetchem QC table in Appendix C of the Quality Assurance Manual. If it is not, determine the cause, fix the problem, and repeat the samples if volume and holding time permits. If not, flag the samples. If the LCS recovery is greater than its upper limit, associated non-detect samples may be reported.

16.3 Analyze a Method Blank for every 20 or fewer samples. The result of the blank must be less than the PQL (Final weights of blanks must vary no more than 1.0 mg from the tare). If it is not, the associated sample results may be used if they are less than the PQL or greater than 10 times the blank value. If this criteria is not met, repeat the samples if volume and holding time permits, or flag the data.

17) Data Records Management

See CE-GEN-003 and ADM-ARCH

18) Contingencies for Handling Out Of Control Data

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, flag and narrate appropriately.

19) Method Performance

19.1 Demonstration of Capability is performed according to CE-QA003.

19.2 Precision Data is available in the referenced method SM 20 2540D.

20) Summary of Changes

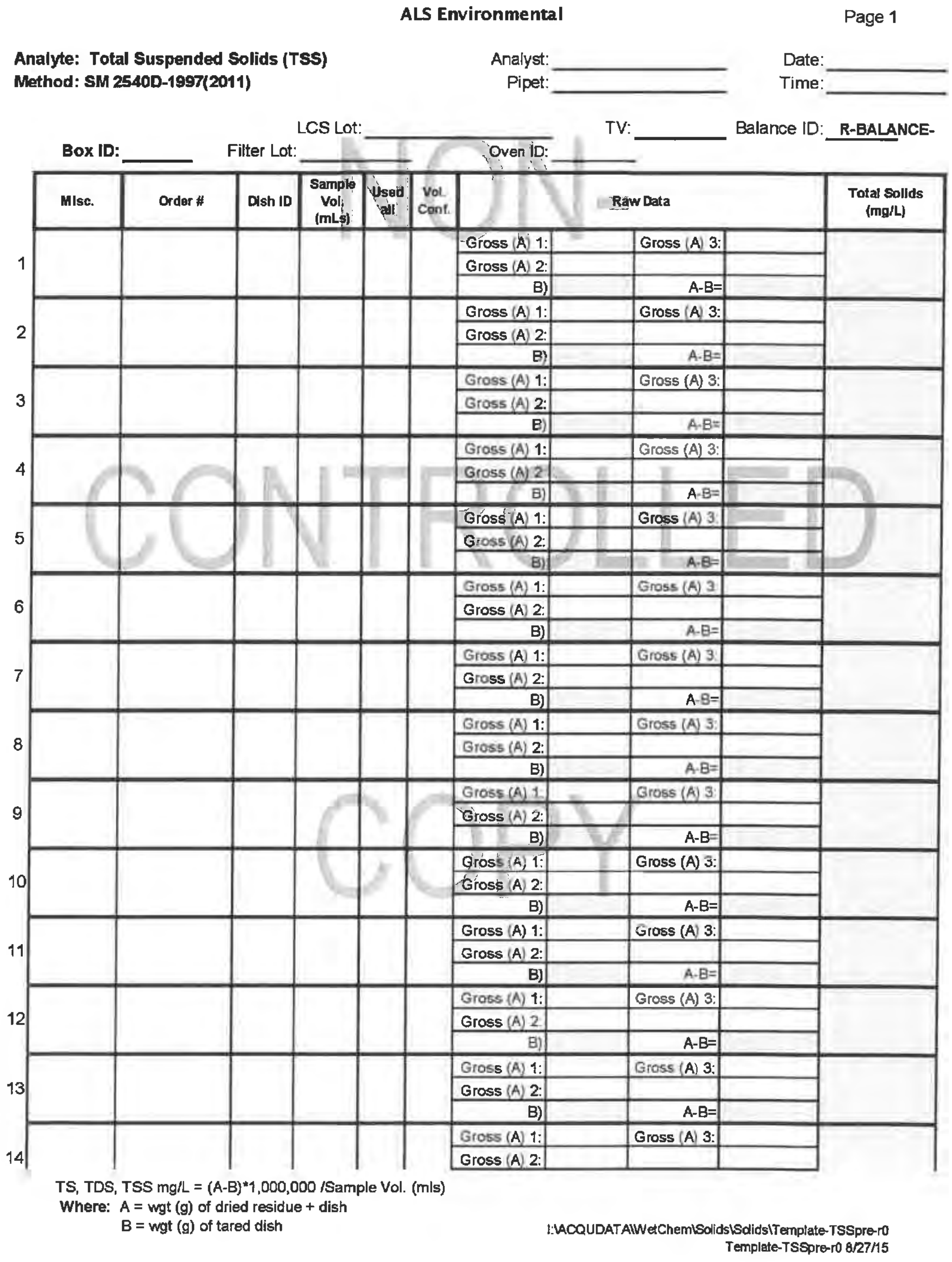
- Added use of purchased, pre-weighed filters throughout.
- Incorporated SOP change form for tightening the DUP limit to <10% RPD with corrective action at 20% RPD.

21) References and Related Documents

- APHA Standard Methods for the Examination of Water and Wastewater, American Public Health Association, Washington, D.C. Method 2540D. 1997-2011.

22) Attachments

For example only – forms are controlled on the Controlled Forms section of the Rochester Intranet.



ALS Environmental

Page 1

Analyte: Total Suspended Solids (TSS)
Method: SM 2540D-1997(2011)

Analyst: ________________
Pipet: ________________

Date: __________
Time: __________

LCS Lot: ________________
TV: __________
Balance ID: R-BALANCE-

Box ID: _______
Filter Lot: __________
Oven ID: _______

	Misc.	Order #	Dish ID	Sample Vol. (mLs)	Used all	Vol. Conf.	Raw Data				Total Solids (mg/L)
1							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
2							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
3							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
4							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
5							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
6							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
7							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
8							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
9							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
10							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
11							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
12							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
13							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				
							B)		A-B=		
14							Gross (A) 1:		Gross (A) 3:		
							Gross (A) 2:				

TS, TDS, TSS mg/L = (A-B)*1,000,000 /Sample Vol. (mls)
Where: A = wgt (g) of dried residue + dish
B = wgt (g) of tared dish

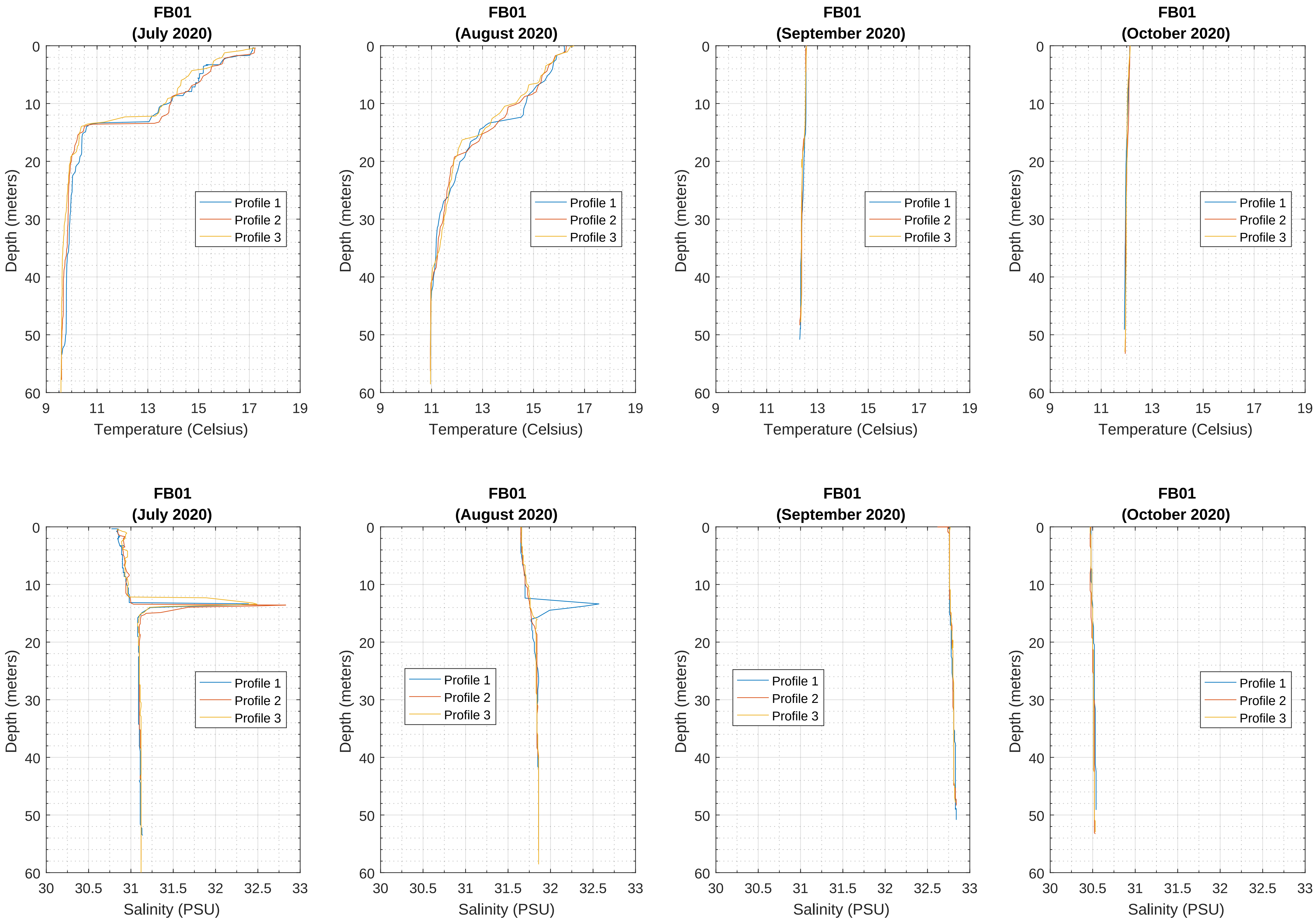
I:\ACQUDATA\WetChem\Solids\Solids\Template-TSSpre-r0
Template-TSSpre-r0 8/27/15

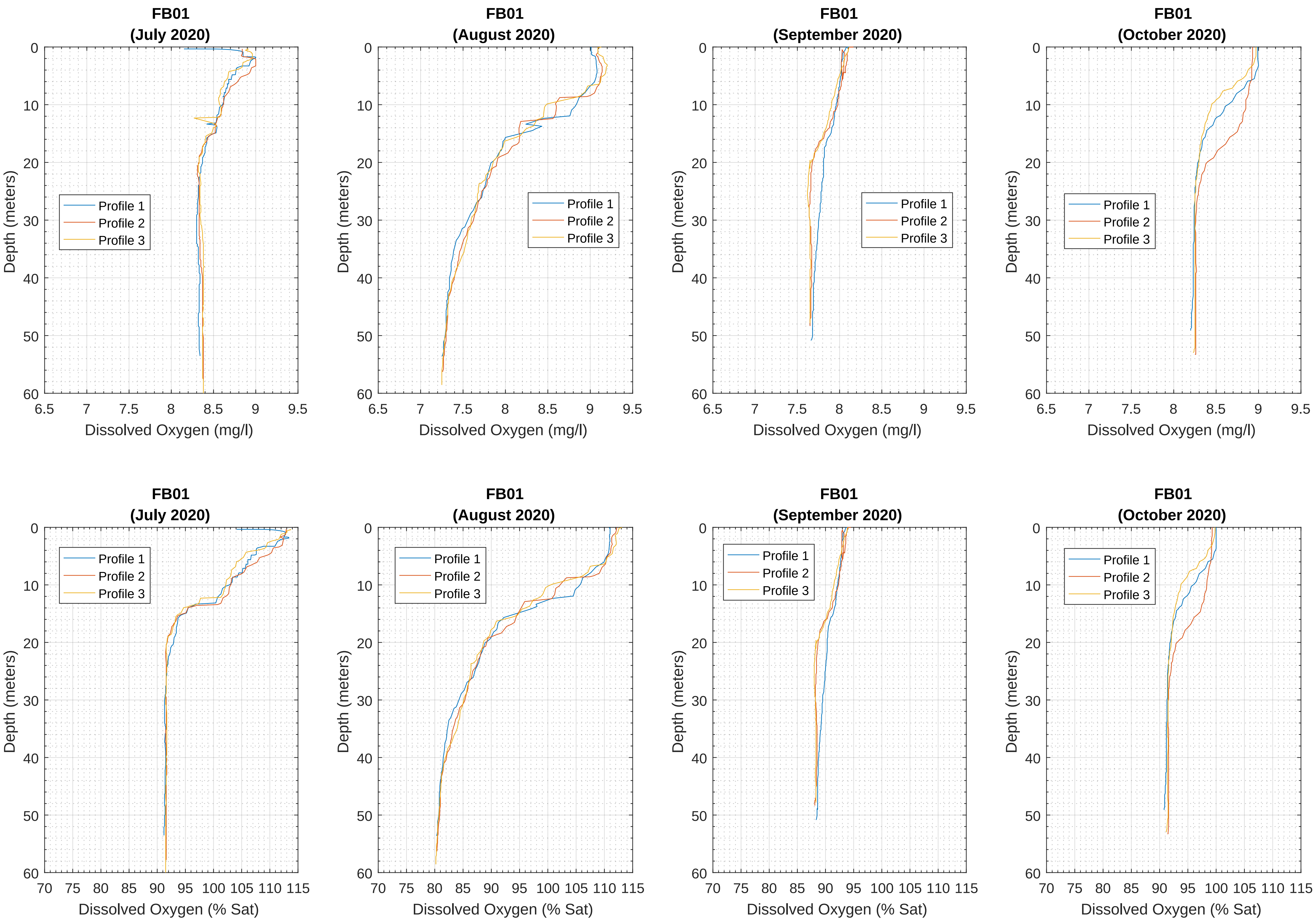
APPENDIX B

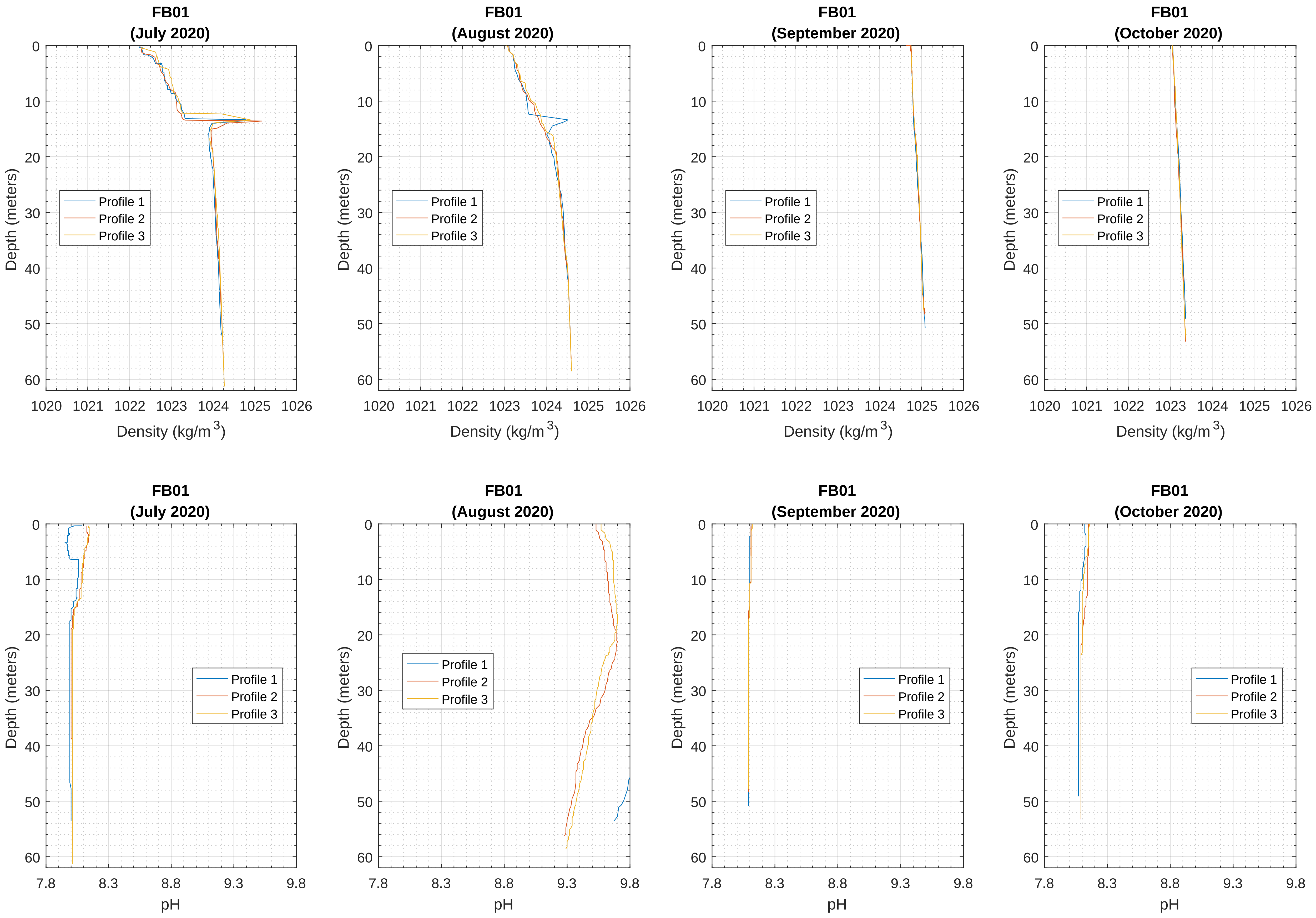
In-situ Water Parameter Vertical Profile Graphs

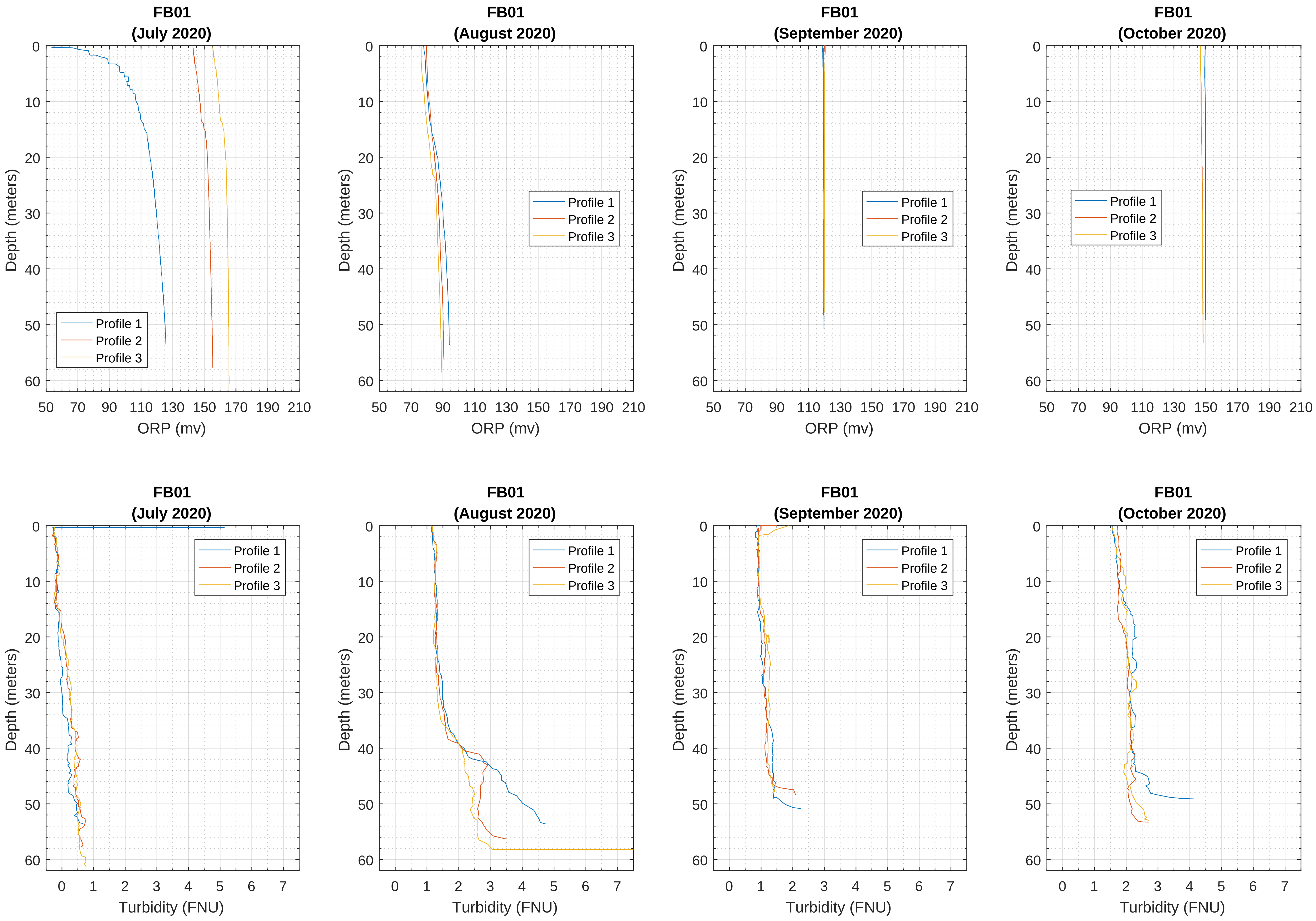
Water Quality Monitoring Report
Frenchman Bay Site FB01
Hancock County, Maine

Ransom Consulting, LLC
Project 201.05012


FB01
(July 2020)
FB01
(August 2020)
FB01
(September 2020)
FB01
(October 2020)
Depth (meters)
0
10
20
30
40
50
60
Temperature (Celsius)
9
11
13
15
17
19
Salinity (PSU)
30
30.5
31
31.5
32
32.5
33
Profile 1
Profile 2
Profile 3


FB01
(July 2020)
FB01
(August 2020)
FB01
(September 2020)
FB01
(October 2020)
Depth (meters)
0
10
20
30
40
50
60
6.5
7
7.5
8
8.5
9
9.5
Dissolved Oxygen (mg/l)
70
75
80
85
90
95
100
105
110
115
Dissolved Oxygen (% Sat)
Profile 1
Profile 2
Profile 3


FB01
(July 2020)
FB01
(August 2020)
FB01
(September 2020)
FB01
(October 2020)
Depth (meters)
0
10
20
30
40
50
60
Density (kg/m ^{3})
1020
1021
1022
1023
1024
1025
1026
Profile 1
Profile 2
Profile 3
pH
7.8
8.3
8.8
9.3
9.8


FB01
(July 2020)
FB01
(August 2020)
FB01
(September 2020)
FB01
(October 2020)
Depth (meters)
0
10
20
30
40
50
60
ORP (mv)
50
70
90
110
130
150
170
190
210
Profile 1
Profile 2
Profile 3
Turbidity (FNU)
0
1
2
3
4
5
6
7

APPENDIX C

Laboratory Analytical Reports

Water Quality Monitoring Report
Frenchman Bay Site FB01
Hancock County, Maine

Ransom Consulting, LLC
Project 201.05012

ANALYTICAL REPORT

Lab Number:	L2031016
Client:	Ransom Consulting, Inc. 112 Corporate Drive Pease International Tradeport Portsmouth, NH 03801
ATTN:	Elizabeth Ransom
Phone:	(603) 436-1490
Project Name:	AA
Project Number:	201.05012.002
Report Date:	09/08/20

Certifications & Approvals: MA (M-MA086), NH NELAP (2064), CT (PH-0574), IL (200077), ME (MA00086), MD (348), NJ (MA935), NY (11148), NC (25700/666), PA (68-03671), RI (LAO00065), TX (T104704476), VT (VT-0935), VA (460195), USDA (Permit #P330-17-00196).

Eight Walkup Drive, Westborough, MA 01581-1019
508-898-9220 (Fax) 508-898-9193 800-624-9220 - www.alphalab.com

Project Name:	AA	**Lab Number:**	L2031016
Project Number:	201.05012.002	**Report Date:**	09/08/20

Alpha Sample ID	Client ID	Matrix	Sample Location	Collection Date/Time	Receive Date
L2031016-01	FB01-G1-193	WATER	MAINE	07/22/20 09:45	07/22/20
L2031016-02	FB01-G1-100	WATER	MAINE	07/22/20 10:15	07/22/20
L2031016-03	FB01-G1-1	WATER	MAINE	07/22/20 10:40	07/22/20
L2031016-04	FB02-G1-BOT	WATER	MAINE	07/22/20 13:45	07/22/20
L2031016-05	FB02-G1-MID	WATER	MAINE	07/22/20 14:05	07/22/20
L2031016-06	FB02-G1-SUR	WATER	MAINE	07/22/20 14:25	07/22/20

Case Narrative

The samples were received in accordance with the Chain of Custody and no significant deviations were encountered during the preparation or analysis unless otherwise noted. Sample Receipt, Container Information, and the Chain of Custody are located at the back of the report.

Results contained within this report relate only to the samples submitted under this Alpha Lab Number and meet NELAP requirements for all NELAP accredited parameters unless otherwise noted in the following narrative. The data presented in this report is organized by parameter (i.e. VOC, SVOC, etc.). Sample specific Quality Control data (i.e. Surrogate Spike Recovery) is reported at the end of the target analyte list for each individual sample, followed by the Laboratory Batch Quality Control at the end of each parameter. Tentatively Identified Compounds (TICs), if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported. If a sample was re-analyzed or re-extracted due to a required quality control corrective action and if both sets of data are reported, the Laboratory ID of the re-analysis or re-extraction is designated with an "R" or "RE", respectively.

When multiple Batch Quality Control elements are reported (e.g. more than one LCS), the associated samples for each element are noted in the grey shaded header line of each data table. Any Laboratory Batch, Sample Specific % recovery or RPD value that is outside the listed Acceptance Criteria is bolded in the report. In reference to questions H (CAM) or 4 (RCP) when "NO" is checked, the performance criteria for CAM and RCP methods allow for some quality control failures to occur and still be within method compliance. In these instances, the specific failure is not narrated but noted in the associated QC Outlier Summary Report, located directly after the Case Narrative. QC information is also incorporated in the Data Usability Assessment table (Format 11) of our Data Merger tool, where it can be reviewed in conjunction with the sample result, associated regulatory criteria and any associated data usability implications.

Soil/sediments, solids and tissues are reported on a dry weight basis unless otherwise noted. Definitions of all data qualifiers and acronyms used in this report are provided in the Glossary located at the back of the report.

HOLD POLICY - For samples submitted on hold, Alpha's policy is to hold samples (with the exception of Air canisters) free of charge for 21 calendar days from the date the project is completed. After 21 calendar days, we will dispose of all samples submitted including those put on hold unless you have contacted your Alpha Project Manager and made arrangements for Alpha to continue to hold the samples. Air canisters will be disposed after 3 business days from the date the project is completed.

Please contact Project Management at 800-624-9220 with any questions.

Case Narrative (continued)

Report Revision

September 08, 2020: At the client's request, The Total Nitrogen calculation has been removed.

August 18, 2020: All non-detect (ND) or estimated concentrations (J-qualified) have been quantitated to the limit noted in the MDL column.

Nitrogen, Ammonia

The WG1396135-4 MS recovery, performed on L2031016-02, is outside the acceptance criteria for nitrogen, ammonia (88%); however, the associated LCS recovery is within criteria. No further action was taken.

I, the undersigned, attest under the pains and penalties of perjury that, to the best of my knowledge and belief and based upon my personal inquiry of those responsible for providing the information contained in this analytical report, such information is accurate and complete. This certificate of analysis is not complete unless this page accompanies any and all pages of this report.

Authorized Signature: Kelly Stenstrom Kelly Stenstrom

Title: Technical Director/Representative Date: 09/08/20

INORGANICS
&
MISCELLANEOUS

Project Name: AA
Lab Number: L2031016
Project Number: 201.05012.002
Report Date: 09/08/20

SAMPLE RESULTS

Lab ID: L2031016-01
Client ID: FB01-G1-193
Sample Location: MAINE
Date Collected: 07/22/20 09:45
Date Received: 07/22/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	17.		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
Nitrogen, Ammonia	0.070	J	mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 21:54	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.073	J	mg/l	0.10	0.023	1	-	07/23/20 08:17	44,353.2	MR
Nitrogen, Total Kjeldahl	0.268	J	mg/l	0.300	0.066	1	08/06/20 04:25	08/06/20 22:20	4,351.3/.1 (M)	AT
Phosphorus, Total	0.028		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:22	121,4500P-E	SD
Phosphorus, Orthophosphate	0.019		mg/l	0.005	0.001	1	-	07/23/20 06:35	121,4500P-E	MA

SAMPLE RESULTS

Lab ID: L2031016-02
Client ID: FB01-G1-100
Sample Location: MAINE
Date Collected: 07/22/20 10:15
Date Received: 07/22/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	20.		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
Nitrogen, Ammonia	0.118		mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 21:55	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.085	J	mg/l	0.10	0.023	1	-	07/23/20 08:23	44,353.2	MR
Nitrogen, Total Kjeldahl	0.276	J	mg/l	0.300	0.066	1	08/06/20 04:25	08/06/20 22:23	4,351.3/.1 (M)	AT
Phosphorus, Total	0.024		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:23	121,4500P-E	SD
Phosphorus, Orthophosphate	0.019		mg/l	0.005	0.001	1	-	07/23/20 06:35	121,4500P-E	MA

SAMPLE RESULTS

Lab ID: L2031016-03
Client ID: FB01-G1-1
Sample Location: MAINE
Date Collected: 07/22/20 10:40
Date Received: 07/22/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	14.		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
Nitrogen, Ammonia	0.060	J	mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 21:58	44,350.1	AT
Nitrogen, Nitrate/Nitrite	ND		mg/l	0.10	0.023	1	-	07/23/20 08:29	44,353.2	MR
Nitrogen, Total Kjeldahl	0.384		mg/l	0.300	0.066	1	08/06/20 04:25	08/06/20 22:24	4,351.3/.1 (M)	AT
Phosphorus, Total	0.020		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:24	121,4500P-E	SD
Phosphorus, Orthophosphate	0.007		mg/l	0.005	0.001	1	-	07/23/20 06:36	121,4500P-E	MA

SAMPLE RESULTS

Lab ID:	L2031016-04	Date Collected:	07/22/20 13:45
Client ID:	FB02-G1-BOT	Date Received:	07/22/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	15.		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
Nitrogen, Ammonia	0.076		mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 21:59	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.070	J	mg/l	0.10	0.023	1	-	07/23/20 08:30	44,353.2	MR
Nitrogen, Total Kjeldahl	0.274	J	mg/l	0.300	0.066	1	08/06/20 04:25	08/06/20 22:25	4,351.3/.1 (M)	AT
Phosphorus, Total	0.017		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:28	121,4500P-E	SD
Phosphorus, Orthophosphate	0.024		mg/l	0.005	0.001	1	-	07/23/20 06:36	121,4500P-E	MA

Project Name:	AA	**Lab Number:**	L2031016
Project Number:	201.05012.002	**Report Date:**	09/08/20

SAMPLE RESULTS

Lab ID:	L2031016-05	Date Collected:	07/22/20 14:05
Client ID:	FB02-G1-MID	Date Received:	07/22/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	14.		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
Nitrogen, Ammonia	0.087		mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 22:00	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.060	J	mg/l	0.10	0.023	1	-	07/23/20 08:31	44,353.2	MR
Nitrogen, Total Kjeldahl	0.477		mg/l	0.300	0.066	1	08/06/20 04:25	08/06/20 22:26	4,351.3/.1 (M)	AT
Phosphorus, Total	0.030		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:29	121,4500P-E	SD
Phosphorus, Orthophosphate	0.024		mg/l	0.005	0.001	1	-	07/23/20 06:37	121,4500P-E	MA

Project Name:	AA	**Lab Number:**	L2031016
Project Number:	201.05012.002	**Report Date:**	09/08/20

SAMPLE RESULTS

Lab ID:	L2031016-06	Date Collected:	07/22/20 14:25
Client ID:	FB02-G1-SUR	Date Received:	07/22/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	13.		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
Nitrogen, Ammonia	0.061	J	mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 22:04	44,350.1	AT
Nitrogen, Nitrate/Nitrite	ND		mg/l	0.10	0.023	1	-	07/23/20 08:32	44,353.2	MR
Nitrogen, Total Kjeldahl	0.288	J	mg/l	0.300	0.066	1	08/06/20 04:25	08/06/20 22:30	4,351.3/.1 (M)	AT
Phosphorus, Total	0.022		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:30	121,4500P-E	SD
Phosphorus, Orthophosphate	0.008		mg/l	0.005	0.001	1	-	07/23/20 06:38	121,4500P-E	MA

Project Name: AA **Lab Number:** L2031016

Project Number: 201.05012.002 **Report Date:** 09/08/20

Method Blank Analysis
Batch Quality Control

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1394865-1										
Phosphorus, Orthophosphate	0.001	J	mg/l	0.005	0.001	1	-	07/23/20 06:29	121,4500P-E	MA
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1394887-1										
Nitrogen, Nitrate/Nitrite	ND		mg/l	0.10	0.023	1	-	07/23/20 08:38	44,353.2	MR
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1395199-1										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	07/25/20 10:15	121,2540D	JT
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1395682-1										
Phosphorus, Total	ND		mg/l	0.010	0.004	1	08/05/20 09:30	08/06/20 09:18	121,4500P-E	SD
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1396132-1										
Nitrogen, Total Kjeldahl	0.106	J	mg/l	0.300	0.022	1	08/06/20 04:25	08/06/20 22:18	4,351.3/.1 (M)	AT
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1396135-1										
Nitrogen, Ammonia	0.050	J	mg/l	0.075	0.024	1	08/06/20 05:43	08/06/20 21:51	44,350.1	AT

Lab Control Sample Analysis

Batch Quality Control

Project Name: AA
Project Number: 201.05012.002
Lab Number: L2031016
Report Date: 09/08/20

Parameter	LCS %Recovery	Qual	LCSD %Recovery	Qual	%Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1394865-2								
Phosphorus, Orthophosphate	93		-		90-110	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1394887-2								
Nitrogen, Nitrate/Nitrite	102		-		90-110	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1395199-2								
Solids, Total Suspended	100		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1395682-2								
Phosphorus, Total	94		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1396132-2								
Nitrogen, Total Kjeldahl	101		-		78-122	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1396135-2								
Nitrogen, Ammonia	103		-		90-110	-		20

Matrix Spike Analysis
Batch Quality Control

Project Name: AA
Project Number: 201.05012.002

Lab Number: L2031016
Report Date: 09/08/20

Parameter	Native Sample	MS Added	MS Found	MS %Recovery	Qual	MSD Found	MSD %Recovery	Qual	Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1394865-4 QC Sample: L2031016-06 Client ID: FB02-G1-SUR												
Phosphorus, Orthophosphate	0.008	0.5	0.485	95		-	-		80-120	-		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1394887-4 QC Sample: L2031016-01 Client ID: FB01-G1-193												
Nitrogen, Nitrate/Nitrite	0.073J	4	4.1	102		-	-		80-120	-		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1396132-4 QC Sample: L2031016-01 Client ID: FB01-G1-193												
Nitrogen, Total Kjeldahl	0.268J	8	6.60	82		-	-		77-111	-		24
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1396135-4 QC Sample: L2031016-02 Client ID: FB01-G1-100												
Nitrogen, Ammonia	0.118	4	3.66	**88**	Q	-	-		90-110	-		20

Lab Duplicate Analysis

Batch Quality Control

Project Name: AA
Project Number: 201.05012.002

Lab Number: L2031016
Report Date: 09/08/20

Parameter	Native Sample	Duplicate Sample	Units	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1394865-3 QC Sample: L2031016-06 Client ID: FB02-G1-SUR						
Phosphorus, Orthophosphate	0.008	0.008	mg/l	0		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1394887-3 QC Sample: L2031016-01 Client ID: FB01-G1-193						
Nitrogen, Nitrate/Nitrite	0.073J	0.077J	mg/l	NC		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1396132-3 QC Sample: L2031016-01 Client ID: FB01-G1-193						
Nitrogen, Total Kjeldahl	0.268J	0.258J	mg/l	NC		24
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1396135-3 QC Sample: L2031016-02 Client ID: FB01-G1-100						
Nitrogen, Ammonia	0.118	0.112	mg/l	5		20

Sample Receipt and Container Information

Were project specific reporting limits specified? YES

Cooler Information

Cooler	*Custody Seal*
A	Present/Intact
B	Present/Intact

Container Information

Container ID	*Container Type*	*Cooler*	*Initial pH*	*Final pH*	*Temp deg C*	*Pres*	*Seal*	*Frozen Date/Time*	*Analysis(*)*
L2031016-01A	Plastic 250ml unpreserved	A	7	7	4.9	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-01B	Plastic 500ml unpreserved	A	7	7	4.9	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-01C	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-01D	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-01E	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		NH3-350(28)
L2031016-01F	Plastic 950ml unpreserved	A	7	7	4.9	Y	Present/Intact		TSS-2540(7)
L2031016-02A	Plastic 250ml unpreserved	A	7	7	4.9	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-02B	Plastic 500ml unpreserved	A	7	7	4.9	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-02C	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-02D	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-02E	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		NH3-350(28)
L2031016-02F	Plastic 950ml unpreserved	A	7	7	4.9	Y	Present/Intact		TSS-2540(7)
L2031016-03A	Plastic 250ml unpreserved	A	7	7	4.9	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-03B	Plastic 500ml unpreserved	A	7	7	4.9	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-03C	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-03D	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-03E	Plastic 500ml H2SO4 preserved	A	<2	<2	4.9	Y	Present/Intact		NH3-350(28)
L2031016-03F	Plastic 950ml unpreserved	A	7	7	4.9	Y	Present/Intact		TSS-2540(7)
L2031016-04A	Plastic 250ml unpreserved	B	7	7	3.1	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-04B	Plastic 500ml unpreserved	B	7	7	3.1	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-04C	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-04D	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)

Container Information

Container ID	Container Type	Cooler	Initial pH	Final pH	Temp deg C	Pres	Seal	Frozen Date/Time	Analysis(*)
L2031016-04E	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		NH3-350(28)
L2031016-04F	Plastic 950ml unpreserved	B	7	7	3.1	Y	Present/Intact		TSS-2540(7)
L2031016-05A	Plastic 250ml unpreserved	B	7	7	3.1	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-05B	Plastic 500ml unpreserved	B	7	7	3.1	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-05C	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-05D	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-05E	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		NH3-350(28)
L2031016-05F	Plastic 950ml unpreserved	B	7	7	3.1	Y	Present/Intact		TSS-2540(7)
L2031016-06A	Plastic 250ml unpreserved	B	7	7	3.1	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-06B	Plastic 500ml unpreserved	B	7	7	3.1	Y	Present/Intact		OPHOS-4500(2),NO3/NO2-353(28)
L2031016-06C	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-06D	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		TKN-351(28),TPHOS-4500(28)
L2031016-06E	Plastic 500ml H2SO4 preserved	B	<2	<2	3.1	Y	Present/Intact		NH3-350(28)
L2031016-06F	Plastic 950ml unpreserved	B	7	7	3.1	Y	Present/Intact		TSS-2540(7)

*Values in parentheses indicate holding time in days

GLOSSARY

Acronyms

DL	- Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the limit of quantitation (LOQ). The DL includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
EDL	- Estimated Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The EDL includes any adjustments from dilutions, concentrations or moisture content, where applicable. The use of EDLs is specific to the analysis of PAHs using Solid-Phase Microextraction (SPME).
EMPC	- Estimated Maximum Possible Concentration: The concentration that results from the signal present at the retention time of an analyte when the ions meet all of the identification criteria except the ion abundance ratio criteria. An EMPC is a worst-case estimate of the concentration.
EPA	- Environmental Protection Agency.
LCS	- Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LCSD	- Laboratory Control Sample Duplicate: Refer to LCS.
LFB	- Laboratory Fortified Blank: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LOD	- Limit of Detection: This value represents the level to which a target analyte can reliably be detected for a specific analyte in a specific matrix by a specific method. The LOD includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
LOQ	- Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.) Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
MDL	- Method Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The MDL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
MS	- Matrix Spike Sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. For Method 332.0, the spike recovery is calculated using the native concentration, including estimated values.
MSD	- Matrix Spike Sample Duplicate: Refer to MS.
NA	- Not Applicable.
NC	- Not Calculated: Term is utilized when one or more of the results utilized in the calculation are non-detect at the parameter's reporting unit.
NDPA/DPA	- N-Nitrosodiphenylamine/Diphenylamine.
NI	- Not Ignitable.
NP	- Non-Plastic: Term is utilized for the analysis of Atterberg Limits in soil.
RL	- Reporting Limit: The value at which an instrument can accurately measure an analyte at a specific concentration. The RL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
RPD	- Relative Percent Difference: The results from matrix and/or matrix spike duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). Values which are less than five times the reporting limit for any individual parameter are evaluated by utilizing the absolute difference between the values; although the RPD value will be provided in the report.
SRM	- Standard Reference Material: A reference sample of a known or certified value that is of the same or similar matrix as the associated field samples.
STLP	- Semi-dynamic Tank Leaching Procedure per EPA Method 1315.
TEF	- Toxic Equivalency Factors: The values assigned to each dioxin and furan to evaluate their toxicity relative to 2,3,7,8-TCDD.
TEQ	- Toxic Equivalent: The measure of a sample's toxicity derived by multiplying each dioxin and furan by its corresponding TEF and then summing the resulting values.
TIC	- Tentatively Identified Compound: A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations.

Footnotes

Report Format: DU Report with 'J' Qualifiers

1 - The reference for this analyte should be considered modified since this analyte is absent from the target analyte list of the original method.

Terms

Analytical Method: Both the document from which the method originates and the analytical reference method. (Example: EPA 8260B is shown as 1,8260B.) The codes for the reference method documents are provided in the References section of the Addendum.

Difference: With respect to Total Oxidizable Precursor (TOP) Assay analysis, the difference is defined as the Post-Treatment value minus the Pre-Treatment value.

Final pH: As it pertains to Sample Receipt & Container Information section of the report, Final pH reflects pH of container determined after adjustment at the laboratory, if applicable. If no adjustment required, value reflects Initial pH.

Frozen Date/Time: With respect to Volatile Organics in soil, Frozen Date/Time reflects the date/time at which associated Reagent Water-preserved vials were initially frozen. Note: If frozen date/time is beyond 48 hours from sample collection, value will be reflected in 'bold'.

Initial pH: As it pertains to Sample Receipt & Container Information section of the report, Initial pH reflects pH of container determined upon receipt, if applicable.

PAH Total: With respect to Alkylated PAH analyses, the 'PAHs, Total' result is defined as the summation of results for all or a subset of the following compounds: Naphthalene, C1-C4 Naphthalenes, 2-Methylnaphthalene, 1-Methylnaphthalene, Biphenyl, Acenaphthylene, Acenaphthene, Fluorene, C1-C3 Fluorenes, Phenanthrene, C1-C4 Phenanthrenes/Anthracenes, Anthracene, Fluoranthene, Pyrene, C1-C4 Fluoranthenes/Pyrenes, Benz(a)anthracene, Chrysene, C1-C4 Chrysenes, Benzo(b)fluoranthene, Benzo(j)+(k)fluoranthene, Benzo(e)pyrene, Benzo(a)pyrene, Perylene, Indeno(1,2,3-cd)pyrene, Dibenz(ah)+(ac)anthracene, Benzo(g,h,i)perylene. If a 'Total' result is requested, the results of its individual components will also be reported.

PFAS Total: With respect to PFAS analyses, the 'PFAS, Total (5)' result is defined as the summation of results for: PFHpA, PFHxS, PFOA, PFNA and PFOS. If a 'Total' result is requested, the results of its individual components will also be reported.

The target compound Chlordane (CAS No. 57-74-9) is reported for GC ECD analyses. Per EPA,this compound "refers to a mixture of chlordane isomers, other chlorinated hydrocarbons and numerous other components." (Reference: USEPA Toxicological Review of Chlordane, In Support of Summary Information on the Integrated Risk Information System (IRIS), December 1997.)

Total: With respect to Organic analyses, a 'Total' result is defined as the summation of results for individual isomers or Aroclors. If a 'Total' result is requested, the results of its individual components will also be reported. This is applicable to 'Total' results for methods 8260, 8081 and 8082.

Data Qualifiers

A - Spectra identified as "Aldol Condensates" are byproducts of the extraction/concentration procedures when acetone is introduced in the process.

B - The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For DOD-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank AND the analyte was detected above one-half the reporting limit (or above the reporting limit for common lab contaminants) in the associated method blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone).

C - Co-elution: The target analyte co-elutes with a known lab standard (i.e. surrogate, internal standards, etc.) for co-extracted analyses.

D - Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.

E - Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.

F - The ratio of quantifier ion response to qualifier ion response falls outside of the laboratory criteria. Results are considered to be an estimated maximum concentration.

G - The concentration may be biased high due to matrix interferences (i.e, co-elution) with non-target compound(s). The result should be considered estimated.

H - The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.

I - The lower value for the two columns has been reported due to obvious interference.

J - Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (MDL) or Estimated Detection Limit (EDL) for SPME-related analyses. This represents an estimated concentration for Tentatively Identified Compounds (TICs).

M - Reporting Limit (RL) exceeds the MCP CAM Reporting Limit for this analyte.

ND - Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for SPME-related analyses.

NJ - Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.

Report Format: DU Report with 'J' Qualifiers

Data Qualifiers

P	- The RPD between the results for the two columns exceeds the method-specified criteria.
Q	- The quality control sample exceeds the associated acceptance criteria. For DOD-related projects, LCS and/or Continuing Calibration Standard exceedences are also qualified on all associated sample results. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)
R	- Analytical results are from sample re-analysis.
RE	- Analytical results are from sample re-extraction.
S	- Analytical results are from modified screening analysis.

REFERENCES

4 Methods for Chemical Analysis of Water and Wastes. EPA 600/4-79-020. Revised March 1983.

44 Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

121 Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

LIMITATION OF LIABILITIES

Alpha Analytical performs services with reasonable care and diligence normal to the analytical testing laboratory industry. In the event of an error, the sole and exclusive responsibility of Alpha Analytical shall be to re-perform the work at it's own expense. In no event shall Alpha Analytical be held liable for any incidental, consequential or special damages, including but not limited to, damages in any way connected with the use of, interpretation of, information or analysis provided by Alpha Analytical.

We strongly urge our clients to comply with EPA protocol regarding sample volume, preservation, cooling, containers, sampling procedures, holding time and splitting of samples in the field.

Alpha Analytical, Inc. ID No.:**17873**
Facility: **Company-wide** Revision 17
Department: **Quality Assurance** Published Date: 4/28/2020 9:42:21 AM
Title: **Certificate/Approval Program Summary** Page 1 of 1

Certification Information

The following analytes are not included in our Primary NELAP Scope of Accreditation:

Westborough Facility
EPA 624/624.1: m/p-xylene, o-xylene, Naphthalene
EPA 8260C: NPW: 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene, Azobenzene; SCM: Iodomethane (methyl iodide), 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene.
EPA 8270D: NPW: Dimethylnaphthalene,1,4-Diphenylhydrazine; SCM: Dimethylnaphthalene,1,4-Diphenylhydrazine.
SM4500: NPW: Amenable Cyanide; SCM: Total Phosphorus, TKN, NO2, NO3.

Mansfield Facility
SM 2540D: TSS
EPA 8082A: NPW: PCB: 1, 5, 31, 87,101, 110, 141, 151, 153, 180, 183, 187.
EPA TO-15: Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 1-Methylnaphthalene.
EPA TO-12 Non-methane organics
EPA 3C Fixed gases
Biological Tissue Matrix: EPA 3050B

The following analytes are included in our Massachusetts DEP Scope of Accreditation

Westborough Facility:

Drinking Water
EPA 300.0: Chloride, Nitrate-N, Fluoride, Sulfate; **EPA 353.2**: Nitrate-N, Nitrite-N; **SM4500NO3-F**: Nitrate-N, Nitrite-N; **SM4500F-C, SM4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, SM2320B, SM2540C, SM4500H-B, SM4500NO2-B**
EPA 332: Perchlorate; **EPA 524.2:** THMs and VOCs; **EPA 504.1:** EDB, DBCP.
Microbiology: **SM9215B; SM9223-P/A, SM9223B-Colilert-QT,SM9222D**.

Non-Potable Water
SM4500H,B, EPA 120.1, SM2510B, SM2540C, SM2320B, SM4500CL-E, SM4500F-BC, SM4500NH3-BH: Ammonia-N and Kjeldahl-N, **EPA 350.1**: Ammonia-N, **LACHAT 10-107-06-1-B**: Ammonia-N, **EPA 351.1, SM4500NO3-F, EPA 353.2**: Nitrate-N**, SM4500P-E, SM4500P-B, E, SM4500SO4-E, SM5220D, EPA 410.4, SM5210B, SM5310C, SM4500CL-D, EPA 1664, EPA 420.1, SM4500-CN-CE, SM2540D, EPA 300: Chloride, Sulfate, Nitrate.**
EPA 624.1: Volatile Halocarbons & Aromatics,
EPA 608.3: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs
EPA 625.1: SVOC (Acid/Base/Neutral Extractables), **EPA 600/4-81-045**: PCB-Oil.
Microbiology: **SM9223B-Colilert-QT; Enterolert-QT, SM9221E, EPA 1600, EPA 1603.**

Mansfield Facility:

Drinking Water
EPA 200.7: Al, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Na, Ag, Ca, Zn. **EPA 200.8:** Al, Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Ni, Se, Ag, TL, Zn. **EPA 245.1** Hg.
EPA 522.

Non-Potable Water
EPA 200.7: Al, Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Sr, TL, Ti, V, Zn.
EPA 200.8: Al, Sb, As, Be, Cd, Cr, Cu, Fe, Pb, Mn, Ni, K, Se, Ag, Na, TL, Zn.
EPA 245.1 Hg.
SM2340B

For a complete listing of analytes and methods, please contact your Alpha Project Manager.

ALPHA ANALYTICAL

CHAIN OF CUSTODY

PAGE 1 OF 1

Date Rec'd in Lab: 7/22/20

ALPHA Job #: L2031016

8 Walkup Drive
Westboro, MA 01581
Tel: 508-898-9220

320 Forbes Blvd
Mansfield, MA 02048
Tel: 508-822-9300

Client Information

Client: Ransom Consulting LLC

Address: 112 Corporate Dr
Portsmouth, NH 03801

Phone: 603-436-1490

Email: elizabeth.ransom@ransomenv.com
cc: drew.fuchs@ransomenv.com

Project Information

Project Name: AA

Project Location: Maine

Project #: 201.05012.002

Project Manager: Elizabeth Ransom

ALPHA Quote #:

Turn-Around Time

☒ Standard ☐ RUSH (only confirmed if pre-approved)

Date Due:

Report Information - Data Deliverables

☒ ADEx ☒ EMAIL

Billing Information

☐ Same as Client info PO #:

Regulatory Requirements & Project Information Requirements

☐ Yes ☒ No MA MCP Analytical Methods ☐ Yes ☐ No CT RCP Analytical Methods

☐ Yes ☒ No Matrix Spike Required on this SDG? (Required for MCP Inorganics)

☐ Yes ☒ No GW1 Standards (Info Required for Metals & EPH with Targets)

☐ Yes ☒ No NPDES RGP

☒ Other State /Fed Program MEDEP Criteria ms

Additional Project Information:

Nitrate + Nitrite, Ammonia, and Orthophosphate were field filtered. BOD analysis is not needed.

SAMPLE INFO
Filtration
☐ Field
☐ Lab to do
Preservation
☐ Lab to do

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler Initials	VOC: ☐ 8260 ☐ 624 ☐ 524.2	SVOC: ☐ ABN ☐ PAH	METALS: ☐ MCP 13 ☐ MCP 14 ☐ RCP 15	METALS: ☐ RCRA5 ☐ RCRA8 ☐ PP13	EPH: ☐ Ranges & Targets ☐ Ranges Only	VPH: ☐ Ranges & Targets ☐ Ranges Only	☐ PCB ☐ PEST	~~TPH: ☐ Quant Only ☐ Fingerprint~~ TSS	Nitrate + Nitrite as N	Ammonia as N	Total Kjeldahl Nitrogen	Total Nitrogen Mixed Forms	Orthophosphate as P	Total Phosphorus	Sample Comments	TOTAL # BOTTLES
31016-01	FB01-G1-193	7-22-20	9:45	Seawater	DAF								X	X	X	X	X	X	X		6
-02	FB01-G1-100	↓	10:15	↓	↓								X	X	X	X	X	X	X		6
-03	FB01-G1-1	↓	10:40	↓	↓								X	X	X	X	X	X	X		6
-04	FB02-G1-BOT	↓	13:45	↓	↓								X	X	X	X	X	X	X		6
-05	FB02-G1-MID	↓	14:05	↓	↓								X	X	X	X	X	X	X		6
-06	FB02-G1-SUR	↓	14:25	↓	↓								X	X	X	X	X	X	X		6
	Container Type												P	P	P	P	P	P	P		
	Preservative												A	A	D	D	D	A	D		

Container Type
P= Plastic
A= Amber glass
V= Vial
G= Glass
B= Bacteria cup
C= Cube
O= Other
E= Encore
D= BOD Bottle

Preservative
A= None
B= HCl
C= HNO_3
D= H_2SO_4
E= NaOH
F= MeOH
G= $NaHSO_4$
H = $Na_2S_2O_3$
I= Ascorbic Acid
J = NH_4Cl
K= Zn Acetate
O= Other

Relinquished By:	Date/Time	Received By:	Date/Time
	7-22-20/17:25	AAL	7/22/2020 1725
AAL	7/22/2020 1843		7/22/2020 1843
			7/22/20 23:58

All samples submitted are subject to Alpha's Terms and Conditions. See reverse side.

FORM NO: 01-01 (rev. 12-Mar-2012)

UNISHIP
COURIER SERVICES LLC

10 Iron Road
Hermon, Maine 04401

Bill Shipping Charge to

Shipper ☐ Recipient ☐
☐ Next Day ☐ Same Day

7/22/20

073237

Special ____________

390 US Route One, #3
Falmouth, Maine 04105

Phone 207•848•7546 ■ Toll-Free 800•427•7547 ■ Fax 207•561•2467

FROM:
Shipper Alpha
Street 75 Cedar St
Origin Brewer ME Zip Code
Phone #

TO:
Recipient Alpha
Street 8 [illegible] Drive
Destination Waldoboro ME Zip Code
Phone #

No. Pieces	Weight Each	Description of Items	Total Weight (Subject to Correction)	Oversize Charge	Shipping Charges
2		[illegible]			
2	TOTAL PIECES	WEIGHT GRAND TOTAL		TOTAL CHARGES	

Shipper authorizes Uniship to deliver this shipment without obtaining a delivery signature.
Shipper's Signature

Please use complete ship to address.
Uniship can not deliver to P.O. Boxes.

RECEIVED, subject to the classifications and tariffs in effect on the date of the issue of this Bill of Lading, the property described above in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned, and destined as indicated above which said carrier (the word carrier being understood throughout this contract as meaning any person or corporation in possession of the property, under the contract) agrees to carry to its usual place of delivery at said destination, if on its route, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed as to each carrier of all or any of said property overall or any portion of said route to destination and as to each party at any time interested in all or any of said property, that every service to be performed hereunder shall be subject to all the bill of lading terms and conditions in the governing classification on the date of shipment.
Shipper hereby certifies that he is familiar with all the bill of lading terms and conditions in the governing classification and the said terms and conditions are hereby agreed to by the shipper and accepted for himself and his assigns.

SHIPPER
PICK-UP TIME 1847
COURIER SIGNATURE CW
DATE 7-22-2020

RECIPIENT
DELIVERY TIME 23:58
COURIER SIGNATURE
DATE 7-23-20

RECIPIENT COPY

SCI SPEC

PRECLEANED CONTAINERS

Since 1963

Custody Seal Custody Seal Custody Seal Custody Seal

DATE [illegible]

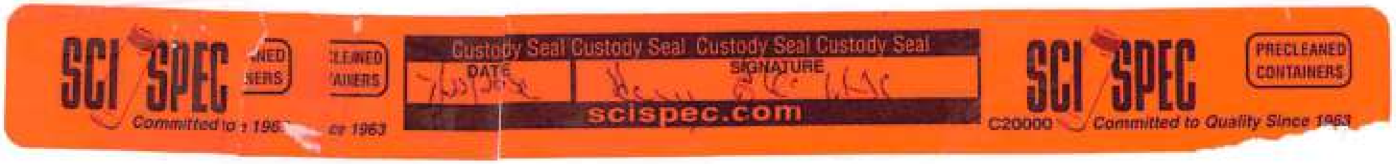
SIGNATURE

scispec.com

SCI SPEC

PRECLEANED CONTAINERS

C20000 Committed to Quality Since 1963



SCI SPEC

Committed to

NED

NERS

LEANED

AINERS

ce 1963

Custody Seal Custody Seal Custody Seal Custody Seal

DATE [illegible]

SIGNATURE

scispec.com

SCI SPEC

PRECLEANED CONTAINERS

C20000 Committed to Quality Since 1963

ANALYTICAL REPORT

Lab Number:	L2034261
Client:	Ransom Consulting, Inc. 112 Corporate Drive Pease International Tradeport Portsmouth, NH 03801
ATTN:	Elizabeth Ransom
Phone:	(603) 436-1490
Project Name:	AAF
Project Number:	201.05012.002
Report Date:	08/27/20

Certifications & Approvals: MA (M-MA086), NH NELAP (2064), CT (PH-0574), IL (200077), ME (MA00086), MD (348), NJ (MA935), NY (11148), NC (25700/666), PA (68-03671), RI (LAO00065), TX (T104704476), VT (VT-0935), VA (460195), USDA (Permit #P330-17-00196).

Eight Walkup Drive, Westborough, MA 01581-1019
508-898-9220 (Fax) 508-898-9193 800-624-9220 - www.alphalab.com

Project Name: AAF
Project Number: 201.05012.002

Lab Number: L2034261
Report Date: 08/27/20

Alpha Sample ID	Client ID	Matrix	Sample Location	Collection Date/Time	Receive Date
L2034261-01	FB01-G1-BOT	WATER	MAINE	08/20/20 11:10	08/21/20
L2034261-02	FB01-G1-MID	WATER	MAINE	08/20/20 11:35	08/21/20
L2034261-03	FB01-G1-SUR	WATER	MAINE	08/20/20 11:55	08/21/20
L2034261-04	FB02-G1-BOT	WATER	MAINE	08/20/20 08:00	08/21/20
L2034261-05	FB02-G1-MID	WATER	MAINE	08/20/20 08:25	08/21/20
L2034261-06	FB02-G1-SUR	WATER	MAINE	08/20/20 08:40	08/21/20

Case Narrative

The samples were received in accordance with the Chain of Custody and no significant deviations were encountered during the preparation or analysis unless otherwise noted. Sample Receipt, Container Information, and the Chain of Custody are located at the back of the report.

Results contained within this report relate only to the samples submitted under this Alpha Lab Number and meet NELAP requirements for all NELAP accredited parameters unless otherwise noted in the following narrative. The data presented in this report is organized by parameter (i.e. VOC, SVOC, etc.). Sample specific Quality Control data (i.e. Surrogate Spike Recovery) is reported at the end of the target analyte list for each individual sample, followed by the Laboratory Batch Quality Control at the end of each parameter. Tentatively Identified Compounds (TICs), if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported. If a sample was re-analyzed or re-extracted due to a required quality control corrective action and if both sets of data are reported, the Laboratory ID of the re-analysis or re-extraction is designated with an "R" or "RE", respectively.

When multiple Batch Quality Control elements are reported (e.g. more than one LCS), the associated samples for each element are noted in the grey shaded header line of each data table. Any Laboratory Batch, Sample Specific % recovery or RPD value that is outside the listed Acceptance Criteria is bolded in the report. In reference to questions H (CAM) or 4 (RCP) when "NO" is checked, the performance criteria for CAM and RCP methods allow for some quality control failures to occur and still be within method compliance. In these instances, the specific failure is not narrated but noted in the associated QC Outlier Summary Report, located directly after the Case Narrative. QC information is also incorporated in the Data Usability Assessment table (Format 11) of our Data Merger tool, where it can be reviewed in conjunction with the sample result, associated regulatory criteria and any associated data usability implications.

Soil/sediments, solids and tissues are reported on a dry weight basis unless otherwise noted. Definitions of all data qualifiers and acronyms used in this report are provided in the Glossary located at the back of the report.

HOLD POLICY - For samples submitted on hold, Alpha's policy is to hold samples (with the exception of Air canisters) free of charge for 21 calendar days from the date the project is completed. After 21 calendar days, we will dispose of all samples submitted including those put on hold unless you have contacted your Alpha Project Manager and made arrangements for Alpha to continue to hold the samples. Air canisters will be disposed after 3 business days from the date the project is completed.

Please contact Project Management at 800-624-9220 with any questions.

Case Narrative (continued)

Report Submission

All non-detect (ND) or estimated concentrations (J-qualified) have been quantitated to the limit noted in the MDL column.

Sample Receipt

The analyses performed were specified by the client.

I, the undersigned, attest under the pains and penalties of perjury that, to the best of my knowledge and belief and based upon my personal inquiry of those responsible for providing the information contained in this analytical report, such information is accurate and complete. This certificate of analysis is not complete unless this page accompanies any and all pages of this report.

Authorized Signature: Michelle M. Morris Michelle M. Morris

Title: Technical Director/Representative Date: 08/27/20

INORGANICS
&
MISCELLANEOUS

SAMPLE RESULTS

Lab ID: L2034261-01
Client ID: FB01-G1-BOT
Sample Location: MAINE

Date Collected: 08/20/20 11:10
Date Received: 08/21/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	20.		mg/l	5.0	NA	1	-	08/23/20 12:14	121,2540D	AA
Nitrogen, Ammonia	0.080		mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:55	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.090	J	mg/l	0.10	0.023	1	-	08/22/20 08:11	44,353.2	MR
Nitrogen, Total Kjeldahl	0.261	J	mg/l	0.300	0.066	1	08/24/20 13:30	08/24/20 19:26	4,351.3/.1 (M)	AT
Phosphorus, Total	0.028		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:31	121,4500P-E	SD
Phosphorus, Orthophosphate	0.022		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW

SAMPLE RESULTS

Lab ID:	L2034261-02	Date Collected:	08/20/20 11:35
Client ID:	FB01-G1-MID	Date Received:	08/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	18.		mg/l	5.0	NA	1	-	08/23/20 12:14	121,2540D	AA
Nitrogen, Ammonia	0.078		mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:55	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.11		mg/l	0.10	0.023	1	-	08/22/20 08:12	44,353.2	MR
Nitrogen, Total Kjeldahl	0.294	J	mg/l	0.300	0.066	1	08/24/20 13:30	08/24/20 19:27	4,351.3/.1 (M)	AT
Phosphorus, Total	0.029		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:34	121,4500P-E	SD
Phosphorus, Orthophosphate	0.019		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW

Project Name: AAF
Project Number: 201.05012.002

Lab Number: L2034261
Report Date: 08/27/20

SAMPLE RESULTS

Lab ID: L2034261-03
Client ID: FB01-G1-SUR
Sample Location: MAINE

Date Collected: 08/20/20 11:55
Date Received: 08/21/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	28.		mg/l	5.0	NA	1	-	08/23/20 13:19	121,2540D	AA
Nitrogen, Ammonia	ND		mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:56	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.033	J	mg/l	0.10	0.023	1	-	08/22/20 08:14	44,353.2	MR
Nitrogen, Total Kjeldahl	0.172	J	mg/l	0.300	0.066	1	08/24/20 13:30	08/24/20 19:28	4,351.3/.1 (M)	AT
Phosphorus, Total	0.017		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:37	121,4500P-E	SD
Phosphorus, Orthophosphate	0.006		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW

SAMPLE RESULTS

Lab ID: L2034261-04
Client ID: FB02-G1-BOT
Sample Location: MAINE
Date Collected: 08/20/20 08:00
Date Received: 08/21/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	18.		mg/l	5.0	NA	1	-	08/23/20 13:19	121,2540D	AA
Nitrogen, Ammonia	0.105		mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:57	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.10		mg/l	0.10	0.023	1	-	08/22/20 08:15	44,353.2	MR
Nitrogen, Total Kjeldahl	0.197	J	mg/l	0.300	0.066	1	08/24/20 13:30	08/24/20 19:29	4,351.3/.1 (M)	AT
Phosphorus, Total	0.042		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:38	121,4500P-E	SD
Phosphorus, Orthophosphate	0.033		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW

Project Name:	AAF	**Lab Number:**	L2034261
Project Number:	201.05012.002	**Report Date:**	08/27/20

SAMPLE RESULTS

Lab ID:	L2034261-05	Date Collected:	08/20/20 08:25
Client ID:	FB02-G1-MID	Date Received:	08/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	25.		mg/l	5.0	NA	1	-	08/23/20 13:19	121,2540D	AA
Nitrogen, Ammonia	0.160		mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:58	44,350.1	AT
Nitrogen, Nitrate/Nitrite	0.083	J	mg/l	0.10	0.023	1	-	08/22/20 08:16	44,353.2	MR
Nitrogen, Total Kjeldahl	0.268	J	mg/l	0.300	0.066	1	08/24/20 13:30	08/24/20 19:30	4,351.3/.1 (M)	AT
Phosphorus, Total	0.028		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:40	121,4500P-E	SD
Phosphorus, Orthophosphate	0.025		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW

Project Name:	AAF	**Lab Number:**	L2034261
Project Number:	201.05012.002	**Report Date:**	08/27/20

SAMPLE RESULTS

Lab ID:	L2034261-06	Date Collected:	08/20/20 08:40
Client ID:	FB02-G1-SUR	Date Received:	08/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	28.		mg/l	5.0	NA	1	-	08/23/20 13:19	121,2540D	AA
Nitrogen, Ammonia	0.043	J	mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:59	44,350.1	AT
Nitrogen, Nitrate/Nitrite	ND		mg/l	0.10	0.023	1	-	08/22/20 08:17	44,353.2	MR
Nitrogen, Total Kjeldahl	0.253	J	mg/l	0.300	0.066	1	08/24/20 13:30	08/24/20 19:31	4,351.3/.1 (M)	AT
Phosphorus, Total	0.017		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:41	121,4500P-E	SD
Phosphorus, Orthophosphate	0.009		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW

Project Name: AAF
Lab Number: L2034261
Project Number: 201.05012.002
Report Date: 08/27/20

Method Blank Analysis
Batch Quality Control

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1401593-1										
Phosphorus, Orthophosphate	ND		mg/l	0.005	0.001	1	-	08/21/20 14:55	121,4500P-E	JW
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1401744-1										
Nitrogen, Nitrate/Nitrite	ND		mg/l	0.10	0.023	1	-	08/22/20 06:56	44,353.2	MR
General Chemistry - Westborough Lab for sample(s): 01-02 Batch: WG1401918-1										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	08/23/20 12:14	121,2540D	AA
General Chemistry - Westborough Lab for sample(s): 03-06 Batch: WG1401938-1										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	08/23/20 13:19	121,2540D	AA
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1402081-1										
Phosphorus, Total	ND		mg/l	0.010	0.004	1	08/24/20 10:30	08/25/20 09:27	121,4500P-E	SD
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1402124-1										
Nitrogen, Ammonia	0.027	J	mg/l	0.075	0.024	1	08/24/20 13:15	08/24/20 19:42	44,350.1	AT
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1402129-1										
Nitrogen, Total Kjeldahl	ND		mg/l	0.300	0.022	1	08/24/20 13:30	08/24/20 19:04	4,351.3/.1 (M)	AT

Lab Control Sample Analysis
Batch Quality Control

Project Name: AAF
Project Number: 201.05012.002

Lab Number: L2034261
Report Date: 08/27/20

Parameter	LCS %Recovery	Qual	LCSD %Recovery	Qual	%Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1401593-2								
Phosphorus, Orthophosphate	99		-		90-110	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1401744-2								
Nitrogen, Nitrate/Nitrite	100		-		90-110	-		
General Chemistry - Westborough Lab Associated sample(s): 01-02 Batch: WG1401918-2								
Solids, Total Suspended	115		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 03-06 Batch: WG1401938-2								
Solids, Total Suspended	115		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1402081-2								
Phosphorus, Total	104		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1402124-2								
Nitrogen, Ammonia	98		-		90-110	-		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1402129-2								
Nitrogen, Total Kjeldahl	98		-		78-122	-		

Matrix Spike Analysis
Batch Quality Control

Project Name: AAF

Project Number: 201.05012.002

Lab Number: L2034261

Report Date: 08/27/20

Parameter	Native Sample	MS Added	MS Found	MS %Recovery	Qual	MSD Found	MSD %Recovery	Qual	Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1401593-4 QC Sample: L2034261-02 Client ID: FB01-G1-MID												
Phosphorus, Orthophosphate	0.019	0.5	0.525	101		-	-		80-120	-		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1402081-3 QC Sample: L2034261-01 Client ID: FB01-G1-BOT												
Phosphorus, Total	0.028	0.5	0.547	104		-	-		75-125	-		20

Lab Duplicate Analysis

Batch Quality Control

Project Name: AAF

Project Number: 201.05012.002

Lab Number: L2034261

Report Date: 08/27/20

Parameter	Native Sample	Duplicate Sample	Units	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1401593-3 QC Sample: L2034261-01 Client ID: FB01-G1-BOT						
Phosphorus, Orthophosphate	0.022	0.022	mg/l	0		20
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1402081-4 QC Sample: L2034261-01 Client ID: FB01-G1-BOT						
Phosphorus, Total	0.028	0.027	mg/l	4		20

Sample Receipt and Container Information

Were project specific reporting limits specified? YES

Cooler Information

Cooler	*Custody Seal*
A	Absent

Container Information

Container ID	*Container Type*	*Cooler*	*Initial pH*	*Final pH*	*Temp deg C*	*Pres*	*Seal*	*Frozen Date/Time*	*Analysis(*)*
L2034261-01A	Plastic 250ml unpreserved	A	7	7	4.7	Y	Absent		OPHOS-4500(2)
L2034261-01B	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-01C	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-01D	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-01E	Plastic 950ml unpreserved	A	7	7	4.7	Y	Absent		TSS-2540(7)
L2034261-02A	Plastic 250ml unpreserved	A	7	7	4.7	Y	Absent		OPHOS-4500(2)
L2034261-02B	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-02C	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-02D	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-02E	Plastic 950ml unpreserved	A	7	7	4.7	Y	Absent		TSS-2540(7)
L2034261-03A	Plastic 250ml unpreserved	A	7	7	4.7	Y	Absent		OPHOS-4500(2)
L2034261-03B	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-03C	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-03D	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-03E	Plastic 950ml unpreserved	A	7	7	4.7	Y	Absent		TSS-2540(7)
L2034261-04A	Plastic 250ml unpreserved	A	7	7	4.7	Y	Absent		OPHOS-4500(2)
L2034261-04B	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-04C	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)
L2034261-04D	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		TKN-351(28),NO3/NO2-353(28),TPHOS-4500(28),NH3-350(28)

*Values in parentheses indicate holding time in days

Container Information

Container ID	Container Type	Cooler	Initial pH	Final pH	Temp deg C	Pres	Seal	Frozen Date/Time	Analysis(*)
L2034261-04E	Plastic 950ml unpreserved	A	7	7	4.7	Y	Absent		TSS-2540(7)
L2034261-05A	Plastic 250ml unpreserved	A	7	7	4.7	Y	Absent		OPHOS-4500(2)
L2034261-05B	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-05C	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-05D	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-05E	Plastic 950ml unpreserved	A	7	7	4.7	Y	Absent		TSS-2540(7)
L2034261-06A	Plastic 250ml unpreserved	A	7	7	4.7	Y	Absent		OPHOS-4500(2)
L2034261-06B	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-06C	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-06D	Plastic 250ml H2SO4 preserved	A	<2	<2	4.7	Y	Absent		NO3/NO2-353(28),TKN-351(28),TPHOS-4500(28),NH3-350(28)
L2034261-06E	Plastic 950ml unpreserved	A	7	7	4.7	Y	Absent		TSS-2540(7)

*Values in parentheses indicate holding time in days

GLOSSARY

Acronyms

DL - Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the limit of quantitation (LOQ). The DL includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)

EDL - Estimated Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The EDL includes any adjustments from dilutions, concentrations or moisture content, where applicable. The use of EDLs is specific to the analysis of PAHs using Solid-Phase Microextraction (SPME).

EMPC - Estimated Maximum Possible Concentration: The concentration that results from the signal present at the retention time of an analyte when the ions meet all of the identification criteria except the ion abundance ratio criteria. An EMPC is a worst-case estimate of the concentration.

EPA - Environmental Protection Agency.

LCS - Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.

LCSD - Laboratory Control Sample Duplicate: Refer to LCS.

LFB - Laboratory Fortified Blank: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.

LOD - Limit of Detection: This value represents the level to which a target analyte can reliably be detected for a specific analyte in a specific matrix by a specific method. The LOD includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)

LOQ - Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)

Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)

MDL - Method Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The MDL includes any adjustments from dilutions, concentrations or moisture content, where applicable.

MS - Matrix Spike Sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. For Method 332.0, the spike recovery is calculated using the native concentration, including estimated values.

MSD - Matrix Spike Sample Duplicate: Refer to MS.

NA - Not Applicable.

NC - Not Calculated: Term is utilized when one or more of the results utilized in the calculation are non-detect at the parameter's reporting unit.

NDPA/DPA - N-Nitrosodiphenylamine/Diphenylamine.

NI - Not Ignitable.

NP - Non-Plastic: Term is utilized for the analysis of Atterberg Limits in soil.

RL - Reporting Limit: The value at which an instrument can accurately measure an analyte at a specific concentration. The RL includes any adjustments from dilutions, concentrations or moisture content, where applicable.

RPD - Relative Percent Difference: The results from matrix and/or matrix spike duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). Values which are less than five times the reporting limit for any individual parameter are evaluated by utilizing the absolute difference between the values; although the RPD value will be provided in the report.

SRM - Standard Reference Material: A reference sample of a known or certified value that is of the same or similar matrix as the associated field samples.

STLP - Semi-dynamic Tank Leaching Procedure per EPA Method 1315.

TEF - Toxic Equivalency Factors: The values assigned to each dioxin and furan to evaluate their toxicity relative to 2,3,7,8-TCDD.

TEQ - Toxic Equivalent: The measure of a sample's toxicity derived by multiplying each dioxin and furan by its corresponding TEF and then summing the resulting values.

TIC - Tentatively Identified Compound: A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations.

Footnotes

1 - The reference for this analyte should be considered modified since this analyte is absent from the target analyte list of the original method.

Terms

Analytical Method: Both the document from which the method originates and the analytical reference method. (Example: EPA 8260B is shown as 1,8260B.) The codes for the reference method documents are provided in the References section of the Addendum.

Difference: With respect to Total Oxidizable Precursor (TOP) Assay analysis, the difference is defined as the Post-Treatment value minus the Pre-Treatment value.

Final pH: As it pertains to Sample Receipt & Container Information section of the report, Final pH reflects pH of container determined after adjustment at the laboratory, if applicable. If no adjustment required, value reflects Initial pH.

Frozen Date/Time: With respect to Volatile Organics in soil, Frozen Date/Time reflects the date/time at which associated Reagent Water-preserved vials were initially frozen. Note: If frozen date/time is beyond 48 hours from sample collection, value will be reflected in 'bold'.

Initial pH: As it pertains to Sample Receipt & Container Information section of the report, Initial pH reflects pH of container determined upon receipt, if applicable.

PAH Total: With respect to Alkylated PAH analyses, the 'PAHs, Total' result is defined as the summation of results for all or a subset of the following compounds: Naphthalene, C1-C4 Naphthalenes, 2-Methylnaphthalene, 1-Methylnaphthalene, Biphenyl, Acenaphthylene, Acenaphthene, Fluorene, C1-C3 Fluorenes, Phenanthrene, C1-C4 Phenanthrenes/Anthracenes, Anthracene, Fluoranthene, Pyrene, C1-C4 Fluoranthenes/Pyrenes, Benz(a)anthracene, Chrysene, C1-C4 Chrysenes, Benzo(b)fluoranthene, Benzo(j)+(k)fluoranthene, Benzo(e)pyrene, Benzo(a)pyrene, Perylene, Indeno(1,2,3-cd)pyrene, Dibenz(ah)+(ac)anthracene, Benzo(g,h,i)perylene. If a 'Total' result is requested, the results of its individual components will also be reported.

PFAS Total: With respect to PFAS analyses, the 'PFAS, Total (5)' result is defined as the summation of results for: PFHpA, PFHxS, PFOA, PFNA and PFOS. If a 'Total' result is requested, the results of its individual components will also be reported.

The target compound Chlordane (CAS No. 57-74-9) is reported for GC ECD analyses. Per EPA,this compound "refers to a mixture of chlordane isomers, other chlorinated hydrocarbons and numerous other components." (Reference: USEPA Toxicological Review of Chlordane, In Support of Summary Information on the Integrated Risk Information System (IRIS), December 1997.)

Total: With respect to Organic analyses, a 'Total' result is defined as the summation of results for individual isomers or Aroclors. If a 'Total' result is requested, the results of its individual components will also be reported. This is applicable to 'Total' results for methods 8260, 8081 and 8082.

Data Qualifiers

A - Spectra identified as "Aldol Condensates" are byproducts of the extraction/concentration procedures when acetone is introduced in the process.

B - The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For DOD-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank AND the analyte was detected above one-half the reporting limit (or above the reporting limit for common lab contaminants) in the associated method blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone).

C - Co-elution: The target analyte co-elutes with a known lab standard (i.e. surrogate, internal standards, etc.) for co-extracted analyses.

D - Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.

E - Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.

F - The ratio of quantifier ion response to qualifier ion response falls outside of the laboratory criteria. Results are considered to be an estimated maximum concentration.

G - The concentration may be biased high due to matrix interferences (i.e, co-elution) with non-target compound(s). The result should be considered estimated.

H - The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.

I - The lower value for the two columns has been reported due to obvious interference.

J - Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (MDL) or Estimated Detection Limit (EDL) for SPME-related analyses. This represents an estimated concentration for Tentatively Identified Compounds (TICs).

M - Reporting Limit (RL) exceeds the MCP CAM Reporting Limit for this analyte.

ND - Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for SPME-related analyses.

NJ - Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.

Report Format: DU Report with 'J' Qualifiers

Data Qualifiers

P	- The RPD between the results for the two columns exceeds the method-specified criteria.
Q	- The quality control sample exceeds the associated acceptance criteria. For DOD-related projects, LCS and/or Continuing Calibration Standard exceedences are also qualified on all associated sample results. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)
R	- Analytical results are from sample re-analysis.
RE	- Analytical results are from sample re-extraction.
S	- Analytical results are from modified screening analysis.

REFERENCES

4 Methods for Chemical Analysis of Water and Wastes. EPA 600/4-79-020. Revised March 1983.

44 Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

121 Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

LIMITATION OF LIABILITIES

Alpha Analytical performs services with reasonable care and diligence normal to the analytical testing laboratory industry. In the event of an error, the sole and exclusive responsibility of Alpha Analytical shall be to re-perform the work at it's own expense. In no event shall Alpha Analytical be held liable for any incidental, consequential or special damages, including but not limited to, damages in any way connected with the use of, interpretation of, information or analysis provided by Alpha Analytical.

We strongly urge our clients to comply with EPA protocol regarding sample volume, preservation, cooling, containers, sampling procedures, holding time and splitting of samples in the field.

Certification Information

The following analytes are not included in our Primary NELAP Scope of Accreditation:

Westborough Facility
EPA 624/624.1: m/p-xylene, o-xylene, Naphthalene
EPA 8260C: NPW: 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene, Azobenzene; SCM: Iodomethane (methyl iodide), 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene.
EPA 8270D: NPW: Dimethylnaphthalene,1,4-Diphenylhydrazine; SCM: Dimethylnaphthalene,1,4-Diphenylhydrazine.
SM4500: NPW: Amenable Cyanide; SCM: Total Phosphorus, TKN, NO2, NO3.

Mansfield Facility
SM 2540D: TSS
EPA 8082A: NPW: PCB: 1, 5, 31, 87,101, 110, 141, 151, 153, 180, 183, 187.
EPA TO-15: Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 1-Methylnaphthalene.
EPA TO-12 Non-methane organics
EPA 3C Fixed gases
Biological Tissue Matrix: EPA 3050B

The following analytes are included in our Massachusetts DEP Scope of Accreditation

Westborough Facility:

Drinking Water
EPA 300.0: Chloride, Nitrate-N, Fluoride, Sulfate; **EPA 353.2**: Nitrate-N, Nitrite-N; **SM4500NO3-F**: Nitrate-N, Nitrite-N; **SM4500F-C, SM4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, SM2320B, SM2540C, SM4500H-B, SM4500NO2-B**
EPA 332: Perchlorate; **EPA 524.2:** THMs and VOCs; **EPA 504.1:** EDB, DBCP.
Microbiology: **SM9215B; SM9223-P/A, SM9223B-Colilert-QT,SM9222D**.

Non-Potable Water
SM4500H,B, EPA 120.1, SM2510B, SM2540C, SM2320B, SM4500CL-E, SM4500F-BC, SM4500NH3-BH: Ammonia-N and Kjeldahl-N, **EPA 350.1**: Ammonia-N, **LACHAT 10-107-06-1-B**: Ammonia-N, **EPA 351.1, SM4500NO3-F, EPA 353.2**: Nitrate-N**, SM4500P-E, SM4500P-B, E, SM4500SO4-E, SM5220D, EPA 410.4, SM5210B, SM5310C, SM4500CL-D, EPA 1664, EPA 420.1, SM4500-CN-CE, SM2540D, EPA 300: Chloride, Sulfate, Nitrate.**
EPA 624.1: Volatile Halocarbons & Aromatics,
EPA 608.3: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs
EPA 625.1: SVOC (Acid/Base/Neutral Extractables), **EPA 600/4-81-045**: PCB-Oil.
Microbiology: **SM9223B-Colilert-QT; Enterolert-QT, SM9221E, EPA 1600, EPA 1603.**

Mansfield Facility:

Drinking Water
EPA 200.7: Al, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Na, Ag, Ca, Zn. **EPA 200.8:** Al, Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Ni, Se, Ag, TL, Zn. **EPA 245.1** Hg.
EPA 522.

Non-Potable Water
EPA 200.7: Al, Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Sr, TL, Ti, V, Zn.
EPA 200.8: Al, Sb**,** As, Be, Cd, Cr, Cu, Fe, Pb, Mn, Ni, K, Se, Ag, Na, TL, Zn.
EPA 245.1 Hg.
SM2340B

For a complete listing of analytes and methods, please contact your Alpha Project Manager.

ALPHA ANALYTICAL

8 Walkup Drive
Westboro, MA 01581
Tel: 508-898-9220

320 Forbes Blvd
Mansfield, MA 02048
Tel: 508-822-9300

CHAIN OF CUSTODY

PAGE 1 OF 1

Date Rec'd in Lab: 8/21/20

ALPHA Job #: L2034261

Client Information

Client: Ransom Consulting LLC
Address: 112 Corporate Dr
Portsmouth, NH 03801
Phone: 603-436-1490
Email: elizabeth.ransom@ransomenv.com
cc: drew.fuchs@ransomenv.com

Project Information

Project Name: AAF
Project Location: Maine
Project #: 206.05012.003
Project Manager: Elizabeth Ransom
ALPHA Quote #:

Turn-Around Time

☒ Standard ☐ RUSH *(only confirmed if pre-approved!)*

Date Due:

Report Information - Data Deliverables

☒ ADEx ☒ EMAIL

Billing Information

☐ Same as Client info PO #:

Regulatory Requirements & Project Information Requirements

☐ Yes ☐ No MA MCP Analytical Methods
☐ Yes ☒ No CT RCP Analytical Methods
☐ Yes ☐ No Matrix Spike Required on this SDG? (Required for MCP Inorganics)
☐ Yes ☐ No GW1 Standards (Info Required for Metals & EPH with Targets)
☐ Yes ☐ No NPDES RGP
☒ Other State /Fed Program ME DEP Criteria

Additional Project Information:

Orthophosphate, ammonia, nitrate/nitrite field filtered.
Attn: Melissa Gulli – to discuss total N with Elizabeth Ransom prior to analyses.

SAMPLE INFO

Filtration
☐ Field
☐ Lab to do

Preservation
☐ Lab to do

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler Initials	VOC: ☐ 8260 ☐ 624 ☐ 524.2	SVOC: ☐ ABN ☐ PAH	METALS: ☐ MCP 13 ☐ MCP 14 ☐ RCP 15	METALS: ☐ RCRA5 ☐ RCRA8 ☐ PP13	EPH: ☐ Ranges & Targets ☐ Ranges Only	VPH: ☐ Ranges & Targets ☐ Ranges Only	☐ PCB ☐ PEST	TPH: ☐ Quant Only ☐ Fingerprint	Total Phosphorus	Orthophosphate	Total Kjeldahl Nitrogen	Ammonia Nitrate/Nitrite	Total Suspended Solids	Total Nitrogen	Sample Comments	TOTAL # BOTTLES
34261-01	FB01-G1-BOT	8-20-20	11:40	Sea water	DAF									X	X	X	X	X	X		5
-02	FB01-G1-MID		11:35											X	X	X	X	X	X		5
-03	FB01-G1-SUR		11:55											X	X	X	X	X	X		5
-04	FB02-G1-BOT		8:00											X	X	X	X	X	X		5
-05	FB02-G1-MID		8:25											X	X	X	X	X	X		5
-06	FB02-G1-SUR		8:40											X	X	X	X	X	X		5
-																					
Container Type														P	P	P	P	P	P		
Preservative														D	A	D	A/D	A	D		

Container Type
P= Plastic
A= Amber glass
V= Vial
G= Glass
B= Bacteria cup
C= Cube
O= Other
E= Encore
D= BOD Bottle

Preservative
A= None
B= HCl
C= HNO_3
D= H_2SO_4
E= NaOH
F= MeOH
G= $NaHSO_4$
H = $Na_2S_2O_3$
I= Ascorbic Acid
J = NH_4Cl
K= Zn Acetate
O= Other

Relinquished By:	Date/Time	Received By:	Date/Time
	8-20-20/22:45	NHSC Storage Locker	8-20-20/22:45
AAF Secured storage	8/21/20 7:45	AAL	8/21/20 7:45
AAL	8/21/20 8:00	Benj Stan	8/21/20 9:20

All samples submitted are subject to Alpha's Terms and Conditions.
See reverse side.

FORM NO: 01-01 (rev. 12-Mar-2012)

September 02, 2020 Service Request No:R2007726

Melissa Gulli
Alpha Analytical Inc. (Westborough, MA)
8 Walkup Drive
Westborough, MA 01581

Laboratory Results for: L2034262

Dear Melissa,

Enclosed are the results of the sample(s) submitted to our laboratory August 25, 2020
For your reference, these analyses have been assigned our service request number **R2007726.**

All testing was performed according to our laboratory's quality assurance program and met the requirements of the TNI standards except as noted in the case narrative report. Any testing not included in the lab's accreditation is identified on a Non-Certified Analytes report. All results are intended to be considered in their entirety. ALS Environmental is not responsible for use of less than the complete report. Results apply only to the individual samples submitted to the lab for analysis, as listed in the report. The measurement uncertainty of the results included in this report is within that expected when using the prescribed method(s), and represented by Laboratory Control Sample control limits. Any events, such as QC failures or Holding Time exceedances, which may add to the uncertainty are explained in the report narrative or are flagged with qualifiers. The flags are explained in the Report Qualifiers and Definitions page of this report.

Please contact me if you have any questions. My extension is 7472. You may also contact me via email at Janice.Jaeger@alsglobal.com.

Respectfully submitted,

ALS Group USA, Corp. dba ALS Environmental

Janice Jaeger
Project Manager

Narrative Documents

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

Client: Alpha Analytical Inc. (Westborough, MA)
Service Request: R2007726
Project: L2034262
Date Received: 08/25/2020
Sample Matrix: Water

CASE NARRATIVE

All analyses were performed consistent with the quality assurance program of ALS Environmental. This report contains analytical results for samples for the Tier II level requested by the client.

Sample Receipt:

Six water samples were received for analysis at ALS Environmental on 08/25/2020. Any discrepancies upon initial sample inspection are annotated on the sample receipt and preservation form included within this report. The samples were stored at minimum in accordance with the analytical method requirements.

General Chemistry:

No significant anomalies were noted with this analysis.

Approved by ______________________ Date 08/31/2020

SAMPLE DETECTION SUMMARY

CLIENT ID: FB01-G1-BOT-S **Lab ID: R2007726-001**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.026			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.069			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.17			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.11			0.10	mg/L	351.2

CLIENT ID: FB01-G1-MID-S **Lab ID: R2007726-002**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.020			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.057			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.24			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.18			0.10	mg/L	351.2

CLIENT ID: FB01-G1-SUR-S **Lab ID: R2007726-003**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.011			0.010	mg/L	350.1

CLIENT ID: FB02-G1-BOT-S **Lab ID: R2007726-004**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.042			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.076			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.28			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.20			0.10	mg/L	351.2

CLIENT ID: FB02-G1-MID-S **Lab ID: R2007726-005**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.010			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.065			0.050	mg/L	353.2

CLIENT ID: FB02-G1-SUR-S **Lab ID: R2007726-006**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.028			0.010	mg/L	350.1
Nitrogen, Total Kjeldahl (TKN)	0.16			0.10	mg/L	351.2

Sample Receipt Information

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

SAMPLE CROSS-REFERENCE

SAMPLE #	CLIENT SAMPLE ID	DATE	TIME
R2007726-001	FB01-G1-BOT-S	8/20/2020	1110
R2007726-002	FB01-G1-MID-S	8/20/2020	1135
R2007726-003	FB01-G1-SUR-S	8/20/2020	1155
R2007726-004	FB02-G1-BOT-S	8/20/2020	0800
R2007726-005	FB02-G1-MID-S	8/20/2020	0825
R2007726-006	FB02-G1-SUR-S	8/20/2020	0840

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2034262

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2034262

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com TKN-351 NH3- by 350 NO2/NO3-353

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB01-G1-BOT-S	08-20-20 11:10	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB01-G1-MID-S	08-20-20 11:35	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB01-G1-SUR-S	08-20-20 11:55	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB02-G1-BOT-S	08-20-20 08:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB02-G1-MID-S	08-20-20 08:25	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB02-G1-SUR-S	08-20-20 08:40	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	

	Relinquished By:	Date/Time:	Received By:	Date/Time:
		8/24/20		8252020 09:30
Form No: AL_subcoc				

R2007726 5
Alpha Analytical Inc. (Westborough, MA)
L0234262

ALS

Cooler Receipt and Preservation Check Form

R2007726 5
Alpha Analytical Inc. (Westborough, MA)
L0234262

Project/Client Alpha Analytical Folder Number

Cooler received on 8-25-2020 by: KE

COURIER: ALS [✓ UPS] FEDEX VELOCITY CLIENT

1	Were **Custody seals** on outside of cooler?	Y [✓ N]
2	**Custody papers** properly completed (ink, signed)?	Y [✓ N]
3	Did all bottles arrive in good **condition** (unbroken)?	[✓ Y] N
4	Circle: [✓ **Wet Ice**] **Dry Ice** **Gel packs** present?	[✓ Y] N

5a	**Perchlorate** samples have required headspace?	Y N [✓ NA]
5b	Did VOA vials, Alk,or Sulfide have sig* **bubbles**?	Y N [✓ NA]
6	Where did the bottles originate? ALS/ROC [✓ CLIENT]	
7	Soil VOA received as: Bulk Encore 5035set [✓ NA]	

8. Temperature Readings Date: 8-25-2020 Time: 10:02 ID: IR#7 [✓ IR#10] From: [✓ Temp Blank] Sample Bottle

Observed Temp (°C)	2.4						
Within 0-6°C?	[✓ Y] N	Y N	Y N	Y N	Y N	Y N	Y N
If <0°C, were samples frozen?	Y N	Y N	Y N	Y N	Y N	Y N	Y N

If out of Temperature, note packing/ice condition: ____________ Ice melted Poorly Packed (described below) Same Day Rule

&Client Approval to Run Samples: __________ Standing Approval Client aware at drop-off Client notified by: ____________

All samples held in storage location: R-002 by KE on 8-25-20 at 10:04
5035 samples placed in storage location: ______ by ______ on ______ at ______ within 48 hours of sampling? Y N

Cooler Breakdown/Preservation Check**: Date: 8/25/2020 Time: 2050 by: AW

9.	Were all bottle labels complete (*i.e.* analysis, preservation, etc.)?	[✓ YES]	NO	
10.	Did all bottle labels and tags agree with custody papers?	[✓ YES]	NO	
11.	Were correct containers used for the tests indicated?	[✓ YES]	NO	
12.	Were 5035 vials acceptable (no extra labels, not leaking)?	YES	NO	[✓ N/A]
13.	Air Samples: Cassettes / Tubes Intact with MS? Canisters Pressurized	Tedlar® Bags Inflated		[✓ N/A]

pH	Lot of test paper	Reagent	Preserved? Yes	Preserved? No	Lot Received	Exp	Sample ID Adjusted	Vol. Added	Lot Added	Final pH
≥12		NaOH								
≤2		HNO_3								
≤2	225419	H_2SO_4	✓		Client					
<4		$NaHSO_4$								
5-9		For 608pest			No=Notify for 3day					
Residual Chlorine (-)		For CN, Phenol, 625, 608pest, 522			If +, contact PM to add $Na_2S_2O_3$ (625, 608, CN), ascorbic (phenol).					
		$Na_2S_2O_3$								
		ZnAcetate	-	-						
		HCl	**	**						

**VOAs and 1664 Not to be tested before analysis. Otherwise, all bottles of all samples with chemical preservatives are checked (not just representatives).

Bottle lot numbers: Client

Explain all Discrepancies/ Other Comments:

HPROD	BULK
HTR	FLDT
SUB	HGFB
ALS	LL3541

Labels secondary reviewed by:
PC Secondary Review: 9/1/20

*significant air bubbles: VOA > 5-6 mm : WC >1 in. diameter

P:\INTRANET\QAQC\Forms Controlled\Cooler Receipt r17.doc 1/16/2020

Miscellaneous Forms

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

REPORT QUALIFIERS AND DEFINITIONS

U	Analyte was analyzed for but not detected. The sample quantitation limit has been corrected for dilution and for percent moisture, unless otherwise noted in the case narrative.
J	Estimated value due to either being a Tentatively Identified Compound (TIC) or that the concentration is between the MRL and the MDL. Concentrations are not verified within the linear range of the calibration. For DoD: concentration >40% difference between two GC columns (pesticides/Arclors).
B	Analyte was also detected in the associated method blank at a concentration that may have contributed to the sample result.
E	Inorganics- Concentration is estimated due to the serial dilution was outside control limits.
E	Organics- Concentration has exceeded the calibration range for that specific analysis.
D	Concentration is a result of a dilution, typically a secondary analysis of the sample due to exceeding the calibration range or that a surrogate has been diluted out of the sample and cannot be assessed.
*	Indicates that a quality control parameter has exceeded laboratory limits. Under the ñNotesò column of the Form I, this qualifier denotes analysis was performed out of Holding Time.
H	Analysis was performed out of hold time for tests that have an ñimmediateò hold time criteria.
#	Spike was diluted out.
+	Correlation coefficient for MSA is <0.995.
N	Inorganics- Matrix spike recovery was outside laboratory limits.
N	Organics- Presumptive evidence of a compound (reported as a TIC) based on the MS library search.
S	Concentration has been determined using Method of Standard Additions (MSA).
W	Post-Digestion Spike recovery is outside control limits and the sample absorbance is <50% of the spike absorbance.
P	Concentration >40% difference between the two GC columns.
C	Confirmed by GC/MS
Q	DoD reports: indicates a pesticide/Aroclor is not confirmed (Ó100% Difference between two GC columns).
X	See Case Narrative for discussion.
MRL LOQ	Method Reporting Limit. Also known as: Limit of Quantitation (LOQ) The lowest concentration at which the method analyte may be reliably quantified under the method conditions.
MDL	Method Detection Limit. A statistical value derived from a study designed to provide the lowest concentration that will be detected 99% of the time. Values between the MDL and MRL are estimated (see J qualifier).
LOD	Limit of Detection. A value at or above the MDL which has been verified to be detectable.
ND	Non-Detect. Analyte was not detected at the concentration listed. Same as U qualifier.

Rochester Lab ID # for State Certifications[1]

Connecticut ID # PH0556	Maine ID #NY0032	Pennsylvania ID# 68-786
Delaware Approved	New Hampshire ID # 2941	Rhode Island ID # 158
DoD ELAP #65817	New York ID # 10145	Virginia #460167
Florida ID # E87674	North Carolina #676	

[1] Analyses were performed according to our laboratoryôs NELAP-approved quality assurance program and any applicable state or agency requirements. The test results meet requirements of the current NELAP/TNI standards or state or agency requirements, where applicable, except as noted in the case narrative. Since not all analyte/method/matrix combinations are offered for state/NELAC accreditation, this report may contain results which are not accredited. For a specific list of accredited analytes, contact the laboratory or go to https://www.alsglobal.com/locations/americas/north-america/usa/new-york/rochester-environmental

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons
tr	Trace level is the concentration of an analyte that is less than the PQL but greater than or equal to the MDL.

Client: Alpha Analytical Inc. (Westborough, MA) **Service Request:** R2007726
Project: L2034262/

Sample Name: FB01-G1-BOT-S **Date Collected:** 08/20/20
Lab Code: R2007726-001 **Date Received:** 08/25/20
Sample Matrix: Water

Analysis Method	**Extracted/Digested By**	**Analyzed By**
350.1		MROGERSON
351.2	KMENGS	GNITAJOUPPI
353.2		MROGERSON

Sample Name: FB01-G1-MID-S **Date Collected:** 08/20/20
Lab Code: R2007726-002 **Date Received:** 08/25/20
Sample Matrix: Water

Analysis Method	**Extracted/Digested By**	**Analyzed By**
350.1		MROGERSON
351.2	KMENGS	GNITAJOUPPI
353.2		MROGERSON

Sample Name: FB01-G1-SUR-S **Date Collected:** 08/20/20
Lab Code: R2007726-003 **Date Received:** 08/25/20
Sample Matrix: Water

Analysis Method	**Extracted/Digested By**	**Analyzed By**
350.1		MROGERSON
351.2	KMENGS	GNITAJOUPPI
353.2		MROGERSON

Sample Name: FB02-G1-BOT-S **Date Collected:** 08/20/20
Lab Code: R2007726-004 **Date Received:** 08/25/20
Sample Matrix: Water

Analysis Method	**Extracted/Digested By**	**Analyzed By**
350.1		MROGERSON
351.2	KMENGS	GNITAJOUPPI
353.2		MROGERSON

ALS Group USA, Corp.

dba ALS Environmental

Analyst Summary report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262/		

Sample Name:	FB02-G1-MID-S	**Date Collected:**	08/20/20
Lab Code:	R2007726-005	**Date Received:**	08/25/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	KMENGS	GNITAJOUPPI
353.2		MROGERSON

Sample Name:	FB02-G1-SUR-S	**Date Collected:**	08/20/20
Lab Code:	R2007726-006	**Date Received:**	08/25/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	KMENGS	GNITAJOUPPI
353.2		MROGERSON

INORGANIC PREPARATION METHODS

The preparation methods associated with this report are found in these tables unless discussed in the case narrative.

Water/Liquid Matrix

Analytical Method	Preparation Method
200.7	200.2
200.8	200.2
6010C	3005A/3010A
6020A	ILM05.3
9034 Sulfide Acid Soluble	9030B
SM 4500-CN-E Residual Cyanide	SM 4500-CN-G
SM 4500-CN-E WAD Cyanide	SM 4500-CN-I

Solid/Soil/Non-Aqueous Matrix

Analytical Method	Preparation Method
6010C	3050B
6020A	3050B
6010C TCLP (1311) extract	3005A/3010A
6010 SPLP (1312) extract	3005A/3010A
7199	3060A
300.0 Anions/ 350.1/ 353.2/ SM 2320B/ SM 5210B/ 9056A Anions	DI extraction
For analytical methods not listed, the preparation method is the same as the analytical method reference.	

Sample Results

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

General Chemistry

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20 11:10
Sample Matrix:	Water	**Date Received:**	08/25/20 09:30
Sample Name:	FB01-G1-BOT-S	**Basis:**	NA
Lab Code:	R2007726-001		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.026**	mg/L	0.010	1	08/26/20 21:12	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.069**	mg/L	0.050	1	08/29/20 14:44	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.17**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.11**	mg/L	0.10	1	08/28/20 12:50	08/27/20	

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20 11:35
Sample Matrix:	Water	**Date Received:**	08/25/20 09:30
Sample Name:	FB01-G1-MID-S	**Basis:**	NA
Lab Code:	R2007726-002		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.020**	mg/L	0.010	1	08/26/20 21:13	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.057**	mg/L	0.050	1	08/29/20 14:45	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.24**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.18**	mg/L	0.10	1	08/28/20 12:51	08/27/20	

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20 11:55
Sample Matrix:	Water	**Date Received:**	08/25/20 09:30
Sample Name:	FB01-G1-SUR-S	**Basis:**	NA
Lab Code:	R2007726-003		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.011**		mg/L	0.010	1	08/26/20 21:14	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	08/29/20 14:46	NA	
Nitrogen, Total as Nitrogen	Calculation	0.1	U	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	08/28/20 12:54	08/27/20	

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20 08:00
Sample Matrix:	Water	**Date Received:**	08/25/20 09:30
Sample Name:	FB02-G1-BOT-S	**Basis:**	NA
Lab Code:	R2007726-004		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.042**	mg/L	0.010	1	08/26/20 21:15	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.076**	mg/L	0.050	1	08/29/20 14:48	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.28**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.20**	mg/L	0.10	1	08/28/20 12:55	08/27/20	

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20 08:25
Sample Matrix:	Water	**Date Received:**	08/25/20 09:30
Sample Name:	FB02-G1-MID-S	**Basis:**	NA
Lab Code:	R2007726-005		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.010**		mg/L	0.010	1	08/26/20 21:16	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.065**		mg/L	0.050	1	08/29/20 14:49	NA	
Nitrogen, Total as Nitrogen	Calculation	0.1	U	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	08/28/20 12:56	08/27/20	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20 08:40
Sample Matrix:	Water	**Date Received:**	08/25/20 09:30
Sample Name:	FB02-G1-SUR-S	**Basis:**	NA
Lab Code:	R2007726-006		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.028**		mg/L	0.010	1	08/26/20 21:23	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	08/29/20 14:51	NA	
Nitrogen, Total as Nitrogen	Calculation	0.1	U	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.16**		mg/L	0.10	1	08/28/20 12:57	08/27/20	

QC Summary Forms

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

General Chemistry

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	NA
Sample Matrix:	Water	**Date Received:**	NA
Sample Name:	Method Blank	**Basis:**	NA
Lab Code:	R2007726-MB1		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	08/26/20 20:41	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	08/29/20 14:25	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	08/28/20 12:46	08/27/20	

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	NA
Sample Matrix:	Water	**Date Received:**	NA
Sample Name:	Method Blank	**Basis:**	NA
Lab Code:	R2007726-MB2		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Q
Ammonia as Nitrogen	350.1	0.010 U	mg/L	0.010	1	08/26/20 21:21	

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20
Sample Matrix:	Water	**Date Received:**	08/25/20
		Date Analyzed:	08/28/20
		Date Extracted:	08/27/20

Duplicate Matrix Spike Summary
Nitrogen, Total Kjeldahl (TKN)

Sample Name:	FB01-G1-MID-S	**Units:**	mg/L
Lab Code:	R2007726-002	**Basis:**	NA
Analysis Method:	351.2		
Prep Method:	Method		

		Matrix Spike R2007726-002MS			**Duplicate Matrix Spike** R2007726-002DMS					
Analyte Name	**Sample Result**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
Nitrogen, Total Kjeldahl (TKN)	0.18	2.65	2.50	99	2.70	2.50	101	90-110	2	20

Results flagged with an asterisk (*) indicate values outside control criteria.

Results flagged with a pound (#) indicate the control criteria is not applicable.

Percent recoveries and relative percent differences (RPD) are determined by the software using values in the calculation which have not been rounded.

Matrix Spike and Matrix Spike Duplicate Data is presented for information purposes only. The matrix may or may not be relevant to samples reported in this report. The laboratory evaluates system performance based on the LCS and LCSD control limits.

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20
Sample Matrix:	Water	**Date Received:**	08/25/20
		Date Analyzed:	08/26/20

Duplicate Matrix Spike Summary
Ammonia as Nitrogen

Sample Name:	FB02-G1-MID-S	**Units:**	mg/L
Lab Code:	R2007726-005	**Basis:**	NA
Analysis Method:	350.1		

		Matrix Spike R2007726-005MS			Duplicate Matrix Spike R2007726-005DMS					
Analyte Name	**Sample Result**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
Ammonia as Nitrogen	0.010	0.237	0.250	91	0.240	0.250	92	90-110	1	20

Results flagged with an asterisk (*) indicate values outside control criteria.

Results flagged with a pound (#) indicate the control criteria is not applicable.

Percent recoveries and relative percent differences (RPD) are determined by the software using values in the calculation which have not been rounded.

Matrix Spike and Matrix Spike Duplicate Data is presented for information purposes only. The matrix may or may not be relevant to samples reported in this report. The laboratory evaluates system performance based on the LCS and LCSD control limits.

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Collected:**	08/20/20
Sample Matrix:	Water	**Date Received:**	08/25/20
		Date Analyzed:	08/26/20

Duplicate Matrix Spike Summary
Ammonia as Nitrogen

Sample Name:	FB02-G1-SUR-S	**Units:**	mg/L
Lab Code:	R2007726-006	**Basis:**	NA
Analysis Method:	350.1		

		Matrix Spike R2007726-006MS			**Duplicate Matrix Spike** R2007726-006DMS					
Analyte Name	**Sample Result**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
Ammonia as Nitrogen	0.028	0.261	0.250	93	0.259	0.250	93	90-110	<1	20

Results flagged with an asterisk (*) indicate values outside control criteria.

Results flagged with a pound (#) indicate the control criteria is not applicable.

Percent recoveries and relative percent differences (RPD) are determined by the software using values in the calculation which have not been rounded.

Matrix Spike and Matrix Spike Duplicate Data is presented for information purposes only. The matrix may or may not be relevant to samples reported in this report. The laboratory evaluates system performance based on the LCS and LCSD control limits.

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Analyzed:**	08/26/20 - 08/29/20
Sample Matrix:	Water		

Lab Control Sample Summary
General Chemistry Parameters

Units:mg/L
Basis:NA

Lab Control Sample
R2007726-LCS1

Analyte Name	Analytical Method	Result	Spike Amount	% Rec	% Rec Limits
Ammonia as Nitrogen	350.1	0.243	0.250	97	90-110
Nitrate+Nitrite as Nitrogen	353.2	0.519	0.500	104	90-110
Nitrogen, Total Kjeldahl (TKN)	351.2	2.52	2.50	101	90-110

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2007726
Project:	L2034262	**Date Analyzed:**	08/26/20
Sample Matrix:	Water		

Lab Control Sample Summary
General Chemistry Parameters

Units:mg/L
Basis:NA

Lab Control Sample
R2007726-LCS2

Analyte Name	Analytical Method	Result	Spike Amount	% Rec	% Rec Limits
Ammonia as Nitrogen	350.1	0.243	0.250	97	90-110

ALPHA ANALYTICAL

8 Walkup Drive
Westboro, MA 01581
Tel: 508-898-9220

320 Forbes Blvd
Mansfield, MA 02048
Tel: 508-822-9300

CHAIN OF CUSTODY

PAGE 1 OF 1

Date Rec'd in Lab: 8/21/20

ALPHA Job #: L2034262

Client Information

Client: Ransom Consulting LLC
Address: 112 Corporate Dr
Portsmouth NH 03801
Phone: 603-436-1490
Email: elizabeth.ransom@ransomenv.com
cc: drew.fuchs@ransomenv.com

Project Information

Project Name: AAF
Project Location: Maine
Project #: 201.05012.002
Project Manager: Elizabeth Ransom
ALPHA Quote #:

Turn-Around Time

☒ Standard ☐ RUSH (only confirmed if pre-approved)

Date Due:

Report Information - Data Deliverables

☒ ADEx ☒ EMAIL

Billing Information

☐ Same as Client info PO #:

Regulatory Requirements & Project Information Requirements

☐ Yes ☒ No MA MCP Analytical Methods ☐ Yes ☒ No CT RCP Analytical Methods
☐ Yes ☒ No Matrix Spike Required on this SDG? (Required for MCP Inorganics)
☐ Yes ☒ No GW1 Standards (Info Required for Metals & EPH with Targets)
☐ Yes ☒ No NPDES RGP
☒ Other State /Fed Program MEDEP Criteria

Additional Project Information:
Ammonia, nitrate/nitrite field filtered
Attn: Melissa Gulli - to be subcontracted
Melissa to discuss total N w/ Elizabeth Ransom prior to analysis

ANALYSIS

SAMPLE INFO
Filtration
☐ Field
☐ Lab to do
Preservation
☐ Lab to do

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler Initials	VOC: ☐ 8260 ☐ 624 ☐ 524.2	SVOC: ☐ ABN ☐ PAH	METALS: ☐ MCP 13 ☐ MCP 14 ☐ RCP 15	METALS: ☐ RCRA5 ☐ RCRA8 ☐ PP13	EPH: ☐ Ranges & Targets ☐ Ranges Only	VPH: ☐ Ranges & Targets ☐ Ranges Only	☐ PCB ☐ PEST	TPH: ☐ Quant Only ☐ Fingerprint	Total Kjeldahl Nitrogen	Ammonia	Nitrate/Nitrite	Total Nitrogen			Sample Comments	TOTAL # BOTTLES
34262-01	FB01-G1-BOT-S	8-20-20	11:10	Sea Water	DAP									X	X	X	X				3
-02	FB01-G1-MID-S	↓	11:35	↓	↓									X	X	X	X				3
-03	FB01-G1-SUR-S	↓	11:55	↓	↓									X	X	X	X				3
-04	FB02-G1-BOT-S	↓	8:00	↓	↓									X	X	X	X				3
-05	FB02-G1-MID-S	↓	8:25	↓	↓									X	X	X	X				3
-06	FB02-G1-SUR-S	↓	8:40	↓	↓									X	X	X	X				3
					Container Type									P	P	P	P			PAF	
					Preservative									D	D	A	D				

Container Type
P= Plastic
A= Amber glass
V= Vial
G= Glass
B= Bacteria cup
C= Cube
O= Other
E= Encore
D= BOD Bottle

Preservative
A= None
B= HCl
C= HNO_3
D= H_2SO_4
E= NaOH
F= MeOH
G= $NaHSO_4$
H = $Na_2S_2O_3$
I= Ascorbic Acid
J = NH_4Cl
K= Zn Acetate
O= Other

Relinquished By:	Date/Time	Received By:	Date/Time
Drew F	8-20-20/22:45	NHSC Storage Locker	8-20-20/22:45
secured storage	8/21/20 745	AAL	8/21/20 745
AAL	8/21/20 800	Stor	8/21/2020 20:

All samples submitted are subject to Alpha's Terms and Conditions.
See reverse side.

FORM NO: 01-01 (rev. 12-Mar-2012)

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2034262

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2034262

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com TKN-351 NH3- by 350 NO2/NO3-353

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB01-G1-BOT-S	08-20-20 11:10	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB01-G1-MID-S	08-20-20 11:35	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB01-G1-SUR-S	08-20-20 11:55	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB02-G1-BOT-S	08-20-20 08:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB02-G1-MID-S	08-20-20 08:25	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	
	FB02-G1-SUR-S	08-20-20 08:40	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen	

Relinquished By:	Date/Time:	Received By:	Date/Time:
	8/24/20		

Form No: AL_subcoc

ANALYTICAL REPORT

Lab Number:	L2040456
Client:	Ransom Consulting, Inc. 112 Corporate Drive Pease International Tradeport Portsmouth, NH 03801
ATTN:	Elizabeth Ransom
Phone:	(603) 436-1490
Project Name:	AAF
Project Number:	201.05012.002
Report Date:	10/12/20

Certifications & Approvals: MA (M-MA086), NH NELAP (2064), CT (PH-0574), IL (200077), ME (MA00086), MD (348), NJ (MA935), NY (11148), NC (25700/666), PA (68-03671), RI (LAO00065), TX (T104704476), VT (VT-0935), VA (460195), USDA (Permit #P330-17-00196).

Eight Walkup Drive, Westborough, MA 01581-1019
508-898-9220 (Fax) 508-898-9193 800-624-9220 - www.alphalab.com

Project Name:	AAF	**Lab Number:**	L2040456
Project Number:	201.05012.002	**Report Date:**	10/12/20

Alpha Sample ID	Client ID	Matrix	Sample Location	Collection Date/Time	Receive Date
L2040456-01	FB01-SUR	WATER	MAINE	09/24/20 09:30	09/24/20
L2040456-02	FB01-MID	WATER	MAINE	09/24/20 09:15	09/24/20
L2040456-03	FB01-BOT	WATER	MAINE	09/24/20 08:50	09/24/20
L2040456-04	FB02-SUR	WATER	MAINE	09/24/20 10:35	09/24/20
L2040456-05	FB02-MID	WATER	MAINE	09/24/20 10:20	09/24/20
L2040456-06	FB02-BOT	WATER	MAINE	09/24/20 10:00	09/24/20

Case Narrative

The samples were received in accordance with the Chain of Custody and no significant deviations were encountered during the preparation or analysis unless otherwise noted. Sample Receipt, Container Information, and the Chain of Custody are located at the back of the report.

Results contained within this report relate only to the samples submitted under this Alpha Lab Number and meet NELAP requirements for all NELAP accredited parameters unless otherwise noted in the following narrative. The data presented in this report is organized by parameter (i.e. VOC, SVOC, etc.). Sample specific Quality Control data (i.e. Surrogate Spike Recovery) is reported at the end of the target analyte list for each individual sample, followed by the Laboratory Batch Quality Control at the end of each parameter. Tentatively Identified Compounds (TICs), if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported. If a sample was re-analyzed or re-extracted due to a required quality control corrective action and if both sets of data are reported, the Laboratory ID of the re-analysis or re-extraction is designated with an "R" or "RE", respectively.

When multiple Batch Quality Control elements are reported (e.g. more than one LCS), the associated samples for each element are noted in the grey shaded header line of each data table. Any Laboratory Batch, Sample Specific % recovery or RPD value that is outside the listed Acceptance Criteria is bolded in the report. In reference to questions H (CAM) or 4 (RCP) when "NO" is checked, the performance criteria for CAM and RCP methods allow for some quality control failures to occur and still be within method compliance. In these instances, the specific failure is not narrated but noted in the associated QC Outlier Summary Report, located directly after the Case Narrative. QC information is also incorporated in the Data Usability Assessment table (Format 11) of our Data Merger tool, where it can be reviewed in conjunction with the sample result, associated regulatory criteria and any associated data usability implications.

Soil/sediments, solids and tissues are reported on a dry weight basis unless otherwise noted. Definitions of all data qualifiers and acronyms used in this report are provided in the Glossary located at the back of the report.

HOLD POLICY - For samples submitted on hold, Alpha's policy is to hold samples (with the exception of Air canisters) free of charge for 21 calendar days from the date the project is completed. After 21 calendar days, we will dispose of all samples submitted including those put on hold unless you have contacted your Alpha Project Manager and made arrangements for Alpha to continue to hold the samples. Air canisters will be disposed after 3 business days from the date the project is completed.

Please contact Project Management at 800-624-9220 with any questions.

Case Narrative (continued)

Report Submission
October 12, 2020: This final report includes the results of all requested analyses.
October 01, 2020: This is a preliminary report.

All non-detect (ND) or estimated concentrations (J-qualified) have been quantitated to the limit noted in the MDL column.

The analyses of Ammonia, Nitrate, Nitrite, Total Kjeldahl Nitrogran, Total Nitrogren and Total Suspended Solids were subcontracted. A copy of the laboratory report is included as an addendum. Please note: This data is only available in PDF format and is not available on Data Merger.

Phosphorus, Orthophosphate
L2040456-05: The OPHOS result is slightly higher than the TPHOS result; however, the sample result is less than five times the reporting limit. Therefore, no further action was taken.

I, the undersigned, attest under the pains and penalties of perjury that, to the best of my knowledge and belief and based upon my personal inquiry of those responsible for providing the information contained in this analytical report, such information is accurate and complete. This certificate of analysis is not complete unless this page accompanies any and all pages of this report.

Authorized Signature: Tiffani Morrissey - Tiffani Morrissey

Title: Technical Director/Representative Date: 10/12/20

INORGANICS
&
MISCELLANEOUS

Project Name:	AAF	**Lab Number:**	L2040456
Project Number:	201.05012.002	**Report Date:**	10/12/20

SAMPLE RESULTS

Lab ID:	L2040456-01	Date Collected:	09/24/20 09:30
Client ID:	FB01-SUR	Date Received:	09/24/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	6.2		mg/l	5.0	NA	1	-	09/29/20 12:15	121,2540D	AC
Phosphorus, Total	0.018		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:16	121,4500P-E	SD
Phosphorus, Orthophosphate	0.023		mg/l	0.005	0.001	1	-	09/26/20 02:50	121,4500P-E	CB

SAMPLE RESULTS

Lab ID: L2040456-02
Client ID: FB01-MID
Sample Location: MAINE
Date Collected: 09/24/20 09:15
Date Received: 09/24/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	09/29/20 12:15	121,2540D	AC
Phosphorus, Total	0.027		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:17	121,4500P-E	SD
Phosphorus, Orthophosphate	0.026		mg/l	0.005	0.001	1	-	09/26/20 02:51	121,4500P-E	CB

SAMPLE RESULTS

Lab ID:	L2040456-03	Date Collected:	09/24/20 08:50
Client ID:	FB01-BOT	Date Received:	09/24/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	5.3		mg/l	5.0	NA	1	-	09/29/20 12:15	121,2540D	AC
Phosphorus, Total	0.026		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:18	121,4500P-E	SD
Phosphorus, Orthophosphate	0.025		mg/l	0.005	0.001	1	-	09/26/20 02:52	121,4500P-E	CB

SAMPLE RESULTS

Lab ID: L2040456-04
Client ID: FB02-SUR
Sample Location: MAINE

Date Collected: 09/24/20 10:35
Date Received: 09/24/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	13.		mg/l	5.0	NA	1	-	09/28/20 13:30	121,2540D	AC
Phosphorus, Total	0.021		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:19	121,4500P-E	SD
Phosphorus, Orthophosphate	0.021		mg/l	0.005	0.001	1	-	09/26/20 02:53	121,4500P-E	CB

SAMPLE RESULTS

Lab ID: L2040456-05
Client ID: FB02-MID
Sample Location: MAINE
Date Collected: 09/24/20 10:20
Date Received: 09/24/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	09/29/20 12:15	121,2540D	AC
Phosphorus, Total	0.017		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:21	121,4500P-E	SD
Phosphorus, Orthophosphate	0.029		mg/l	0.005	0.001	1	-	09/26/20 02:53	121,4500P-E	CB

Project Name:	AAF	**Lab Number:**	L2040456
Project Number:	201.05012.002	**Report Date:**	10/12/20

SAMPLE RESULTS

Lab ID:	L2040456-06	Date Collected:	09/24/20 10:00
Client ID:	FB02-BOT	Date Received:	09/24/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	6.0		mg/l	5.0	NA	1	-	09/29/20 12:15	121,2540D	AC
Phosphorus, Total	0.037		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:23	121,4500P-E	SD
Phosphorus, Orthophosphate	0.021		mg/l	0.005	0.001	1	-	09/26/20 02:54	121,4500P-E	CB

Method Blank Analysis
Batch Quality Control

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1414822-1										
Phosphorus, Orthophosphate	0.002	J	mg/l	0.005	0.001	1	-	09/26/20 02:50	121,4500P-E	CB
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1415087-1										
Phosphorus, Total	ND		mg/l	0.010	0.004	1	09/27/20 09:10	09/27/20 13:00	121,4500P-E	SD
General Chemistry - Westborough Lab for sample(s): 04 Batch: WG1415331-1										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	09/28/20 13:30	121,2540D	AC
General Chemistry - Westborough Lab for sample(s): 01-03,05-06 Batch: WG1415856-1										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	09/29/20 12:15	121,2540D	AC

Lab Control Sample Analysis
Batch Quality Control

Project Name: AAF
Project Number: 201.05012.002

Lab Number: L2040456
Report Date: 10/12/20

Parameter	LCS %Recovery	Qual	LCSD %Recovery	Qual	%Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1414822-2								
Phosphorus, Orthophosphate	102		-		90-110	-		
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1415087-2								
Phosphorus, Total	101		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 04 Batch: WG1415331-2								
Solids, Total Suspended	98		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 01-03,05-06 Batch: WG1415856-2								
Solids, Total Suspended	118		-		80-120	-		

Matrix Spike Analysis
Batch Quality Control

Project Name: AAF
Project Number: 201.05012.002
Lab Number: L2040456
Report Date: 10/12/20

Parameter	Native Sample	MS Added	MS Found	MS %Recovery	Qual	MSD Found	MSD %Recovery	Qual	Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1414822-4 QC Sample: L2040456-02 Client ID: FB01-MID												
Phosphorus, Orthophosphate	0.026	0.5	0.517	98		-	-		80-120	-		20

Lab Duplicate Analysis

Batch Quality Control

Project Name: AAF

Project Number: 201.05012.002

Lab Number: L2040456

Report Date: 10/12/20

Parameter	Native Sample	Duplicate Sample	Units	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 QC Batch ID: WG1414822-3 QC Sample: L2040456-01 Client ID: FB01-SUR						
Phosphorus, Orthophosphate	0.023	0.023	mg/l	0		20

Sample Receipt and Container Information

Were project specific reporting limits specified? YES

Cooler Information

Cooler	Custody Seal
A	Present/Intact
B	Present/Intact

Container Information

Container ID	Container Type	Cooler	Initial pH	Final pH	Temp deg C	Pres	Seal	Frozen Date/Time	Analysis(*)
L2040456-01A	Plastic 120ml unpreserved	A	7	7	3.0	Y	Present/Intact		OPHOS-4500(2)
L2040456-01B	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		TPHOS-4500(28)
L2040456-01C	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-01D	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-01E	Plastic 500ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-TKN(),SUB-TOTAL NITROGEN()
L2040456-01F	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		SUB-TSS(7)
L2040456-01G	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		TSS-2540(7)
L2040456-02A	Plastic 120ml unpreserved	A	7	7	3.0	Y	Present/Intact		OPHOS-4500(2)
L2040456-02B	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		TPHOS-4500(28)
L2040456-02C	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-02D	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-02E	Plastic 500ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-TKN(),SUB-TOTAL NITROGEN()
L2040456-02F	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		SUB-TSS(7)
L2040456-02G	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		TSS-2540(7)
L2040456-03A	Plastic 120ml unpreserved	A	7	7	3.0	Y	Present/Intact		OPHOS-4500(2)
L2040456-03B	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		TPHOS-4500(28)
L2040456-03C	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-03D	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-03E	Plastic 500ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-TOTAL NITROGEN(),SUB-TKN()
L2040456-03F	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		SUB-TSS(7)
L2040456-03G	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		TSS-2540(7)
L2040456-04A	Plastic 120ml unpreserved	B	7	7	4.9	Y	Present/Intact		OPHOS-4500(2)

*Values in parentheses indicate holding time in days

Container Information

Container ID	Container Type	Cooler	Initial pH	Final pH	Temp deg C	Pres	Seal	Frozen Date/Time	Analysis(*)
L2040456-04B	Plastic 250ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		TPHOS-4500(28)
L2040456-04C	Plastic 250ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-04D	Plastic 250ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-04E	Plastic 500ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		SUB-TOTAL NITROGEN(),SUB-TKN()
L2040456-04F	Plastic 950ml unpreserved	B	7	7	4.9	Y	Present/Intact		SUB-TSS(7)
L2040456-04G	Plastic 950ml unpreserved	B	7	7	4.9	Y	Present/Intact		TSS-2540(7)
L2040456-05A	Plastic 120ml unpreserved	B	7	7	4.9	Y	Present/Intact		OPHOS-4500(2)
L2040456-05B	Plastic 250ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		TPHOS-4500(28)
L2040456-05C	Plastic 250ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-05D	Plastic 250ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-05E	Plastic 500ml H2SO4 preserved	B	<2	<2	4.9	Y	Present/Intact		SUB-TKN()
L2040456-05F	Plastic 950ml unpreserved	B	7	7	4.9	Y	Present/Intact		SUB-TSS(7)
L2040456-05G	Plastic 950ml unpreserved	B	7	7	4.9	Y	Present/Intact		TSS-2540(7)
L2040456-06A	Plastic 120ml unpreserved	A	7	7	3.0	Y	Present/Intact		OPHOS-4500(2)
L2040456-06B	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		TPHOS-4500(28)
L2040456-06C	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-06D	Plastic 250ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-NH3(28),SUB-NO3/NO2()
L2040456-06E	Plastic 500ml H2SO4 preserved	A	<2	<2	3.0	Y	Present/Intact		SUB-TKN()
L2040456-06F	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		SUB-TSS(7)
L2040456-06G	Plastic 950ml unpreserved	A	7	7	3.0	Y	Present/Intact		TSS-2540(7)

*Values in parentheses indicate holding time in days

GLOSSARY

Acronyms

DL	- Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the limit of quantitation (LOQ). The DL includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
EDL	- Estimated Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The EDL includes any adjustments from dilutions, concentrations or moisture content, where applicable. The use of EDLs is specific to the analysis of PAHs using Solid-Phase Microextraction (SPME).
EMPC	- Estimated Maximum Possible Concentration: The concentration that results from the signal present at the retention time of an analyte when the ions meet all of the identification criteria except the ion abundance ratio criteria. An EMPC is a worst-case estimate of the concentration.
EPA	- Environmental Protection Agency.
LCS	- Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LCSD	- Laboratory Control Sample Duplicate: Refer to LCS.
LFB	- Laboratory Fortified Blank: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LOD	- Limit of Detection: This value represents the level to which a target analyte can reliably be detected for a specific analyte in a specific matrix by a specific method. The LOD includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
LOQ	- Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.) Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
MDL	- Method Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The MDL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
MS	- Matrix Spike Sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. For Method 332.0, the spike recovery is calculated using the native concentration, including estimated values.
MSD	- Matrix Spike Sample Duplicate: Refer to MS.
NA	- Not Applicable.
NC	- Not Calculated: Term is utilized when one or more of the results utilized in the calculation are non-detect at the parameter's reporting unit.
NDPA/DPA	- N-Nitrosodiphenylamine/Diphenylamine.
NI	- Not Ignitable.
NP	- Non-Plastic: Term is utilized for the analysis of Atterberg Limits in soil.
NR	- No Results: Term is utilized when 'No Target Compounds Requested' is reported for the analysis of Volatile or Semivolatile Organic TIC only requests.
RL	- Reporting Limit: The value at which an instrument can accurately measure an analyte at a specific concentration. The RL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
RPD	- Relative Percent Difference: The results from matrix and/or matrix spike duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). Values which are less than five times the reporting limit for any individual parameter are evaluated by utilizing the absolute difference between the values; although the RPD value will be provided in the report.
SRM	- Standard Reference Material: A reference sample of a known or certified value that is of the same or similar matrix as the associated field samples.
STLP	- Semi-dynamic Tank Leaching Procedure per EPA Method 1315.
TEF	- Toxic Equivalency Factors: The values assigned to each dioxin and furan to evaluate their toxicity relative to 2,3,7,8-TCDD.
TEQ	- Toxic Equivalent: The measure of a sample's toxicity derived by multiplying each dioxin and furan by its corresponding TEF and then summing the resulting values.
TIC	- Tentatively Identified Compound: A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations.

Footnotes

1 - The reference for this analyte should be considered modified since this analyte is absent from the target analyte list of the original method.

Terms

Analytical Method: Both the document from which the method originates and the analytical reference method. (Example: EPA 8260B is shown as 1,8260B.) The codes for the reference method documents are provided in the References section of the Addendum.

Difference: With respect to Total Oxidizable Precursor (TOP) Assay analysis, the difference is defined as the Post-Treatment value minus the Pre-Treatment value.

Final pH: As it pertains to Sample Receipt & Container Information section of the report, Final pH reflects pH of container determined after adjustment at the laboratory, if applicable. If no adjustment required, value reflects Initial pH.

Frozen Date/Time: With respect to Volatile Organics in soil, Frozen Date/Time reflects the date/time at which associated Reagent Water-preserved vials were initially frozen. Note: If frozen date/time is beyond 48 hours from sample collection, value will be reflected in 'bold'.

Initial pH: As it pertains to Sample Receipt & Container Information section of the report, Initial pH reflects pH of container determined upon receipt, if applicable.

PAH Total: With respect to Alkylated PAH analyses, the 'PAHs, Total' result is defined as the summation of results for all or a subset of the following compounds: Naphthalene, C1-C4 Naphthalenes, 2-Methylnaphthalene, 1-Methylnaphthalene, Biphenyl, Acenaphthylene, Acenaphthene, Fluorene, C1-C3 Fluorenes, Phenanthrene, C1-C4 Phenanthrenes/Anthracenes, Anthracene, Fluoranthene, Pyrene, C1-C4 Fluoranthenes/Pyrenes, Benz(a)anthracene, Chrysene, C1-C4 Chrysenes, Benzo(b)fluoranthene, Benzo(j)+(k)fluoranthene, Benzo(e)pyrene, Benzo(a)pyrene, Perylene, Indeno(1,2,3-cd)pyrene, Dibenz(ah)+(ac)anthracene, Benzo(g,h,i)perylene. If a 'Total' result is requested, the results of its individual components will also be reported.

PFAS Total: With respect to PFAS analyses, the 'PFAS, Total (5)' result is defined as the summation of results for: PFHpA, PFHxS, PFOA, PFNA and PFOS. If a 'Total' result is requested, the results of its individual components will also be reported.

The target compound Chlordane (CAS No. 57-74-9) is reported for GC ECD analyses. Per EPA,this compound "refers to a mixture of chlordane isomers, other chlorinated hydrocarbons and numerous other components." (Reference: USEPA Toxicological Review of Chlordane, In Support of Summary Information on the Integrated Risk Information System (IRIS), December 1997.)

Total: With respect to Organic analyses, a 'Total' result is defined as the summation of results for individual isomers or Aroclors. If a 'Total' result is requested, the results of its individual components will also be reported. This is applicable to 'Total' results for methods 8260, 8081 and 8082.

Data Qualifiers

A	- Spectra identified as "Aldol Condensates" are byproducts of the extraction/concentration procedures when acetone is introduced in the process.
B	- The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For DOD-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank AND the analyte was detected above one-half the reporting limit (or above the reporting limit for common lab contaminants) in the associated method blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone).
C	- Co-elution: The target analyte co-elutes with a known lab standard (i.e. surrogate, internal standards, etc.) for co-extracted analyses.
D	- Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.
E	- Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.
F	- The ratio of quantifier ion response to qualifier ion response falls outside of the laboratory criteria. Results are considered to be an estimated maximum concentration.
G	- The concentration may be biased high due to matrix interferences (i.e, co-elution) with non-target compound(s). The result should be considered estimated.
H	- The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.
I	- The lower value for the two columns has been reported due to obvious interference.
J	- Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (MDL) or Estimated Detection Limit (EDL) for SPME-related analyses. This represents an estimated concentration for Tentatively Identified Compounds (TICs).
M	- Reporting Limit (RL) exceeds the MCP CAM Reporting Limit for this analyte.
ND	- Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for SPME-related analyses.
NJ	- Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where

Data Qualifiers

the identification is based on a mass spectral library search.

P - The RPD between the results for the two columns exceeds the method-specified criteria.

Q - The quality control sample exceeds the associated acceptance criteria. For DOD-related projects, LCS and/or Continuing Calibration Standard exceedences are also qualified on all associated sample results. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)

R - Analytical results are from sample re-analysis.

RE - Analytical results are from sample re-extraction.

S - Analytical results are from modified screening analysis.

REFERENCES

121 Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

LIMITATION OF LIABILITIES

Alpha Analytical, Inc. ID No.:**17873**
Facility: **Company-wide** Revision 17
Department: **Quality Assurance** Published Date: 4/28/2020 9:42:21 AM
Title: **Certificate/Approval Program Summary** Page 1 of 1

Certification Information

The following analytes are not included in our Primary NELAP Scope of Accreditation:

Westborough Facility
EPA 624/624.1: m/p-xylene, o-xylene, Naphthalene
EPA 8260C: NPW: 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene, Azobenzene; SCM: Iodomethane (methyl iodide), 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene.
EPA 8270D: NPW: Dimethylnaphthalene,1,4-Diphenylhydrazine; SCM: Dimethylnaphthalene,1,4-Diphenylhydrazine.
SM4500: NPW: Amenable Cyanide; SCM: Total Phosphorus, TKN, NO2, NO3.

Mansfield Facility
SM 2540D: TSS
EPA 8082A: NPW: PCB: 1, 5, 31, 87,101, 110, 141, 151, 153, 180, 183, 187.
EPA TO-15: Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 1-Methylnaphthalene.
EPA TO-12 Non-methane organics
EPA 3C Fixed gases
Biological Tissue Matrix: EPA 3050B

The following analytes are included in our Massachusetts DEP Scope of Accreditation

Westborough Facility:

Drinking Water
EPA 300.0: Chloride, Nitrate-N, Fluoride, Sulfate; **EPA 353.2**: Nitrate-N, Nitrite-N; **SM4500NO3-F**: Nitrate-N, Nitrite-N; **SM4500F-C, SM4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, SM2320B, SM2540C, SM4500H-B, SM4500NO2-B**
EPA 332: Perchlorate; **EPA 524.2:** THMs and VOCs; **EPA 504.1:** EDB, DBCP.
Microbiology: **SM9215B; SM9223-P/A, SM9223B-Colilert-QT,SM9222D**.

Non-Potable Water
SM4500H,B, EPA 120.1, SM2510B, SM2540C, SM2320B, SM4500CL-E, SM4500F-BC, SM4500NH3-BH: Ammonia-N and Kjeldahl-N, **EPA 350.1**: Ammonia-N, **LACHAT 10-107-06-1-B**: Ammonia-N, **EPA 351.1, SM4500NO3-F, EPA 353.2**: Nitrate-N**, SM4500P-E, SM4500P-B, E, SM4500SO4-E, SM5220D, EPA 410.4, SM5210B, SM5310C, SM4500CL-D, EPA 1664, EPA 420.1, SM4500-CN-CE, SM2540D, EPA 300: Chloride, Sulfate, Nitrate.**
EPA 624.1: Volatile Halocarbons & Aromatics,
EPA 608.3: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs
EPA 625.1: SVOC (Acid/Base/Neutral Extractables), **EPA 600/4-81-045**: PCB-Oil.
Microbiology: **SM9223B-Colilert-QT; Enterolert-QT, SM9221E, EPA 1600, EPA 1603.**

Mansfield Facility:

Drinking Water
EPA 200.7: Al, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Na, Ag, Ca, Zn. **EPA 200.8:** Al, Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Ni, Se, Ag, TL, Zn. **EPA 245.1** Hg.
EPA 522.

Non-Potable Water
EPA 200.7: Al, Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Sr, TL, Ti, V, Zn.
EPA 200.8: Al, Sb, As, Be, Cd, Cr, Cu, Fe, Pb, Mn, Ni, K, Se, Ag, Na, TL, Zn.
EPA 245.1 Hg.
SM2340B

For a complete listing of analytes and methods, please contact your Alpha Project Manager.

ALPHA ANALYTICAL
World Class Chemistry

8 Walkup Drive
Westboro, MA 01581
Tel: 508-898-9220

320 Forbes Blvd
Mansfield, MA 02048
Tel: 508-822-9300

CHAIN OF CUSTODY

PAGE 1 OF 1

Date Rec'd in Lab: 09/24/20

ALPHA Job #: L2040456

Client Information

Client: Ransom Consulting LLC
Address: 112 Corporate Drive
Portsmouth NH 03801
Phone: 603-436-1490
Email: elizabeth.ransom@ransomenv.com
cc: drew.fuchs@ransomenv.com

Additional Project Information:
Attn: Melissa Gullo
Subcontract: Ammonia, Nitrate/Nitrite, TKN, TSS
Alpha: Total P, orthophosphate, TSS

Project Information

Project Name: AAF
Project Location: Maine
Project #: 201.05012.002
Project Manager: Elizabeth Ransom
ALPHA Quote #:

Turn-Around Time

☒ Standard ☐ RUSH (only confirmed if pre-approved!)
Date Due:

Report Information - Data Deliverables

☒ ADEx ☒ EMAIL

Billing Information

☐ Same as Client info PO #:

Regulatory Requirements & Project Information Requirements

☐ Yes ☒ No MA MCP Analytical Methods ☐ Yes ☒ No CT RCP Analytical Methods
☐ Yes ☒ No Matrix Spike Required on this SDG? (Required for MCP Inorganics)
☐ Yes ☒ No GW1 Standards (Info Required for Metals & EPH with Targets)
☐ Yes ☒ No NPDES RGP
☒ Other State /Fed Program Maine DMR Criteria

ANALYSIS

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler Initials	VOC: ☐ 8260 ☐ 624 ☐ 524.2	SVOC: ☐ ABN ☐ PAH	METALS: ☐ MCP 13 ☐ MCP 14 ☐ RCP 15	METALS: ☐ RCRA5 ☐ RCRA8 ☐ PP13	EPH: ☐ Ranges & Targets ☐ Ranges Only	VPH: ☐ Ranges & Targets ☐ Ranges Only	☐ PCB ☐ PEST	TPH: ☐ Quant Only ☐ Fingerprint	Total Phosphate	Orthophosphate (Dissolved)	TSS	Ammonia (Dissolved)	Nitrate/Nitrite (Dissolved)	Total Kjeldahl Nitrogen	Sample Comments	TOTAL # BOTTLES
40456-01	FB01-SUR	9-24-20	9:30	Seawater	DAF									X	X	X	X	X	X		7
-02	FB01-MID	↓	9:15	↓	↓									X	X	X	X	X	X		7
-03	FB01-BOT		8:50											X	X	X	X	X	X		7
-04	FB02-SUR		10:35											X	X	X	X	X	X		7
-05	FB02-MID		10:20											X	X	X	X	X	X		7
-06	FB02-BOT		10:00											X	X	X	X	X	X		7
				Container Type										P	P	P	P	P	P		
				Preservative										D	A	A	D	D	D		

SAMPLE INFO
Filtration
☐ Field
☐ Lab to do
Preservation
☐ Lab to do

Container Type
P= Plastic
A= Amber glass
V= Vial
G= Glass
B= Bacteria cup
C= Cube
O= Other
E= Encore
D= BOD Bottle

Preservative
A= None
B= HCl
C= HNO_3
D= H_2SO_4
E= NaOH
F= MeOH
G= $NaHSO_4$
H = $Na_2S_2O_3$
I= Ascorbic Acid
J = NH_4Cl
K= Zn Acetate
O= Other

Relinquished By:	Date/Time	Received By:	Date/Time
Drew Fuchs	9-24-20/16:15	[illegible] Alpha [illegible]	9/24/20 16:15
[illegible] Alpha [illegible]	9/24/20 1910	[illegible]	9/24/20 1910
[illegible]			9/24/20 2340

All samples submitted are subject to Alpha's Terms and Conditions. See reverse side.
FORM NO: 01-01 (rev. 12-Mar-2012)

UNISHIP
COURIER SERVICES LLC

10 Iron Road
Hermon, Maine 04401

Bill Shipping Charge to

Shipper ☐ Recipient ☐
☐ Next Day ☐ Same Day

Special ____________

080875

Phone 207•848•7546 ■ Toll-Free 800•427•7547 ■ Fax 207•561•2467

390 US Route One, #3
Falmouth, Maine 04105

FROM: Shipper Alpha
Street 72 Center St.
Origin Brewer ME Zip Code 04412
Phone #

TO: Recipient Alpha
Street 8 Walkup Drive
Destination Westboro MA Zip Code 01581
Phone #

No. Pieces	Weight Each	Description of Items	Total Weight (Subject to Correction)	Oversize Charge	Shipping Charges
5		Coolers			
5	◀ TOTAL PIECES	WEIGHT GRAND TOTAL ▶		TOTAL CHARGES ▶	

Shipper authorizes Uniship to deliver this shipment without obtaining a delivery signature.
Shipper's Signature ____________

Please use complete ship to address.
Uniship can not deliver to P.O. Boxes.

RECEIVED, subject to the classifications and tariffs in effect on the date of the issue of this Bill of Lading, the property described above in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned, and destined as indicated above which said carrier (the word carrier being understood throughout this contract as meaning any person or corporation in possession of the property, under the contract) agrees to carry to its usual place of delivery at said destination, if on its route, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed as to each carrier of all or any of said property overall or any portion of said route to destination and as to each party at any time interested in all or any of said property, that every service to be performed hereunder shall be subject to all the bill of lading terms and conditions in the governing classification on the date of shipment.

Shipper hereby certifies that he is familiar with all the bill of lading terms and conditions in the governing classification and the said terms and conditions are hereby agreed to by the shipper and accepted for himself and his assigns.

SHIPPER
PICK-UP TIME 18:10
COURIER SIGNATURE
DATE 9-24-20
RECIPIENT
DELIVERY TIME 23:40
COURIER SIGNATURE
DATE 9-24-20

RECIPIENT COPY

A


SCI SPEC
PRECLEANED CONTAINERS
Committed to Quality Since 1963
Custody Seal Custody Seal Custody Seal Custody Seal
DATE 9/29/2020
SIGNATURE
scispec.com
SCI SPEC
PRECLEANED CONTAINERS
C20000 Committed to Quality Since 1963

B


SCI SPEC
PRECLEANED CONTAINERS
Committed to Quality Since 1963
Custody Seal Custody Seal Custody Seal Custody Seal
DATE 9/29/2020
SIGNATURE
scispec.com
SCI SPEC
PRECLEANED CONTAINERS
C20000 Committed to Quality Since 1963

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2040456

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2040456

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com Samples FF for NH3, NO3/NO2

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB01-SUR	09-24-20 09:30	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-MID	09-24-20 09:15	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-BOT	09-24-20 08:50	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-SUR	09-24-20 10:35	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-MID	09-24-20 10:20	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-BOT	09-24-20 10:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	

Relinquished By:	Date/Time:	Received By:	Date/Time:
	9/28/20		

Form No: AL_subcoc

October 12, 2020

Service Request No:R2008982

Melissa Gulli
Alpha Analytical Inc. (Westborough, MA)
8 Walkup Drive
Westborough, MA 01581

Laboratory Results for: L2040456

Dear Melissa,

Enclosed are the results of the sample(s) submitted to our laboratory September 29, 2020
For your reference, these analyses have been assigned our service request number **R2008982.**

All testing was performed according to our laboratory's quality assurance program and met the requirements of the TNI standards except as noted in the case narrative report. Any testing not included in the lab's accreditation is identified on a Non-Certified Analytes report. All results are intended to be considered in their entirety. ALS Environmental is not responsible for use of less than the complete report. Results apply only to the individual samples submitted to the lab for analysis, as listed in the report. The measurement uncertainty of the results included in this report is within that expected when using the prescribed method(s), and represented by Laboratory Control Sample control limits. Any events, such as QC failures or Holding Time exceedances, which may add to the uncertainty are explained in the report narrative or are flagged with qualifiers. The flags are explained in the Report Qualifiers and Definitions page of this report.

Please contact me if you have any questions. My extension is 7472. You may also contact me via email at Janice.Jaeger@alsglobal.com.

Respectfully submitted,

ALS Group USA, Corp. dba ALS Environmental

Vicky Collom for:

Janice Jaeger
Project Manager

ADDRESS 1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
PHONE +1 585 288 5380 | **FAX** +1 585 288 8475
ALS Group USA, Corp.
dba ALS Environmental

Narrative Documents

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Received:**	09/29/2020
Sample Matrix:	Water		

CASE NARRATIVE

All analyses were performed consistent with the quality assurance program of ALS Environmental. This report contains analytical results for samples for the Tier II level requested by the client.

Sample Receipt:

Six water samples were received for analysis at ALS Environmental on 09/29/2020. Any discrepancies upon initial sample inspection are annotated on the sample receipt and preservation form included within this report. The samples were stored at minimum in accordance with the analytical method requirements.

General Chemistry:

No significant anomalies were noted with this analysis.

Approved by ______________________ Date 10/12/2020

SAMPLE DETECTION SUMMARY

CLIENT ID: FB01-SUR **Lab ID: R2008982-001**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.010			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.075			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.24			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.17			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	5.8			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB01-MID **Lab ID: R2008982-002**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.019			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.103			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.23			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.13			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	3.8			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB01-BOT **Lab ID: R2008982-003**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.036			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.093			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.26			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.17			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	5.1			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-SUR **Lab ID: R2008982-004**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrate+Nitrite as Nitrogen	0.065			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.20			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.14			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	4.8			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-MID **Lab ID: R2008982-005**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrate+Nitrite as Nitrogen	0.089			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.21			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.12			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	3.5			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-BOT **Lab ID: R2008982-006**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.016			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.099			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.29			0.1	mg/L	Calculation

SAMPLE DETECTION SUMMARY

CLIENT ID: FB02-BOT			Lab ID: R2008982-006			
Analyte	**Results**	**Flag**	**MDL**	**MRL**	**Units**	**Method**
Nitrogen, Total Kjeldahl (TKN)	0.19			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	4.9			1.0	mg/L	SM 2540 D-1997 (2011)

Sample Receipt Information

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

Client: Alpha Analytical Inc. (Westborough, MA) **Service Request:**R2008982
Project: L2040456

SAMPLE CROSS-REFERENCE

SAMPLE #	CLIENT SAMPLE ID	DATE	TIME
R2008982-001	FB01-SUR	9/24/2020	0930
R2008982-002	FB01-MID	9/24/2020	0915
R2008982-003	FB01-BOT	9/24/2020	0850
R2008982-004	FB02-SUR	9/24/2020	1035
R2008982-005	FB02-MID	9/24/2020	1020
R2008982-006	FB02-BOT	9/24/2020	1000

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2040456

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2040456

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com Samples FF for NH3, NO3/NO2

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB01-SUR	09-24-20 09:30	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-MID	09-24-20 09:15	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-BOT	09-24-20 08:50	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-SUR	09-24-20 10:35	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-MID	09-24-20 10:20	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-BOT	09-24-20 10:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	

Relinquished By:	Date/Time:	Received By:	Date/Time:
	9/28/20		9/29/20 0930

Form No: AL_subcoc

R2008982 5
Alpha Analytical Inc. (Westborough, MA)
L2040456

R2008982 5
Alpha Analytical Inc. (Westborough, MA)
L2040466

Cooler Receipt and Preservation Check Form

Project/Client Alpha Folder Number

Cooler received on 9/29/2020 by:

COURIER: ALS [✓ UPS] FEDEX VELOCITY CLIENT

1	Were **Custody seals** on outside of cooler?	Y [✓ N]	5a	**Perchlorate** samples have required headspace?	Y N [✓ NA]
2	**Custody papers** properly completed (ink, signed)?	[✓ Y] N	5b	Did VOA vials, Alk,or Sulfide have sig* **bubbles**?	Y N [✓ NA]
3	Did all bottles arrive in good **condition** (unbroken)?	[✓ Y] N	6	Where did the bottles originate? ALS/ROC [✓ CLIENT]	
4	Circle: [✓ **Wet Ice**] **Dry Ice** **Gel packs** present?	[✓ Y] N	7	Soil VOA received as: Bulk Encore 5035set [✓ NA]	

8. Temperature Readings Date: 9/29/2020 Time: 0937 ID: IR#7 [✓ IR#10] From: [✓ Temp Blank] [✓ Sample Bottle]

Observed Temp (°C)	0.3	0.6	0.7				
Within 0-6°C?	[✓ Y] N	[✓ Y] N	[✓ Y] N	Y N	Y N	Y N	Y N
If <0°C, were samples frozen?	Y N	Y N	Y N	Y N	Y N	Y N	Y N

If out of Temperature, note packing/ice condition: ____________ Ice melted Poorly Packed (described below) Same Day Rule

&Client Approval to Run Samples: __________ Standing Approval Client aware at drop-off Client notified by: ______________

All samples held in storage location: R-002 by on 9/29/2020 at 0946
5035 samples placed in storage location: ________ by ________ on ______ at ______ within 48 hours of sampling? Y N

Cooler Breakdown/Preservation Check**: Date : 9/29/2020 Time: 1410 by:

9. Were all bottle labels complete (*i.e.* analysis, preservation, etc.)? [✓ YES] ~~NO~~
10. Did all bottle labels and tags agree with custody papers? [✓ YES] NO
11. Were correct containers used for the tests indicated? [✓ YES] NO
12. Were 5035 vials acceptable (no extra labels, not leaking)? YES NO [✓ N/A]
13. Air Samples: Cassettes / Tubes Intact with MS? Canisters Pressurized Tedlar® Bags Inflated [✓ N/A]

pH	Lot of test paper	Reagent	Preserved? Yes	Preserved? No	Lot Received	Exp	Sample ID Adjusted	Vol. Added	Lot Added	Final pH
≥12		NaOH								
≤2		HNO_3								
≤2	223419	H_2SO_4	✓		Client					
<4		$NaHSO_4$								
5-9		For 608pest			No=Notify for 3day					
Residual Chlorine (-)		For CN, Phenol, 625, 608pest, 522			If +, contact PM to add $Na_2S_2O_3$ (625, 608, CN), ascorbic (phenol).					
		$Na_2S_2O_3$								
		ZnAcetate	-	-						
		HCl	**	**						

**VOAs and 1664 Not to be tested before analysis. Otherwise, all bottles of all samples with chemical preservatives are checked (not just representatives).

Bottle lot numbers: Client

Explain all Discrepancies/ Other Comments:

* Excessive H_2SO_4 volume rec'd

HPROD	BULK
HTR	FLDT
SUB	HGFB
ALS	LL3541

Labels secondary reviewed by:

PC Secondary Review: AWI 10/6/20 *significant air bubbles: VOA > 5-6 mm : WC >1 in. diameter

Miscellaneous Forms

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

REPORT QUALIFIERS AND DEFINITIONS

U	Analyte was analyzed for but not detected. The sample quantitation limit has been corrected for dilution and for percent moisture, unless otherwise noted in the case narrative.
J	Estimated value due to either being a Tentatively Identified Compound (TIC) or that the concentration is between the MRL and the MDL. Concentrations are not verified within the linear range of the calibration. For DoD: concentration >40% difference between two GC columns (pesticides/Arclors).
B	Analyte was also detected in the associated method blank at a concentration that may have contributed to the sample result.
E	Inorganics- Concentration is estimated due to the serial dilution was outside control limits.
E	Organics- Concentration has exceeded the calibration range for that specific analysis.
D	Concentration is a result of a dilution, typically a secondary analysis of the sample due to exceeding the calibration range or that a surrogate has been diluted out of the sample and cannot be assessed.
*	Indicates that a quality control parameter has exceeded laboratory limits. Under the ñNotesò column of the Form I, this qualifier denotes analysis was performed out of Holding Time.
H	Analysis was performed out of hold time for tests that have an ñimmediateò hold time criteria.
#	Spike was diluted out.
+	Correlation coefficient for MSA is <0.995.
N	Inorganics- Matrix spike recovery was outside laboratory limits.
N	Organics- Presumptive evidence of a compound (reported as a TIC) based on the MS library search.
S	Concentration has been determined using Method of Standard Additions (MSA).
W	Post-Digestion Spike recovery is outside control limits and the sample absorbance is <50% of the spike absorbance.
P	Concentration >40% difference between the two GC columns.
C	Confirmed by GC/MS
Q	DoD reports: indicates a pesticide/Aroclor is not confirmed (Ó100% Difference between two GC columns).
X	See Case Narrative for discussion.
MRL LOQ	Method Reporting Limit. Also known as: Limit of Quantitation (LOQ) The lowest concentration at which the method analyte may be reliably quantified under the method conditions.
MDL	Method Detection Limit. A statistical value derived from a study designed to provide the lowest concentration that will be detected 99% of the time. Values between the MDL and MRL are estimated (see J qualifier).
LOD	Limit of Detection. A value at or above the MDL which has been verified to be detectable.
ND	Non-Detect. Analyte was not detected at the concentration listed. Same as U qualifier.

Rochester Lab ID # for State Certifications[1]

Connecticut ID # PH0556	Maine ID #NY0032	Pennsylvania ID# 68-786
Delaware Approved	New Hampshire ID # 2941	Rhode Island ID # 158
DoD ELAP #65817	New York ID # 10145	Virginia #460167
Florida ID # E87674	North Carolina #676	

---

[1] Analyses were performed according to our laboratoryôs NELAP-approved quality assurance program and any applicable state or agency requirements. The test results meet requirements of the current NELAP/TNI standards or state or agency requirements, where applicable, except as noted in the case narrative. Since not all analyte/method/matrix combinations are offered for state/NELAC accreditation, this report may contain results which are not accredited. For a specific list of accredited analytes, contact the laboratory or go to https://www.alsglobal.com/locations/americas/north-america/usa/new-york/rochester-environmental

---

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons
tr	Trace level is the concentration of an analyte that is less than the PQL but greater than or equal to the MDL.

ALS Group USA, Corp.
dba ALS Environmental

Analyst Summary report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456/		

Sample Name:	FB01-SUR	**Date Collected:**	09/24/20
Lab Code:	R2008982-001	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		SMEDBURY
351.2	KMENGS	GNITAJOUPPI
353.2		GNITAJOUPPI
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB01-MID	**Date Collected:**	09/24/20
Lab Code:	R2008982-002	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		SMEDBURY
351.2	KMENGS	GNITAJOUPPI
353.2		GNITAJOUPPI
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB01-BOT	**Date Collected:**	09/24/20
Lab Code:	R2008982-003	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		SMEDBURY
351.2	KMENGS	GNITAJOUPPI
353.2		GNITAJOUPPI
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-SUR	**Date Collected:**	09/24/20
Lab Code:	R2008982-004	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		SMEDBURY

ALS Group USA, Corp.
dba ALS Environmental

Analyst Summary report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456/		

Sample Name:	FB02-SUR	**Date Collected:**	09/24/20
Lab Code:	R2008982-004	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	**Extracted/Digested By**	**Analyzed By**
351.2	KMENGS	GNITAJOUPPI
353.2		GNITAJOUPPI
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-MID	**Date Collected:**	09/24/20
Lab Code:	R2008982-005	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	**Extracted/Digested By**	**Analyzed By**
350.1		SMEDBURY
351.2	KMENGS	GNITAJOUPPI
353.2		GNITAJOUPPI
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-BOT	**Date Collected:**	09/24/20
Lab Code:	R2008982-006	**Date Received:**	09/29/20
Sample Matrix:	Water		

Analysis Method	**Extracted/Digested By**	**Analyzed By**
350.1		SMEDBURY
351.2	KMENGS	GNITAJOUPPI
353.2		GNITAJOUPPI
SM 2540 D-1997(2011)		KAWONG

INORGANIC PREPARATION METHODS

The preparation methods associated with this report are found in these tables unless discussed in the case narrative.

Water/Liquid Matrix

Analytical Method	Preparation Method
200.7	200.2
200.8	200.2
6010C	3005A/3010A
6020A	ILM05.3
9034 Sulfide Acid Soluble	9030B
SM 4500-CN-E Residual Cyanide	SM 4500-CN-G
SM 4500-CN-E WAD Cyanide	SM 4500-CN-I

Solid/Soil/Non-Aqueous Matrix

Analytical Method	Preparation Method
6010C	3050B
6020A	3050B
6010C TCLP (1311) extract	3005A/3010A
6010 SPLP (1312) extract	3005A/3010A
7199	3060A
300.0 Anions/ 350.1/ 353.2/ SM 2320B/ SM 5210B/ 9056A Anions	DI extraction
For analytical methods not listed, the preparation method is the same as the analytical method reference.	

Sample Results

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

General Chemistry

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20 09:30
Sample Matrix:	Water	**Date Received:**	09/29/20 09:30
Sample Name:	FB01-SUR	**Basis:**	NA
Lab Code:	R2008982-001		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.010**	mg/L	0.010	1	10/07/20 19:01	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.075**	mg/L	0.050	1	10/05/20 15:23	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.24**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.17**	mg/L	0.10	1	10/09/20 13:12	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**5.8**	mg/L	1.0	1	09/29/20 12:25	NA	

ALS Group USA, Corp.

dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20 09:15
Sample Matrix:	Water	**Date Received:**	09/29/20 09:30
Sample Name:	FB01-MID	**Basis:**	NA
Lab Code:	R2008982-002		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.019**	mg/L	0.010	1	10/07/20 19:02	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.103**	mg/L	0.050	1	10/05/20 15:24	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.23**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.13**	mg/L	0.10	1	10/09/20 13:19	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**3.8**	mg/L	1.0	1	09/29/20 12:25	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20 08:50
Sample Matrix:	Water	**Date Received:**	09/29/20 09:30
Sample Name:	FB01-BOT	**Basis:**	NA
Lab Code:	R2008982-003		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.036**	mg/L	0.010	1	10/07/20 19:04	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.093**	mg/L	0.050	1	10/05/20 15:26	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.26**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.17**	mg/L	0.10	1	10/09/20 13:20	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**5.1**	mg/L	1.0	1	09/29/20 12:25	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20 10:35
Sample Matrix:	Water	**Date Received:**	09/29/20 09:30
Sample Name:	FB02-SUR	**Basis:**	NA
Lab Code:	R2008982-004		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/07/20 19:05	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.065**		mg/L	0.050	1	10/05/20 15:31	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.20**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.14**		mg/L	0.10	1	10/09/20 13:14	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**4.8**		mg/L	1.0	1	09/29/20 12:25	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20 10:20
Sample Matrix:	Water	**Date Received:**	09/29/20 09:30
Sample Name:	FB02-MID	**Basis:**	NA
Lab Code:	R2008982-005		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/07/20 19:07	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.089**		mg/L	0.050	1	10/05/20 15:33	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.21**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.12**		mg/L	0.10	1	10/09/20 13:15	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**3.5**		mg/L	1.0	1	09/29/20 12:25	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20 10:00
Sample Matrix:	Water	**Date Received:**	09/29/20 09:30
Sample Name:	FB02-BOT	**Basis:**	NA
Lab Code:	R2008982-006		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.016**	mg/L	0.010	1	10/07/20 19:12	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.099**	mg/L	0.050	1	10/05/20 15:34	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.29**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.19**	mg/L	0.10	1	10/09/20 11:35	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**4.9**	mg/L	1.0	1	09/29/20 12:25	NA	

QC Summary Forms

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

General Chemistry

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	NA
Sample Matrix:	Water	**Date Received:**	NA
Sample Name:	Method Blank	**Basis:**	NA
Lab Code:	R2008982-MB1		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/07/20 18:38	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/05/20 14:39	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	10/09/20 11:11	10/08/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	1.0	U	mg/L	1.0	1	09/29/20 12:25	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	NA
Sample Matrix:	Water	**Date Received:**	NA
Sample Name:	Method Blank	**Basis:**	NA
Lab Code:	R2008982-MB2		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/05/20 15:28	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	10/09/20 12:54	10/08/20	

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Collected:**	09/24/20
Sample Matrix:	Water	**Date Received:**	09/29/20
		Date Analyzed:	10/9/20
		Date Extracted:	10/8/20

Duplicate Matrix Spike Summary
Nitrogen, Total Kjeldahl (TKN)

Sample Name:	FB02-MID	**Units:**	mg/L
Lab Code:	R2008982-005	**Basis:**	NA
Analysis Method:	351.2		
Prep Method:	Method		

		Matrix Spike R2008982-005MS			**Duplicate Matrix Spike** R2008982-005DMS					
Analyte Name	**Sample Result**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
Nitrogen, Total Kjeldahl (TKN)	0.12	2.39	2.50	91	2.29	2.50	87 *	90-110	4	20

Results flagged with an asterisk (*) indicate values outside control criteria.

Results flagged with a pound (#) indicate the control criteria is not applicable.

Percent recoveries and relative percent differences (RPD) are determined by the software using values in the calculation which have not been rounded.

Matrix Spike and Matrix Spike Duplicate Data is presented for information purposes only. The matrix may or may not be relevant to samples reported in this report. The laboratory evaluates system performance based on the LCS and LCSD control limits.

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Analyzed:**	10/05/20 - 10/09/20
Sample Matrix:	Water		

Lab Control Sample Summary
General Chemistry Parameters

Units:mg/L
Basis:NA

Lab Control Sample
R2008982-LCS2

Analyte Name	Analytical Method	Result	Spike Amount	% Rec	% Rec Limits
Ammonia as Nitrogen	350.1	0.0902	0.100	90	90-110
Nitrate+Nitrite as Nitrogen	353.2	0.492	0.500	98	90-110
Nitrogen, Total Kjeldahl (TKN)	351.2	2.31	2.50	92	90-110

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Analyzed:**	10/05/20 - 10/09/20
Sample Matrix:	Water		

Lab Control Sample Summary
General Chemistry Parameters

Units:mg/L
Basis:NA

Lab Control Sample
R2008982-LCS3

Analyte Name	Analytical Method	Result	Spike Amount	% Rec	% Rec Limits
Nitrate+Nitrite as Nitrogen	353.2	0.503	0.500	101	90-110
Nitrogen, Total Kjeldahl (TKN)	351.2	2.46	2.50	98	90-110

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2008982
Project:	L2040456	**Date Analyzed:**	09/29/20
Sample Matrix:	Water		

Duplicate Lab Control Sample Summary
General Chemistry Parameters

Units:mg/L
Basis:NA

		Lab Control Sample R2008982-LCS1			Duplicate Lab Control Sample R2008982-DLCS1					
Analyte Name	**Analytical Method**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	183	214	85	182	214	85	80-120	<1	10

ANALYTICAL REPORT

Lab Number:	L2045732
Client:	Ransom Consulting, Inc. 112 Corporate Drive Pease International Tradeport Portsmouth, NH 03801
ATTN:	Elizabeth Ransom
Phone:	(603) 436-1490
Project Name:	Not Specified
Project Number:	201.05012.002
Report Date:	11/10/20

Certifications & Approvals: MA (M-MA086), NH NELAP (2064), CT (PH-0574), IL (200077), ME (MA00086), MD (348), NJ (MA935), NY (11148), NC (25700/666), PA (68-03671), RI (LAO00065), TX (T104704476), VT (VT-0935), VA (460195), USDA (Permit #P330-17-00196).

Eight Walkup Drive, Westborough, MA 01581-1019
508-898-9220 (Fax) 508-898-9193 800-624-9220 - www.alphalab.com

Project Name:	Not Specified	**Lab Number:**	L2045732
Project Number:	201.05012.002	**Report Date:**	11/10/20

Alpha Sample ID	Client ID	Matrix	Sample Location	Collection Date/Time	Receive Date
L2045732-01	FB01-BOT	WATER	MAINE	10/21/20 11:30	10/21/20
L2045732-02	FB01-MID	WATER	MAINE	10/21/20 11:35	10/21/20
L2045732-03	FB01-SUR	WATER	MAINE	10/21/20 11:50	10/21/20
L2045732-04	FB01-DUP	WATER	MAINE	10/21/20 11:50	10/21/20
L2045732-05	FB02-BOT	WATER	MAINE	10/21/20 09:15	10/21/20
L2045732-06	FB02-MID	WATER	MAINE	10/21/20 09:45	10/21/20
L2045732-07	FB02-SUR	WATER	MAINE	10/21/20 10:00	10/21/20
L2045732-08	FB02-DUP	WATER	MAINE	10/21/20 10:00	10/21/20

Case Narrative

The samples were received in accordance with the Chain of Custody and no significant deviations were encountered during the preparation or analysis unless otherwise noted. Sample Receipt, Container Information, and the Chain of Custody are located at the back of the report.

Results contained within this report relate only to the samples submitted under this Alpha Lab Number and meet NELAP requirements for all NELAP accredited parameters unless otherwise noted in the following narrative. The data presented in this report is organized by parameter (i.e. VOC, SVOC, etc.). Sample specific Quality Control data (i.e. Surrogate Spike Recovery) is reported at the end of the target analyte list for each individual sample, followed by the Laboratory Batch Quality Control at the end of each parameter. Tentatively Identified Compounds (TICs), if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported. If a sample was re-analyzed or re-extracted due to a required quality control corrective action and if both sets of data are reported, the Laboratory ID of the re-analysis or re-extraction is designated with an "R" or "RE", respectively.

When multiple Batch Quality Control elements are reported (e.g. more than one LCS), the associated samples for each element are noted in the grey shaded header line of each data table. Any Laboratory Batch, Sample Specific % recovery or RPD value that is outside the listed Acceptance Criteria is bolded in the report. In reference to questions H (CAM) or 4 (RCP) when "NO" is checked, the performance criteria for CAM and RCP methods allow for some quality control failures to occur and still be within method compliance. In these instances, the specific failure is not narrated but noted in the associated QC Outlier Summary Report, located directly after the Case Narrative. QC information is also incorporated in the Data Usability Assessment table (Format 11) of our Data Merger tool, where it can be reviewed in conjunction with the sample result, associated regulatory criteria and any associated data usability implications.

Soil/sediments, solids and tissues are reported on a dry weight basis unless otherwise noted. Definitions of all data qualifiers and acronyms used in this report are provided in the Glossary located at the back of the report.

HOLD POLICY - For samples submitted on hold, Alpha's policy is to hold samples (with the exception of Air canisters) free of charge for 21 calendar days from the date the project is completed. After 21 calendar days, we will dispose of all samples submitted including those put on hold unless you have contacted your Alpha Project Manager and made arrangements for Alpha to continue to hold the samples. Air canisters will be disposed after 3 business days from the date the project is completed.

Please contact Project Management at 800-624-9220 with any questions.

Case Narrative (continued)

Report Submission

November 10, 2020: This final report includes the results of all requested analyses.

November 09, 2020: This preliminary report includes the results of all requested subcontracted analyses.

October 28, 2020: This is a preliminary report.

The Nitrogen series and Total Suspended Solids analyses were subcontracted. A copy of the laboratory report is included as an addendum. Please note: This data is only available in PDF format and is not available on Data Merger.

All non-detect (ND) or estimated concentrations (J-qualified) have been quantitated to the limit noted in the MDL column.

I, the undersigned, attest under the pains and penalties of perjury that, to the best of my knowledge and belief and based upon my personal inquiry of those responsible for providing the information contained in this analytical report, such information is accurate and complete. This certificate of analysis is not complete unless this page accompanies any and all pages of this report.

Authorized Signature: Tiffani Morrissey - Tiffani Morrissey

Title: Technical Director/Representative Date: 11/10/20

INORGANICS
&
MISCELLANEOUS

Project Name:	Not Specified	**Lab Number:**	L2045732
Project Number:	201.05012.002	**Report Date:**	11/10/20

SAMPLE RESULTS

Lab ID:	L2045732-01	Date Collected:	10/21/20 11:30
Client ID:	FB01-BOT	Date Received:	10/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	15.		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.031		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 16:13	121,4500P-E	SD
Phosphorus, Orthophosphate	0.022		mg/l	0.005	0.001	1	-	10/23/20 01:12	121,4500P-E	AW

SAMPLE RESULTS

Lab ID:	L2045732-02	Date Collected:	10/21/20 11:35
Client ID:	FB01-MID	Date Received:	10/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	5.0		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.028		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 16:14	121,4500P-E	SD
Phosphorus, Orthophosphate	0.021		mg/l	0.005	0.001	1	-	10/23/20 01:12	121,4500P-E	AW

Project Name:	Not Specified	**Lab Number:**	L2045732
Project Number:	201.05012.002	**Report Date:**	11/10/20

SAMPLE RESULTS

Lab ID:	L2045732-03	Date Collected:	10/21/20 11:50
Client ID:	FB01-SUR	Date Received:	10/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	14.		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.028		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 16:15	121,4500P-E	SD
Phosphorus, Orthophosphate	0.020		mg/l	0.005	0.001	1	-	10/23/20 01:13	121,4500P-E	AW

Project Name:	Not Specified	**Lab Number:**	L2045732
Project Number:	201.05012.002	**Report Date:**	11/10/20

SAMPLE RESULTS

Lab ID:	L2045732-04	Date Collected:	10/21/20 11:50
Client ID:	FB01-DUP	Date Received:	10/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	9.5		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.024		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 16:16	121,4500P-E	SD
Phosphorus, Orthophosphate	0.018		mg/l	0.005	0.001	1	-	10/23/20 01:13	121,4500P-E	AW

SAMPLE RESULTS

Lab ID: L2045732-05
Client ID: FB02-BOT
Sample Location: MAINE

Date Collected: 10/21/20 09:15
Date Received: 10/21/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	6.7		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.041		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 16:16	121,4500P-E	SD
Phosphorus, Orthophosphate	0.027		mg/l	0.005	0.001	1	-	10/23/20 01:14	121,4500P-E	AW

SAMPLE RESULTS

Lab ID:	L2045732-06	Date Collected:	10/21/20 09:45
Client ID:	FB02-MID	Date Received:	10/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	11.		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.028		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 16:17	121,4500P-E	SD
Phosphorus, Orthophosphate	0.019		mg/l	0.005	0.001	1	-	10/23/20 01:14	121,4500P-E	AW

Project Name:	Not Specified	**Lab Number:**	L2045732
Project Number:	201.05012.002	**Report Date:**	11/10/20

SAMPLE RESULTS

Lab ID:	L2045732-07	Date Collected:	10/21/20 10:00
Client ID:	FB02-SUR	Date Received:	10/21/20
Sample Location:	MAINE	Field Prep:	Refer to COC
Sample Depth:			
Matrix:	Water		

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	9.3		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.021		mg/l	0.010	0.004	1	10/27/20 10:10	10/27/20 14:14	121,4500P-E	SD
Phosphorus, Orthophosphate	0.013		mg/l	0.005	0.001	1	-	10/23/20 01:14	121,4500P-E	AW

SAMPLE RESULTS

Lab ID: L2045732-08
Client ID: FB02-DUP
Sample Location: MAINE

Date Collected: 10/21/20 10:00
Date Received: 10/21/20
Field Prep: Refer to COC

Sample Depth:
Matrix: Water

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab										
Solids, Total Suspended	8.3		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
Phosphorus, Total	0.026		mg/l	0.010	0.004	1	10/27/20 10:10	10/27/20 14:16	121,4500P-E	SD
Phosphorus, Orthophosphate	0.013		mg/l	0.005	0.001	1	-	10/23/20 01:15	121,4500P-E	AW

Method Blank Analysis
Batch Quality Control

Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor	Date Prepared	Date Analyzed	Analytical Method	Analyst
General Chemistry - Westborough Lab for sample(s): 01-06 Batch: WG1425085-1										
Phosphorus, Total	ND		mg/l	0.010	0.004	1	10/22/20 11:15	10/22/20 15:49	121,4500P-E	SD
General Chemistry - Westborough Lab for sample(s): 01-08 Batch: WG1425417-1										
Phosphorus, Orthophosphate	0.001	J	mg/l	0.005	0.001	1	-	10/23/20 01:01	121,4500P-E	AW
General Chemistry - Westborough Lab for sample(s): 01-08 Batch: WG1426500-1										
Solids, Total Suspended	ND		mg/l	5.0	NA	1	-	10/26/20 13:15	121,2540D	AC
General Chemistry - Westborough Lab for sample(s): 07-08 Batch: WG1427034-1										
Phosphorus, Total	ND		mg/l	0.010	0.004	1	10/27/20 10:10	10/27/20 14:12	121,4500P-E	SD

Lab Control Sample Analysis

Batch Quality Control

Project Name: Not Specified
Project Number: 201.05012.002

Lab Number: L2045732
Report Date: 11/10/20

Parameter	LCS %Recovery	Qual	LCSD %Recovery	Qual	%Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-06 Batch: WG1425085-2								
Phosphorus, Total	101		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 01-08 Batch: WG1425417-2								
Phosphorus, Orthophosphate	93		-		90-110	-		
General Chemistry - Westborough Lab Associated sample(s): 01-08 Batch: WG1426500-2								
Solids, Total Suspended	90		-		80-120	-		
General Chemistry - Westborough Lab Associated sample(s): 07-08 Batch: WG1427034-2								
Phosphorus, Total	104		-		80-120	-		

Matrix Spike Analysis
Batch Quality Control

Project Name: Not Specified
Project Number: 201.05012.002
Lab Number: L2045732
Report Date: 11/10/20

Parameter	Native Sample	MS Added	MS Found	MS %Recovery	Qual	MSD Found	MSD %Recovery	Qual	Recovery Limits	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-08 QC Batch ID: WG1425417-4 QC Sample: L2045732-02 Client ID: FB01-MID												
Phosphorus, Orthophosphate	0.021	0.5	0.442	84		-	-		80-120	-		20
General Chemistry - Westborough Lab Associated sample(s): 07-08 QC Batch ID: WG1427034-3 QC Sample: L2045732-07 Client ID: FB02-SUR												
Phosphorus, Total	0.021	0.5	0.426	81		-	-		75-125	-		20

Lab Duplicate Analysis

Batch Quality Control

Project Name: Not Specified
Project Number: 201.05012.002

Lab Number: L2045732
Report Date: 11/10/20

Parameter	Native Sample	Duplicate Sample	Units	RPD	Qual	RPD Limits
General Chemistry - Westborough Lab Associated sample(s): 01-08 QC Batch ID: WG1425417-3 QC Sample: L2045732-03 Client ID: FB01-SUR						
Phosphorus, Orthophosphate	0.020	0.018	mg/l	11		20
General Chemistry - Westborough Lab Associated sample(s): 07-08 QC Batch ID: WG1427034-4 QC Sample: L2045732-07 Client ID: FB02-SUR						
Phosphorus, Total	0.021	0.024	mg/l	13		20

Sample Receipt and Container Information

Were project specific reporting limits specified? YES

Cooler Information

Cooler	***Custody Seal***
A	Present/Intact
B	Present/Intact

Container Information

Container ID	***Container Type***	***Cooler***	***Initial pH***	***Final pH***	***Temp deg C***	***Pres***	***Seal***	***Frozen Date/Time***	***Analysis(*)***
L2045732-01A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-01B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-01C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TOTAL NITROGEN(),SUB-TKN()
L2045732-01D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-01E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-01F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-02A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-02B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-02C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TKN(),SUB-TOTAL NITROGEN()
L2045732-02D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-02E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-02F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-03A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-03B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-03C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TKN(),SUB-TOTAL NITROGEN()
L2045732-03D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-03E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-03F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-04A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-04B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-04C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TKN(),SUB-TOTAL NITROGEN()

*Values in parentheses indicate holding time in days

Container Information

Container ID	Container Type	Cooler	Initial pH	Final pH	Temp deg C	Pres	Seal	Frozen Date/Time	Analysis(*)
L2045732-04D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-04E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-04F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-05A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-05B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-05C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TKN(),SUB-TOTAL NITROGEN()
L2045732-05D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-05E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-05F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-06A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-06B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-06C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TKN(),SUB-TOTAL NITROGEN()
L2045732-06D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-06E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-06F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-07A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-07B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-07C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TKN(),SUB-TOTAL NITROGEN()
L2045732-07D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-07E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-07F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)
L2045732-08A	Plastic 120ml unpreserved	A	7	7	5.3	Y	Present/Intact		OPHOS-4500(2)
L2045732-08B	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		TPHOS-4500(28)
L2045732-08C	Plastic 120ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NO3/NO2(),SUB-TOTAL NITROGEN(),SUB-TKN()
L2045732-08D	Plastic 500ml H2SO4 preserved	A	<2	<2	5.3	Y	Present/Intact		SUB-NH3(28)
L2045732-08E	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		SUB-TSS(7)
L2045732-08F	Plastic 950ml unpreserved	B	7	7	5.4	Y	Present/Intact		TSS-2540(7)

*Values in parentheses indicate holding time in days

Container Information

Container ID	Container Type	Cooler	Initial pH	Final pH	Temp deg C	Pres	Seal	Frozen Date/Time	Analysis(*)

*Values in parentheses indicate holding time in days

GLOSSARY

Acronyms

DL	- Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the limit of quantitation (LOQ). The DL includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
EDL	- Estimated Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The EDL includes any adjustments from dilutions, concentrations or moisture content, where applicable. The use of EDLs is specific to the analysis of PAHs using Solid-Phase Microextraction (SPME).
EMPC	- Estimated Maximum Possible Concentration: The concentration that results from the signal present at the retention time of an analyte when the ions meet all of the identification criteria except the ion abundance ratio criteria. An EMPC is a worst-case estimate of the concentration.
EPA	- Environmental Protection Agency.
LCS	- Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LCSD	- Laboratory Control Sample Duplicate: Refer to LCS.
LFB	- Laboratory Fortified Blank: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes.
LOD	- Limit of Detection: This value represents the level to which a target analyte can reliably be detected for a specific analyte in a specific matrix by a specific method. The LOD includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
LOQ	- Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.) Limit of Quantitation: The value at which an instrument can accurately measure an analyte at a specific concentration. The LOQ includes any adjustments from dilutions, concentrations or moisture content, where applicable. (DoD report formats only.)
MDL	- Method Detection Limit: This value represents the level to which target analyte concentrations are reported as estimated values, when those target analyte concentrations are quantified below the reporting limit (RL). The MDL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
MS	- Matrix Spike Sample: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. For Method 332.0, the spike recovery is calculated using the native concentration, including estimated values.
MSD	- Matrix Spike Sample Duplicate: Refer to MS.
NA	- Not Applicable.
NC	- Not Calculated: Term is utilized when one or more of the results utilized in the calculation are non-detect at the parameter's reporting unit.
NDPA/DPA	- N-Nitrosodiphenylamine/Diphenylamine.
NI	- Not Ignitable.
NP	- Non-Plastic: Term is utilized for the analysis of Atterberg Limits in soil.
NR	- No Results: Term is utilized when 'No Target Compounds Requested' is reported for the analysis of Volatile or Semivolatile Organic TIC only requests.
RL	- Reporting Limit: The value at which an instrument can accurately measure an analyte at a specific concentration. The RL includes any adjustments from dilutions, concentrations or moisture content, where applicable.
RPD	- Relative Percent Difference: The results from matrix and/or matrix spike duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). Values which are less than five times the reporting limit for any individual parameter are evaluated by utilizing the absolute difference between the values; although the RPD value will be provided in the report.
SRM	- Standard Reference Material: A reference sample of a known or certified value that is of the same or similar matrix as the associated field samples.
STLP	- Semi-dynamic Tank Leaching Procedure per EPA Method 1315.
TEF	- Toxic Equivalency Factors: The values assigned to each dioxin and furan to evaluate their toxicity relative to 2,3,7,8-TCDD.
TEQ	- Toxic Equivalent: The measure of a sample's toxicity derived by multiplying each dioxin and furan by its corresponding TEF and then summing the resulting values.
TIC	- Tentatively Identified Compound: A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations.

Report Format: DU Report with 'J' Qualifiers

Footnotes

1 - The reference for this analyte should be considered modified since this analyte is absent from the target analyte list of the original method.

Terms

Analytical Method: Both the document from which the method originates and the analytical reference method. (Example: EPA 8260B is shown as 1,8260B.) The codes for the reference method documents are provided in the References section of the Addendum.

Difference: With respect to Total Oxidizable Precursor (TOP) Assay analysis, the difference is defined as the Post-Treatment value minus the Pre-Treatment value.

Final pH: As it pertains to Sample Receipt & Container Information section of the report, Final pH reflects pH of container determined after adjustment at the laboratory, if applicable. If no adjustment required, value reflects Initial pH.

Frozen Date/Time: With respect to Volatile Organics in soil, Frozen Date/Time reflects the date/time at which associated Reagent Water-preserved vials were initially frozen. Note: If frozen date/time is beyond 48 hours from sample collection, value will be reflected in 'bold'.

Initial pH: As it pertains to Sample Receipt & Container Information section of the report, Initial pH reflects pH of container determined upon receipt, if applicable.

PAH Total: With respect to Alkylated PAH analyses, the 'PAHs, Total' result is defined as the summation of results for all or a subset of the following compounds: Naphthalene, C1-C4 Naphthalenes, 2-Methylnaphthalene, 1-Methylnaphthalene, Biphenyl, Acenaphthylene, Acenaphthene, Fluorene, C1-C3 Fluorenes, Phenanthrene, C1-C4 Phenanthrenes/Anthracenes, Anthracene, Fluoranthene, Pyrene, C1-C4 Fluoranthenes/Pyrenes, Benz(a)anthracene, Chrysene, C1-C4 Chrysenes, Benzo(b)fluoranthene, Benzo(j)+(k)fluoranthene, Benzo(e)pyrene, Benzo(a)pyrene, Perylene, Indeno(1,2,3-cd)pyrene, Dibenz(ah)+(ac)anthracene, Benzo(g,h,i)perylene. If a 'Total' result is requested, the results of its individual components will also be reported.

PFAS Total: With respect to PFAS analyses, the 'PFAS, Total (5)' result is defined as the summation of results for: PFHpA, PFHxS, PFOA, PFNA and PFOS. If a 'Total' result is requested, the results of its individual components will also be reported.

The target compound Chlordane (CAS No. 57-74-9) is reported for GC ECD analyses. Per EPA,this compound "refers to a mixture of chlordane isomers, other chlorinated hydrocarbons and numerous other components." (Reference: USEPA Toxicological Review of Chlordane, In Support of Summary Information on the Integrated Risk Information System (IRIS), December 1997.)

Total: With respect to Organic analyses, a 'Total' result is defined as the summation of results for individual isomers or Aroclors. If a 'Total' result is requested, the results of its individual components will also be reported. This is applicable to 'Total' results for methods 8260, 8081 and 8082.

Data Qualifiers

A	- Spectra identified as "Aldol Condensates" are byproducts of the extraction/concentration procedures when acetone is introduced in the process.
B	- The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank. For DOD-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than ten times (10x) the concentration found in the blank AND the analyte was detected above one-half the reporting limit (or above the reporting limit for common lab contaminants) in the associated method blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone).
C	- Co-elution: The target analyte co-elutes with a known lab standard (i.e. surrogate, internal standards, etc.) for co-extracted analyses.
D	- Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.
E	- Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.
F	- The ratio of quantifier ion response to qualifier ion response falls outside of the laboratory criteria. Results are considered to be an estimated maximum concentration.
G	- The concentration may be biased high due to matrix interferences (i.e, co-elution) with non-target compound(s). The result should be considered estimated.
H	- The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.
I	- The lower value for the two columns has been reported due to obvious interference.
J	- Estimated value. The Target analyte concentration is below the quantitation limit (RL), but above the Method Detection Limit (MDL) or Estimated Detection Limit (EDL) for SPME-related analyses. This represents an estimated concentration for Tentatively Identified Compounds (TICs).
M	- Reporting Limit (RL) exceeds the MCP CAM Reporting Limit for this analyte.
ND	- Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for SPME-related analyses.
NJ	- Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where

Data Qualifiers

the identification is based on a mass spectral library search.

P	- The RPD between the results for the two columns exceeds the method-specified criteria.
Q	- The quality control sample exceeds the associated acceptance criteria. For DOD-related projects, LCS and/or Continuing Calibration Standard exceedences are also qualified on all associated sample results. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)
R	- Analytical results are from sample re-analysis.
RE	- Analytical results are from sample re-extraction.
S	- Analytical results are from modified screening analysis.

REFERENCES

121 Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

LIMITATION OF LIABILITIES

Alpha Analytical, Inc. ID No.:**17873**
Facility: **Company-wide** Revision 17
Department: **Quality Assurance** Published Date: 4/28/2020 9:42:21 AM
Title: **Certificate/Approval Program Summary** Page 1 of 1

Certification Information

The following analytes are not included in our Primary NELAP Scope of Accreditation:

Westborough Facility
EPA 624/624.1: m/p-xylene, o-xylene, Naphthalene
EPA 8260C: NPW: 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene, Azobenzene; SCM: Iodomethane (methyl iodide), 1,2,4,5-Tetramethylbenzene; 4-Ethyltoluene.
EPA 8270D: NPW: Dimethylnaphthalene,1,4-Diphenylhydrazine; SCM: Dimethylnaphthalene,1,4-Diphenylhydrazine.
SM4500: NPW: Amenable Cyanide; SCM: Total Phosphorus, TKN, NO2, NO3.

Mansfield Facility
SM 2540D: TSS
EPA 8082A: NPW: PCB: 1, 5, 31, 87,101, 110, 141, 151, 153, 180, 183, 187.
EPA TO-15: Halothane, 2,4,4-Trimethyl-2-pentene, 2,4,4-Trimethyl-1-pentene, Thiophene, 2-Methylthiophene, 3-Methylthiophene, 2-Ethylthiophene, 1,2,3-Trimethylbenzene, Indan, Indene, 1,2,4,5-Tetramethylbenzene, Benzothiophene, 1-Methylnaphthalene.
EPA TO-12 Non-methane organics
EPA 3C Fixed gases
Biological Tissue Matrix: EPA 3050B

The following analytes are included in our Massachusetts DEP Scope of Accreditation

Westborough Facility:

Drinking Water
EPA 300.0: Chloride, Nitrate-N, Fluoride, Sulfate; **EPA 353.2**: Nitrate-N, Nitrite-N; **SM4500NO3-F**: Nitrate-N, Nitrite-N; **SM4500F-C, SM4500CN-CE, EPA 180.1, SM2130B, SM4500Cl-D, SM2320B, SM2540C, SM4500H-B, SM4500NO2-B**
EPA 332: Perchlorate; **EPA 524.2:** THMs and VOCs; **EPA 504.1:** EDB, DBCP.
Microbiology: **SM9215B; SM9223-P/A, SM9223B-Colilert-QT,SM9222D**.

Non-Potable Water
SM4500H,B, EPA 120.1, SM2510B, SM2540C, SM2320B, SM4500CL-E, SM4500F-BC, SM4500NH3-BH: Ammonia-N and Kjeldahl-N, **EPA 350.1**: Ammonia-N, **LACHAT 10-107-06-1-B**: Ammonia-N, **EPA 351.1, SM4500NO3-F, EPA 353.2**: Nitrate-N**, SM4500P-E, SM4500P-B, E, SM4500SO4-E, SM5220D, EPA 410.4, SM5210B, SM5310C, SM4500CL-D, EPA 1664, EPA 420.1, SM4500-CN-CE, SM2540D, EPA 300: Chloride, Sulfate, Nitrate.**
EPA 624.1: Volatile Halocarbons & Aromatics,
EPA 608.3: Chlordane, Toxaphene, Aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Dieldrin, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin Aldehyde, Heptachlor, Heptachlor Epoxide, PCBs
EPA 625.1: SVOC (Acid/Base/Neutral Extractables), **EPA 600/4-81-045**: PCB-Oil.
Microbiology: **SM9223B-Colilert-QT; Enterolert-QT, SM9221E, EPA 1600, EPA 1603.**

Mansfield Facility:

Drinking Water
EPA 200.7: Al, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Na, Ag, Ca, Zn. **EPA 200.8:** Al, Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Ni, Se, Ag, TL, Zn. **EPA 245.1** Hg.
EPA 522.

Non-Potable Water
EPA 200.7: Al, Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Sr, TL, Ti, V, Zn.
EPA 200.8: Al, Sb, As, Be, Cd, Cr, Cu, Fe, Pb, Mn, Ni, K, Se, Ag, Na, TL, Zn.
EPA 245.1 Hg.
SM2340B

For a complete listing of analytes and methods, please contact your Alpha Project Manager.

CHAIN OF CUSTODY

PAGE 1 OF 1

Date Rec'd in Lab: 10/22/20

ALPHA Job #: L2045732

ALPHA ANALYTICAL
World Class Chemistry

8 Walkup Drive
Westboro, MA 01581
Tel: 508-898-9220

320 Forbes Blvd
Mansfield, MA 02048
Tel: 508-822-9300

Project Information

Project Name:
Project Location: Maine
Project #: 201.05012.002
Project Manager: Elizabeth Ransom
ALPHA Quote #:

Report Information - Data Deliverables

☒ ADEx ☒ EMAIL

Billing Information

☐ Same as Client info PO #:

Client Information

Client: Ransom Consulting Inc.
Address: 400 Commercial St.
Portland ME 04101
Phone: 207.772.2891
Email: thomas.neilson@ransomenv.com

Regulatory Requirements & Project Information Requirements

☐ Yes ☒ No MA MCP Analytical Methods
☐ Yes ☒ No CT RCP Analytical Methods
☐ Yes ☒ No Matrix Spike Required on this SDG? (Required for MCP Inorganics)
☐ Yes ☒ No GW1 Standards (Info Required for Metals & EPH with Targets)
☐ Yes ☒ No NPDES RGP
☐ Other State /Fed Program ____________ Criteria ____________

Turn-Around Time

☒ Standard ☐ RUSH (only confirmed if pre-approved!)

Date Due:

Additional Project Information:

Ammonia, Nitrate/Nitrite and TKN to be subcontracted to ALS Labs.
TSS samples to be split with ALS labs.

ANALYSIS

SAMPLE INFO
Filtration
☒ Field
☐ Lab to do
Preservation
☐ Lab to do

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler Initials	VOC: ☐ 8260 ☐ 624 ☐ 524.2	SVOC: ☐ ABN ☐ PAH	METALS: ☐ MCP 13 ☐ MCP 14 ☐ RCP 15	METALS: ☐ RCRA5 ☐ RCRA8 ☐ PP13	EPH: ☐ Ranges & Targets ☐ Ranges Only	VPH: ☐ Ranges & Targets ☐ Ranges Only	☐ PCB ☐ PEST	TPH: ☐ Quant Only ☐ Fingerprint	total phosphate	orthophosphate	Ammonia	Nitrate/Nitrite	TKN	TSS	Sample Comments	TOTAL # BOTTLES
45732-01	FB01-BOT	10/21/20	11:30	SW	TGN									X	X	X	X	X	X		6
-02	FB01-MID	10/21/20	11:35	SW	TGN									X	X	X	X	X	X		6
-03	FB01-SUR	10/21/20	11:50	SW	TGN									X	X	X	X	X	X		6
-04	FB01-DUP	10/21/20	11:50	SW	TGN									X	X	X	X	X	X		6
-05	FB02-BOT	10/21/20	09:15	SW	TGN									X	X	X	X	X	X		6
-06	FB02-MID	10/21/20	09:45	SW	TGN									X	X	X	X	X	X		6
-07	FB02-SUR	10/21/20	10:00	SW	TGN									X	X	X	X	X	X		6
-08	FB02-DUP	10/21/20	10:00	SW	TGN									X	X	X	X	X	X		6
Container Type														P	P	P	P	P	P		
Preservative														D	A	A	D	D	A		

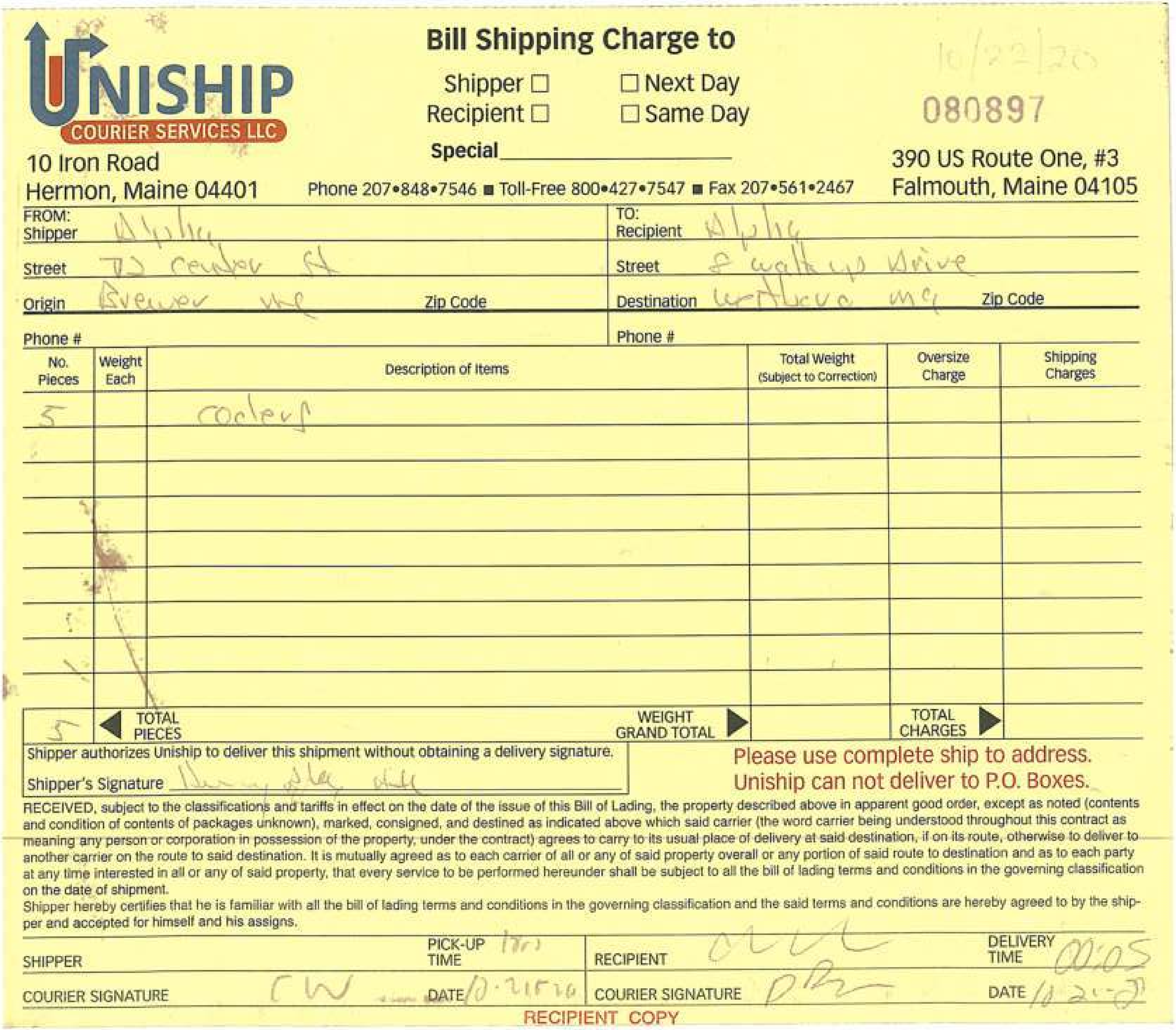
Container Type
P= Plastic
A= Amber glass
V= Vial
G= Glass
B= Bacteria cup
C= Cube
O= Other
E= Encore
D= BOD Bottle

Preservative
A= None
B= HCl
C= HNO_3
D= H_2SO_4
E= NaOH
F= MeOH
G= $NaHSO_4$
H = $Na_2S_2O_3$
I= Ascorbic Acid
J = NH_4Cl
K= Zn Acetate
O= Other

Relinquished By:	Date/Time	Received By:	Date/Time
Erik	10/21/20 16:05	AAL	10/21/20 16:15
AAL	10/21/20 18:00		10/21/20 18:00
			10/22/20 02:05

All samples submitted are subject to Alpha's Terms and Conditions. See reverse side.

FORM NO: 01-01 (rev. 12-Mar-2012)



UNISHIP
COURIER SERVICES LLC
10 Iron Road
Hermon, Maine 04401

Bill Shipping Charge to

Shipper ☐ ☐ Next Day
Recipient ☐ ☐ Same Day

Special ____________

10/22/20
080897

390 US Route One, #3
Falmouth, Maine 04105

Phone 207•848•7546 ■ Toll-Free 800•427•7547 ■ Fax 207•561•2467

FROM: Shipper Alpha
Street 72 Center St
Origin Brewer ME Zip Code
Phone #

TO: Recipient Alpha
Street 8 Walker Drive
Destination Winthrop ME Zip Code
Phone #

No. Pieces	Weight Each	Description of Items	Total Weight (Subject to Correction)	Oversize Charge	Shipping Charges
5		Coolers			
5	TOTAL PIECES	WEIGHT GRAND TOTAL		TOTAL CHARGES	

Shipper authorizes Uniship to deliver this shipment without obtaining a delivery signature.
Shipper's Signature

Please use complete ship to address.
Uniship can not deliver to P.O. Boxes.

RECEIVED, subject to the classifications and tariffs in effect on the date of the issue of this Bill of Lading, the property described above in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned, and destined as indicated above which said carrier (the word carrier being understood throughout this contract as meaning any person or corporation in possession of the property, under the contract) agrees to carry to its usual place of delivery at said destination, if on its route, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed as to each carrier of all or any of said property overall or any portion of said route to destination and as to each party at any time interested in all or any of said property, that every service to be performed hereunder shall be subject to all the bill of lading terms and conditions in the governing classification on the date of shipment.
Shipper hereby certifies that he is familiar with all the bill of lading terms and conditions in the governing classification and the said terms and conditions are hereby agreed to by the shipper and accepted for himself and his assigns.

SHIPPER	PICK-UP TIME 1800	RECIPIENT	DELIVERY TIME 00:05
COURIER SIGNATURE CW	DATE 10-21-20	COURIER SIGNATURE	DATE 10-21-20

RECIPIENT COPY

A

SCI SPEC PRECLEANED CONTAINERS Committed to Quality Since 1963

Custody Seal Custody Seal Custody Seal Custody Seal

DATE [illegible]

SIGNATURE

scispec.com

C20000

B

SCI SPEC PRECLEANED CONTAINERS Committed to Quality Since 1963

Custody Seal Custody Seal Custody Seal Custody Seal

DATE [illegible]

SIGNATURE

scispec.com

C20000

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2045732

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2045732

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB01-BOT	10-21-20 11:30	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-MID	10-21-20 11:35	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-SUR	10-21-20 11:50	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-DUP	10-21-20 11:50	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-BOT	10-21-20 09:15	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-MID	10-21-20 09:45	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	

Relinquished By:	Date/Time:	Received By:	Date/Time:
CTebeau	10/22/20		

Form No: AL_subcoc

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2045732

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2045732

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB02-SUR	10-21-20 10:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-DUP	10-21-20 10:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	

Relinquished By:	Date/Time:	Received By:	Date/Time:
C Jelrean	10/22/20		

Form No: AL_subcoc

November 09, 2020

Service Request No:R2010012

Melissa Gulli
Alpha Analytical Inc. (Westborough, MA)
8 Walkup Drive
Westborough, MA 01581

Laboratory Results for: L2045732

Dear Melissa,

Enclosed are the results of the sample(s) submitted to our laboratory October 23, 2020
For your reference, these analyses have been assigned our service request number **R2010012.**

All testing was performed according to our laboratory’s quality assurance program and met the requirements of the TNI standards except as noted in the case narrative report. Any testing not included in the lab's accreditation is identified on a Non-Certified Analytes report. All results are intended to be considered in their entirety. ALS Environmental is not responsible for use of less than the complete report. Results apply only to the individual samples submitted to the lab for analysis, as listed in the report. The measurement uncertainty of the results included in this report is within that expected when using the prescribed method(s), and represented by Laboratory Control Sample control limits. Any events, such as QC failures or Holding Time exceedances, which may add to the uncertainty are explained in the report narrative or are flagged with qualifiers. The flags are explained in the Report Qualifiers and Definitions page of this report.

Please contact me if you have any questions. My extension is 7472. You may also contact me via email at Janice.Jaeger@alsglobal.com.

Respectfully submitted,

ALS Group USA, Corp. dba ALS Environmental

Vicky Collom for:

Janice Jaeger
Project Manager

ADDRESS 1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
PHONE +1 585 288 5380 | FAX +1 585 288 8475
ALS Group USA, Corp.
dba ALS Environmental

Narrative Documents

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

Client: Alpha Analytical Inc. (Westborough, MA)
Project: L2045732
Sample Matrix: Water

Service Request: R2010012
Date Received: 10/23/2020

CASE NARRATIVE

All analyses were performed consistent with the quality assurance program of ALS Environmental. This report contains analytical results for samples for the Tier II level requested by the client.

Sample Receipt:

Eight water samples were received for analysis at ALS Environmental on 10/23/2020. Any discrepancies upon initial sample inspection are annotated on the sample receipt and preservation form included within this report. The samples were stored at minimum in accordance with the analytical method requirements.

General Chemistry:

No significant anomalies were noted with this analysis.

Approved by ______________________ Date 11/03/2020

SAMPLE DETECTION SUMMARY

CLIENT ID: FB01-BOT **Lab ID: R2010012-001**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrate+Nitrite as Nitrogen	0.077			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.18			0.1	mg/L	Calculation
Solids, Total Suspended (TSS)	4.7			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB01-MID **Lab ID: R2010012-002**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrate+Nitrite as Nitrogen	0.064			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.20			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.14			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	5.8			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB01-SUR **Lab ID: R2010012-003**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrogen, Total as Nitrogen	0.17			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.13			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	3.6			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB01-DUP **Lab ID: R2010012-004**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrogen, Total as Nitrogen	0.16			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.13			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	4.2			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-BOT **Lab ID: R2010012-005**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.018			0.010	mg/L	350.1
Nitrate+Nitrite as Nitrogen	0.082			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.26			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.18			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	5.7			1.1	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-MID **Lab ID: R2010012-006**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrate+Nitrite as Nitrogen	0.052			0.050	mg/L	353.2
Nitrogen, Total as Nitrogen	0.16			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.10			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	3.7			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-SUR **Lab ID: R2010012-007**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrogen, Total as Nitrogen	0.67			0.1	mg/L	Calculation

SAMPLE DETECTION SUMMARY

CLIENT ID: FB02-SUR **Lab ID: R2010012-007**

Analyte	Results	Flag	MDL	MRL	Units	Method
Nitrogen, Total Kjeldahl (TKN)	0.66			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	3.6			1.0	mg/L	SM 2540 D-1997 (2011)

CLIENT ID: FB02-DUP **Lab ID: R2010012-008**

Analyte	Results	Flag	MDL	MRL	Units	Method
Ammonia as Nitrogen	0.015			0.010	mg/L	350.1
Nitrogen, Total as Nitrogen	0.14			0.1	mg/L	Calculation
Nitrogen, Total Kjeldahl (TKN)	0.13			0.10	mg/L	351.2
Solids, Total Suspended (TSS)	2.9			1.0	mg/L	SM 2540 D-1997 (2011)

Sample Receipt Information

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

Client: Alpha Analytical Inc. (Westborough, MA) **Service Request:**R2010012
Project: L2045732

SAMPLE CROSS-REFERENCE

SAMPLE #	CLIENT SAMPLE ID	DATE	TIME
R2010012-001	FB01-BOT	10/21/2020	1130
R2010012-002	FB01-MID	10/21/2020	1135
R2010012-003	FB01-SUR	10/21/2020	1150
R2010012-004	FB01-DUP	10/21/2020	1150
R2010012-005	FB02-BOT	10/21/2020	0915
R2010012-006	FB02-MID	10/21/2020	0945
R2010012-007	FB02-SUR	10/21/2020	1000
R2010012-008	FB02-DUP	10/21/2020	1000

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2045732

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2045732

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB01-BOT	10-21-20 11:30	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-MID	10-21-20 11:35	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-SUR	10-21-20 11:50	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB01-DUP	10-21-20 11:50	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-BOT	10-21-20 09:15	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-MID	10-21-20 09:45	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	

Relinquished By:	Date/Time:	Received By:	Date/Time:
C Tebeau 10/	22/20		10/23/2020 0930

Form No: AL_subcoc

R2010012 5
Alpha Analytical Inc. (Westborough, MA)
L2045732

ALPHA ANALYTICAL
World Class Chemistry

Subcontract Chain of Custody

ALS Rochester
1565 Jefferson Road
Rochester, NY 14623

Alpha Job Number
L2045732

Client Information

Client: Alpha Analytical Labs
Address: Eight Walkup Drive
Westborough, MA 01581-1019

Phone: 603.319.5010
Email: mgulli@alphalab.com

Project Information

Project Location: ME
Project Manager: Melissa Gulli

Turnaround & Deliverables Information

Due Date:
Deliverables:

Regulatory Requirements/Report Limits

State/Federal Program:
Regulatory Criteria:

Project Specific Requirements and/or Report Requirements

Reference following Alpha Job Number on final report/deliverables: L2045732

Report to include Method Blank, LCS/LCSD:

Additional Comments: Send all results/reports to subreports@alphalab.com

Lab ID	Client ID	Collection Date/Time	Sample Matrix	Analysis	Batch QC
	FB02-SUR	10-21-20 10:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	
	FB02-DUP	10-21-20 10:00	WATER	Ammonia Nitrogen;NO3/NO2 combined analysis;Total Kjeldahl Nitrogen;Total Nitrogen;Total Suspended Solids 2540	

Relinquished By:	Date/Time:	Received By:	Date/Time:
C Delrean	10/22/20		10/23/20 0930

Form No: AL_subcoc

R2010012 - 5
Alpha Analytical Inc. (Westborough, MA)
L2045732

R2010012 5
Alpha Analytical Inc. (Westborough, MA)
L2046732

Cooler Receipt and Preservation Check Form

Project/Client Alpha Folder Number

Cooler received on 10/23/2020 by: @ **COURIER:** ALS [✓ UPS] FEDEX VELOCITY CLIENT

1	Were **Custody seals** on outside of cooler?	Y [✓ N]
2	**Custody papers** properly completed (ink, signed)?	[✓ Y] N
3	Did all bottles arrive in good **condition** (unbroken)?	[✓ Y] N
4	Circle: [✓ **Wet Ice**] **Dry Ice** **Gel packs** present?	[✓ Y] N

5a	**Perchlorate** samples have required headspace?	Y N [✓ NA]
5b	Did VOA vials, Alk,or Sulfide have sig* **bubbles**?	Y N [✓ NA]
6	Where did the bottles originate? ALS/ROC [✓ CLIENT]	
7	Soil VOA received as: Bulk Encore 5035set [✓ NA]	

8. Temperature Readings Date: 10/23/2020 Time: 0950 ID: IR#7 [✓ IR#10] From: ~~Temp Blank~~ [✓ Sample Bottle]

Observed Temp (°C)	3.8	3.0					
Within 0-6°C?	[✓ Y] N	[✓ Y] N	Y N	Y N	Y N	Y N	Y N
If <0°C, were samples frozen?	Y N	Y N	Y N	Y N	Y N	Y N	Y N

If out of Temperature, note packing/ice condition: ______ Ice melted Poorly Packed (described below) Same Day Rule

&Client Approval to Run Samples: ______ Standing Approval Client aware at drop-off Client notified by: ______

All samples held in storage location: R-002 by @ on 10/23/20 at 0956
5035 samples placed in storage location: ______ by ______ on ______ at ______ within 48 hours of sampling? Y N

Cooler Breakdown/Preservation Check**: Date : 10/23/2020 Time: 1630 by: AW

9. Were all bottle labels complete (*i.e.* analysis, preservation, etc.)? [✓ YES] NO
10. Did all bottle labels and tags agree with custody papers? [✓ YES] NO
11. Were correct containers used for the tests indicated? [✓ YES] NO
12. Were 5035 vials acceptable (no extra labels, not leaking)? YES NO [✓ N/A]
13. Air Samples: Cassettes / Tubes Intact Y / N with MS Y / N Canisters Pressurized Tedlar® Bags Inflated [✓ N/A]

pH	Lot of test paper	Reagent	Preserved?		Lot Received	Exp	Sample ID Adjusted	Vol. Added	Lot Added	Final pH
			Yes	No						
≥12		NaOH								
≤2		HNO_3								
≤2	223419	H_2SO_4	✓		client					
<4		$NaHSO_4$								
5-9		For 608pest			No=Notify for 3day					
Residual Chlorine (-)		For CN, Phenol, 625, 608pest, 522			If +, contact PM to add $Na_2S_2O_3$ (625, 608, CN), ascorbic (phenol).					
		$Na_2S_2O_3$								
		ZnAcetate	-	-						
		HCl	**	**						

**VOAs and 1664 Not to be tested before analysis. Otherwise, all bottles of all samples with chemical preservatives are checked (not just representatives).

Bottle lot numbers: client

Explain all Discrepancies/ Other Comments:

HPROD	BULK
HTR	FLDT
SUB	HGFB
ALS	LL3541

Labels secondary reviewed by: AW

PC Secondary Review: [illegible] 10/26/20 *significant air bubbles: VOA > 5-6 mm : WC >1 in. diameter

P:\INTRANET\QAQC\Forms Controlled\Cooler Receipt r18.doc 10/20/2020

Miscellaneous Forms

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

ALS Environmental

REPORT QUALIFIERS AND DEFINITIONS

U	Analyte was analyzed for but not detected. The sample quantitation limit has been corrected for dilution and for percent moisture, unless otherwise noted in the case narrative.
J	Estimated value due to either being a Tentatively Identified Compound (TIC) or that the concentration is between the MRL and the MDL. Concentrations are not verified within the linear range of the calibration. For DoD: concentration >40% difference between two GC columns (pesticides/Arclors).
B	Analyte was also detected in the associated method blank at a concentration that may have contributed to the sample result.
E	Inorganics- Concentration is estimated due to the serial dilution was outside control limits.
E	Organics- Concentration has exceeded the calibration range for that specific analysis.
D	Concentration is a result of a dilution, typically a secondary analysis of the sample due to exceeding the calibration range or that a surrogate has been diluted out of the sample and cannot be assessed.
*	Indicates that a quality control parameter has exceeded laboratory limits. Under the ñNotesò column of the Form I, this qualifier denotes analysis was performed out of Holding Time.
H	Analysis was performed out of hold time for tests that have an ñimmediateò hold time criteria.
#	Spike was diluted out.
+	Correlation coefficient for MSA is <0.995.
N	Inorganics- Matrix spike recovery was outside laboratory limits.
N	Organics- Presumptive evidence of a compound (reported as a TIC) based on the MS library search.
S	Concentration has been determined using Method of Standard Additions (MSA).
W	Post-Digestion Spike recovery is outside control limits and the sample absorbance is <50% of the spike absorbance.
P	Concentration >40% difference between the two GC columns.
C	Confirmed by GC/MS
Q	DoD reports: indicates a pesticide/Aroclor is not confirmed (Ó100% Difference between two GC columns).
X	See Case Narrative for discussion.
MRL LOQ	Method Reporting Limit. Also known as: Limit of Quantitation (LOQ) The lowest concentration at which the method analyte may be reliably quantified under the method conditions.
MDL	Method Detection Limit. A statistical value derived from a study designed to provide the lowest concentration that will be detected 99% of the time. Values between the MDL and MRL are estimated (see J qualifier).
LOD	Limit of Detection. A value at or above the MDL which has been verified to be detectable.
ND	Non-Detect. Analyte was not detected at the concentration listed. Same as U qualifier.

ACCREDITED IN ACCORDANCE WITH nelac

Rochester Lab ID # for State Certifications[1]

Connecticut ID # PH0556	Maine ID #NY0032	Pennsylvania ID# 68-786
Delaware Approved	New Hampshire ID # 2941	Rhode Island ID # 158
DoD ELAP #65817	New York ID # 10145	Virginia #460167
Florida ID # E87674	North Carolina #676	

[1] Analyses were performed according to our laboratoryôs NELAP-approved quality assurance program and any applicable state or agency requirements. The test results meet requirements of the current NELAP/TNI standards or state or agency requirements, where applicable, except as noted in the case narrative. Since not all analyte/method/matrix combinations are offered for state/NELAC accreditation, this report may contain results which are not accredited. For a specific list of accredited analytes, contact the laboratory or go to https://www.alsglobal.com/locations/americas/north-america/usa/new-york/rochester-environmental

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons
tr	Trace level is the concentration of an analyte that is less than the PQL but greater than or equal to the MDL.

ALS Group USA, Corp.
dba ALS Environmental

Analyst Summary report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732/		

Sample Name:	FB01-BOT	**Date Collected:**	10/21/20
Lab Code:	R2010012-001	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB01-MID	**Date Collected:**	10/21/20
Lab Code:	R2010012-002	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB01-SUR	**Date Collected:**	10/21/20
Lab Code:	R2010012-003	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB01-DUP	**Date Collected:**	10/21/20
Lab Code:	R2010012-004	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON

ALS Group USA, Corp.
dba ALS Environmental

Analyst Summary report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732/		

Sample Name:	FB01-DUP	**Date Collected:**	10/21/20
Lab Code:	R2010012-004	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-BOT	**Date Collected:**	10/21/20
Lab Code:	R2010012-005	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-MID	**Date Collected:**	10/21/20
Lab Code:	R2010012-006	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-SUR	**Date Collected:**	10/21/20
Lab Code:	R2010012-007	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI

ALS Group USA, Corp.
dba ALS Environmental

Analyst Summary report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732/		

Sample Name:	FB02-SUR	**Date Collected:**	10/21/20
Lab Code:	R2010012-007	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

Sample Name:	FB02-DUP	**Date Collected:**	10/21/20
Lab Code:	R2010012-008	**Date Received:**	10/23/20
Sample Matrix:	Water		

Analysis Method	Extracted/Digested By	Analyzed By
350.1		MROGERSON
351.2	GNITAJOUPPI	GNITAJOUPPI
353.2		MROGERSON
SM 2540 D-1997(2011)		KAWONG

INORGANIC PREPARATION METHODS

The preparation methods associated with this report are found in these tables unless discussed in the case narrative.

Water/Liquid Matrix

Analytical Method	Preparation Method
200.7	200.2
200.8	200.2
6010C	3005A/3010A
6020A	ILM05.3
9034 Sulfide Acid Soluble	9030B
SM 4500-CN-E Residual Cyanide	SM 4500-CN-G
SM 4500-CN-E WAD Cyanide	SM 4500-CN-I

Solid/Soil/Non-Aqueous Matrix

Analytical Method	Preparation Method
6010C	3050B
6020A	3050B
6010C TCLP (1311) extract	3005A/3010A
6010 SPLP (1312) extract	3005A/3010A
7199	3060A
300.0 Anions/ 350.1/ 353.2/ SM 2320B/ SM 5210B/ 9056A Anions	DI extraction
For analytical methods not listed, the preparation method is the same as the analytical method reference.	

Sample Results

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

General Chemistry

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

ALS Group USA, Corp.

dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 11:30
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB01-BOT	**Basis:**	NA
Lab Code:	R2010012-001		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:39	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.077**		mg/L	0.050	1	10/27/20 19:52	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.18**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	10/30/20 13:22	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**4.7**		mg/L	1.0	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 11:35
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB01-MID	**Basis:**	NA
Lab Code:	R2010012-002		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:43	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.064**		mg/L	0.050	1	10/27/20 19:53	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.20**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.14**		mg/L	0.10	1	10/30/20 13:23	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**5.8**		mg/L	1.0	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 11:50
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB01-SUR	**Basis:**	NA
Lab Code:	R2010012-003		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:44	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/27/20 19:54	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.17**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.13**		mg/L	0.10	1	10/30/20 13:23	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**3.6**		mg/L	1.0	1	10/28/20 08:40	NA	

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 11:50
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB01-DUP	**Basis:**	NA
Lab Code:	R2010012-004		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:45	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/27/20 19:56	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.16**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.13**		mg/L	0.10	1	10/30/20 13:24	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**4.2**		mg/L	1.0	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 09:15
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB02-BOT	**Basis:**	NA
Lab Code:	R2010012-005		

Inorganic Parameters

Analyte Name	Analysis Method	Result	Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.018**	mg/L	0.010	1	10/26/20 20:46	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.082**	mg/L	0.050	1	10/27/20 19:57	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.26**	mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.18**	mg/L	0.10	1	10/30/20 13:25	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**5.7**	mg/L	1.1	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 09:45
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB02-MID	**Basis:**	NA
Lab Code:	R2010012-006		

Inorganic Parameters

Analyte Name	**Analysis Method**	**Result**		**Units**	**MRL**	**Dil.**	**Date Analyzed**	**Date Extracted**	**Q**
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:47	NA	
Nitrate+Nitrite as Nitrogen	353.2	**0.052**		mg/L	0.050	1	10/27/20 19:59	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.16**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.10**		mg/L	0.10	1	10/30/20 13:26	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**3.7**		mg/L	1.0	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 10:00
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB02-SUR	**Basis:**	NA
Lab Code:	R2010012-007		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:48	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/27/20 20:00	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.67**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.66**		mg/L	0.10	1	10/30/20 13:27	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**3.6**		mg/L	1.0	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20 10:00
Sample Matrix:	Water	**Date Received:**	10/23/20 09:30
Sample Name:	FB02-DUP	**Basis:**	NA
Lab Code:	R2010012-008		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	**0.015**		mg/L	0.010	1	10/26/20 20:49	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/27/20 20:01	NA	
Nitrogen, Total as Nitrogen	Calculation	**0.14**		mg/L	0.1	1	NA	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	**0.13**		mg/L	0.10	1	10/30/20 13:32	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	**2.9**		mg/L	1.0	1	10/28/20 08:40	NA	

QC Summary Forms

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

General Chemistry

ALS Environmental—Rochester Laboratory
1565 Jefferson Road, Building 300, Suite 360, Rochester, NY 14623
Phone (585) 288-5380 Fax (585) 288-8475
www.alsglobal.com

ALS Group USA, Corp.
dba ALS Environmental

Analytical Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	NA
Sample Matrix:	Water	**Date Received:**	NA
Sample Name:	Method Blank	**Basis:**	NA
Lab Code:	R2010012-MB		

Inorganic Parameters

Analyte Name	Analysis Method	Result		Units	MRL	Dil.	Date Analyzed	Date Extracted	Q
Ammonia as Nitrogen	350.1	0.010	U	mg/L	0.010	1	10/26/20 20:15	NA	
Nitrate+Nitrite as Nitrogen	353.2	0.050	U	mg/L	0.050	1	10/27/20 19:31	NA	
Nitrogen, Total Kjeldahl (TKN)	351.2	0.10	U	mg/L	0.10	1	10/30/20 13:02	10/29/20	
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	1.0	U	mg/L	1.0	1	10/28/20 08:40	NA	

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Collected:**	10/21/20
Sample Matrix:	Water	**Date Received:**	10/23/20
		Date Analyzed:	10/26/20 - 10/30/20

Duplicate Matrix Spike Summary
General Chemistry Parameters

Sample Name:	FB02-DUP	**Units:**	mg/L
Lab Code:	R2010012-008	**Basis:**	NA

			Matrix Spike R2010012-008MS			**Duplicate Matrix Spike** R2010012-008DMS					
Analyte Name	**Method**	**Sample Result**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
Ammonia as Nitrogen	350.1	0.015	0.248	0.250	93	0.247	0.250	93	90-110	<1	20
Nitrogen, Total Kjeldahl (TKN)	351.2	0.13	2.23	2.50	84 *	2.42	2.50	92	90-110	8	20

Results flagged with an asterisk (*) indicate values outside control criteria.

Results flagged with a pound (#) indicate the control criteria is not applicable.

Percent recoveries and relative percent differences (RPD) are determined by the software using values in the calculation which have not been rounded.

ALS Group USA, Corp.
dba ALS Environmental

QA/QC Report

Client:	Alpha Analytical Inc. (Westborough, MA)	**Service Request:**	R2010012
Project:	L2045732	**Date Analyzed:**	10/26/20 - 10/30/20
Sample Matrix:	Water		

Lab Control Sample Summary
General Chemistry Parameters

Units:mg/L
Basis:NA

Lab Control Sample
R2010012-LCS

Analyte Name	Analytical Method	Result	Spike Amount	% Rec	% Rec Limits
Ammonia as Nitrogen	350.1	0.247	0.250	99	90-110
Nitrate+Nitrite as Nitrogen	353.2	0.539	0.500	108	90-110
Nitrogen, Total Kjeldahl (TKN)	351.2	2.38	2.50	95	90-110
Solids, Total Suspended (TSS)	SM 2540 D-1997(2011)	189	214	88	80-120

MANUAL

SHIPBOARD OIL POLLUTION EMERGENCY PLAN

FOR

MS MAR FORTUNE

- LEZI -
IMO 9267273

OWNER:

Amar Shipping III AS
Haugesund 123,
4262 Avaldsnes
Norway

Doc. No: 0435-109-001

Rev. No.		Date/Sign.
0	Issued for approval	

INDEX

EXPLANATORY NOTES

Regulation 37 of Annex I of MARPOL requires every oil tanker of 150 gt and above and every ship other than an oil tanker of 400 gt and above to carry a shipboard oil pollution emergency plan (SOPEP) approved by the Administration. Regulation 17 of Annex II of MARPOL, as amended by Resolution MEPC.137(53), requires every ship of 150gt and above that is certified to carry noxious liquid substances in bulk to carry on board a pollution emergency plan for noxious liquid substances not later than 1st January 2003. Ships to which both regulations apply may have a combined plan called a Shipboard Marine Pollution Emergency Plan, or SMPEP.

This model shipboard marine pollution emergency plan has been developed with the above requirements in mind. The purpose of a ship having such a plan is to give the master guidance on the action to be taken if a spill of oil or a noxious liquid substance occurs or is threatened, whether from an error during routine operations or after a major incident such as a collision, fire or explosion. The model plan is based on the SOPEP guidelines issued by IMO in resolution MEPC 54(32) dated 6th March 1992 as amended by resolution MEPC.86(44), and the SMPEP guidelines in resolution MEPC.85(44) dated 13th March 2000. A plan prepared according to this model will also satisfy the requirements for oil pollution emergency plans and relevant oil pollution reporting procedures that are contained in Articles 3 and 4, respectively, of the International Convention on Oil Pollution Preparedness, Response and Co-operation (OPRC), 1990.

The model is intended as the basis for the development of plans for individual ships. The text has therefore been devised so that relevant sections can be incorporated either in full or in part into ship-specific plans. [To assist compilers this model may be downloaded in electronic form for expansion as required.] [An outline of the plan, incorporating standard texts, is available on disk.]

A ship-specific plan will need to comprise extracts from existing company instructions and details of the particular ship's equipment and arrangements. Shaded sections in the model plan give guidance on factors to be considered, and/or indicate when the particular size, type or trade of a ship may influence the substance of the advice to be given. It should be noted that terms such as "pollution prevention team" or “marine pollution prevention team” have been used without shading where a reference to action by the ship is suggested. The precise contents of such sections of the ship's plan, and the terms to be used, are for individual decision by a company or on a ship, bearing in mind that the plan must be approved by the Administration.

The information and guidance in Section 5 of the model and in the Appendices are intended as guidance only. Companies may elect to adopt their own style and layout within the framework set out in the IMO Guidelines. Information in Section 5 or in the appendices may, for example, be transferred to Section 2 or 3 as long as the Administration so approves. The critical thing is that the actual plan should clearly outline the action to be taken and who must take it.

It will be apparent that ships other than tankers which do not carry oil or noxious liquid substances in bulk as cargo will not require the same extent of information that is likely to be applicable to tankers. A plan for ships other than tankers can accordingly be simple, incorporating relevant items from the model plan. However, it is important to acknowledge that, whether carried as cargo or bunkers, spilled oil can be environmentally damaging.

The List of Effective Pages, will need to be altered whenever there is an amendment or addition to the text, possibly by replacing the existing page if the plan is in loose-leaf format. An example of such a page after three changes is included in the model plan.

Further guidance on the compilation of shipboard oil or marine pollution emergency plans can be obtained from your national shipowners' association or your administration.

SHIPBOARD OIL POLLUTION
EMERGENCY PLAN

MS MAR FORTUNE

IMPORTANT

ANY SPILLAGE OF OIL OR NOXIOUS LIQUID SUBSTANCES SHOULD BE TREATED AS AN EMERGENCY

IT IS VITALLY IMPORTANT TO PREVENT ANY SPILLAGE OF BUNKERS OIL FROM FLOWING OVERBOARD

WHENEVER SAFETY CONSIDERATIONS PERMIT,
OIL OR NOXIOUS LIQUIDS SPILT ON DECK
SHOULD BE PREVENTED FROM FLOWING OVERBOARD.

EVERY CREW MEMBER HAS A RESPONSIBILITY
TO PREVENT POLLUTION

CONTENTS and LIST OF EFFECTIVE PAGES

Page No(s).		Change No.	Remarks
Name of Ship	5	Original	
Contents	6	Original	
Record of changes	7	Original	Reverse blank
*Ship's particulars	8-9	Original	
Regulatory requirements	10-11	Original	
*Introduction	12	Original	
*Preamble	Sec. 1	Original	
*Reporting requirements	Sec. 2	Original	
*Steps to control discharge	Sec. 3	Original	
*National and local co-ordination	Sec. 4	Original	
Additional information	Sec. 5	Original	
List of appendices	List	Original	
Appendix 1	Enclosure	Appendix 2	
Appendix 3		Original	
Appendix 4		Original	
Appendix 5		Original	Reverse blank
Appendix 6		Original	Reverse blank
Appendix 7		Original	
Appendix 8		Original	
Appendix 9		Original	Reverse blank
Appendix 10		Original	

- Note that changes to these pages are required to be approved by the Administration.

RECORD OF CHANGES

Change number	Date entered	Person entering	Position

SHIP'S PARTICULARS

Name : **Mar Fortune**

Type : **Multi Purpose Service Vessel**

Flag : **Norway**

No. Persons onboard : **10**

IMO : **9267273**

Call Sign : **LEZI**

Port of Registry : **Haugesund**

Builder : **Bollinger Shipyard, Lockport, LA, USA**

Year of Build : **2002**

Clasification Society : **Bureau Veritas**

Length (O.A.) : **41.00 m**

Breadth (Mld) : **11.00 m**

Depth (Mld) : **3.50 m**

Summenr Draft : **2.80 m**

Keel to top of mast : **16.40 m**

Side Thrusters fitted : **frame 53,5 & 8,5**

G.R.T. : **499**

MMSI : **257011630**

INMARSAT MES : **425900987**

VHF : **2-ICOM**

MF SSB : **Furuno**

HF SSB : **Furuno**

Oily water separator/filter provided : **Yes**

Oil Spill Clean up equipment

- Material and Quantity : **Saw dust, Oil Booms, Absorbent pads, Hand Broom, Plastic Bags, Rubber Gloves, Dust Pan, Safety Googles**
- Location : **Inside SOPEP drum**

OWNER: Amar Shipping III AS

ADDRESS:
Haugesund 123
4262 Avaldsnes
Norway

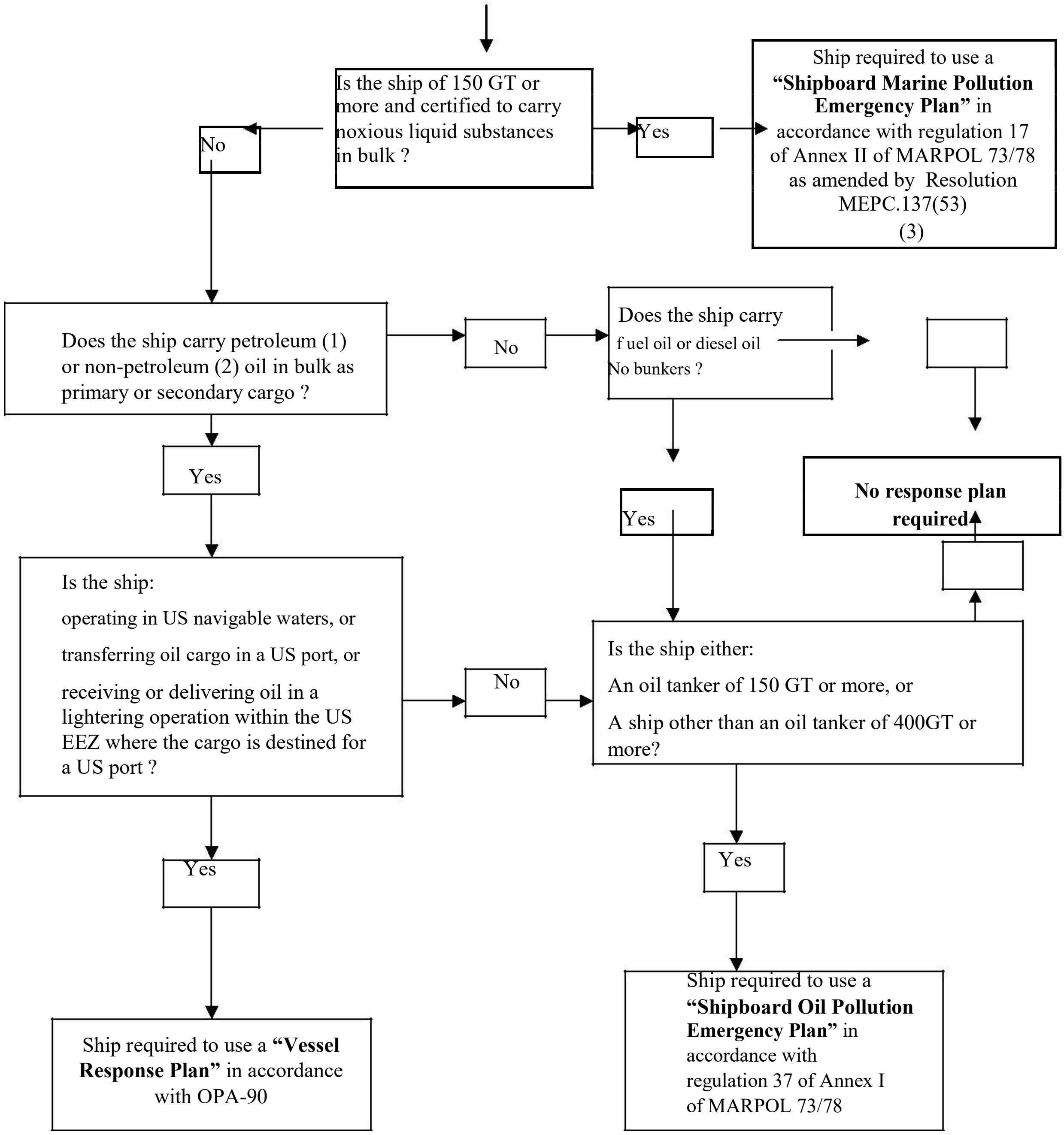
REGULATORY REQUIREMENTS

1. Regulation 37 of Annex I of MARPOL 73/78 requires every oil tanker of 150 tons gross tonnage and above, and regulation 17 of Annex II of MARPOL 73/78, as amended by Resolution MEPC.137(53), requires every ship of 150 tons gross tonnage and above certified to carry noxious liquid substances in bulk, to have a shipboard emergency plan with four elements:
   - procedures for reporting pollution incidents;
   - a listing of authorities to be notified;
   - a detailed description of actions to be taken by the ship's crew to reduce or control a discharge of oil or a noxious liquid substance; and
   - procedures for co-ordinating shipboard activities with national and local authorities.

2. Without interfering with shipowners' liability, some coastal States consider that it is their responsibility to define techniques and means to be taken against a marine pollution incident, and approve such operations which might cause further pollution. The plan required by MARPOL in the regulations referred to in paragraph 1 above, will not fully meet regulations in such States applicable to ships which carry oil in bulk. The USA is the notable example, and owners or operators of ships carrying oil as cargo in US waters must additionally:
   - identify and ensure, through contract or other approved means, the availability of private firefighting, salvage, lightering and clean-up resources;
   - identify a qualified individual with full authority to implement the response plan, including the activation and funding of contracted clean-up resources; and
   - describe training and drill procedures.

3. The following flow diagram should be used to ascertain whether the vessel has to be ready to put into effect the MARPOL "Shipboard Oil Pollution Emergency Plan" or the U.S. "Vessel Response Plan".

RESPONSE PLAN REQUIREMENTS



(1) Any petroleum based oil including, but not limited to, petroleum fuel oil, oil refuse and oil mixed with wastes other than dredged spoil.
(2) Any non-petroleum based oil, including but not limited to, animal and vegetable oils.
(3) It is expected that USA will make similar requirements for NLS transfers as now exist for oil transfers

Shipboard Marine Pollution Emergency Plan

INTRODUCTION

1. This plan is written in accordance with the requirements of regulation 37 of Annex I and/or regulation 17 of Annex II of the International Convention for the Prevention of Pollution from Ships, 1973, as modified by the Protocol of 1978 relating thereto (MARPOL 73/78), as amended by Resolution MEPC.137(53),
2. The purpose of the plan is to provide guidance to the master and officers on board the ship with respect to the steps to be taken when a pollution incident has occurred or is likely to occur.
3. The plan contains all information and operational instructions required by the Guidelines* The appendices contain names, telephone, telex numbers, etc., of all contacts referred to in the plan, as well as other reference material.
4. This plan has been approved by the Administration and, except as provided below, no alteration or revision shall be made to any part of it without the prior approval of the Administration.
5. Changes to Section 5 and the appendices will not be required to be approved by the Administration.

   The appendices should be maintained up to date by the owners, operators and managers.
6. The Technical Manager is responsible for regular review and update of the SMPEP.
   Updating shall be done not later than July 1st each year.
7. If the plan has been activated, there must be a thorough review of its effectiveness.

---

* "Guidelines for the development of Shipboard Marine Pollution Emergency Plans"; (IMO resolution MEPC.85(44); adopted on 13 March 2000.

SECTION 1 - PREAMBLE

1. This Shipboard Marine Pollution Emergency Plan is provided to assist personnel in dealing with an unexpected discharge of oil or a noxious liquid substance. Its primary purpose is to set in motion the necessary actions to stop or minimize the discharge and to mitigate its effects. Effective planning ensures that the necessary actions are taken in a structured, logical, safe and timely manner.
2. The plan makes use of flowcharts and checklists to guide the master through the various actions and decisions which will be required in an incident response. The charts and checklists provide a visible form of information, thus reducing the chance of oversight or error during the early stages of dealing with an emergency situation.
3. Extensive background information about the ship and its cargoes has been avoided, but for ready reference, tank plans, pipeline diagrams and capacity charts, with a general arrangement of the hull and upper deck, are appended to the plan.
4. The plan is designed to link into the Company's corporate plan for dealing with pollution emergencies; and the master will be backed up on-scene by management appointed personnel as the circumstances and the position of the vessel at the time of the incident, require.
5. For any plan to be effective it has to be:
    - familiar to those with key functions on board the ship;
    - evaluated, reviewed and updated regularly; and
    - tested for viability in regular practices.
6. Training and exercises in implementation of the shipboard mitigation procedures must be held at regular intervals. Similarly, exercises in the communications procedure will be necessary to verify that the Company's corporate plan is also effective.

SECTION 2 - REPORTING REQUIREMENTS

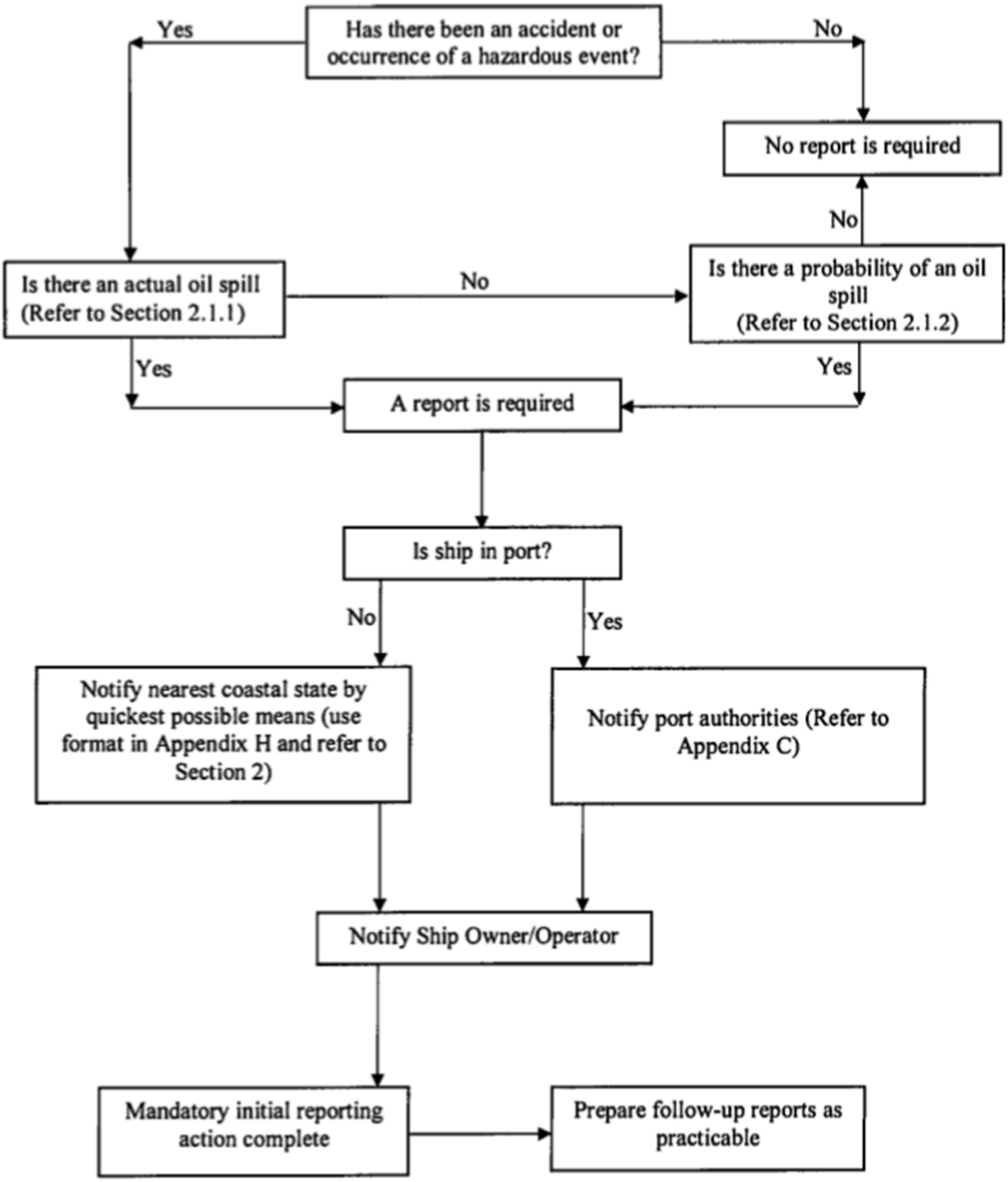
GENERAL

1. Article 8 and Protocol I of MARPOL 73/78 require that the nearest coastal state should be notified of actual or probable discharges of harmful substances to the sea. The intent of the requirement is to ensure that coastal states are informed without delay of any incident giving rise to pollution or threat of pollution of the marine environment, as well as the need for assistance and salvage measures, so that appropriate action may be taken.
2. The reporting procedure to be followed by the master or other person in charge of the ship after a pollution incident involving oil or noxious liquid substances is based on guidelines developed by the International Maritime Organization.*
3. If the ship is involved in a pollution incident, reports must be made both to coastal state or port contacts as appropriate, and to contacts representing interest in the ship.
4. A flow chart indicating the reporting procedure to be followed in accordance with the MARPOL requirements is given overleaf.
5. Some coastal states consider that it is their responsibility to define techniques and means to be taken against a marine pollution incident, and to approve such operations which might cause further pollution, e.g. lightening. The United States of America is the notable example of this.

---

* "General principles for ship reporting system and ship reporting requirements, including Guidelines for reporting incidents involving dangerous goods, harmful substances and/or marine pollutants" adopted by the International Maritime Organization by resolution A.851(20), as amended by MEPC.138(53)

REPORTS AS PRACTICABLE



SECTION 2.1: WHEN TO REPORT

2.1.1 Actual discharge.

A report is required whenever there is:

- a discharge of oil or noxious liquid substances resulting from damage to the ship or its equipment; or
- an intentional discharge for the purpose of securing the safety of a ship or saving life at sea; or
- during the operation of the ship there is a discharge of oil or a noxious liquid substance in excess of the quantity or instantaneous rate permitted under applicable marine pollution regulations.

Reports to coastal states should be in the style given in Section 2.2.

2.1.2 Probable discharge.

Although an actual discharge may not have occurred, a report is required if there is the probability of a discharge.

In judging whether there is such a probability, and thus whether a report must be made, the following factors should be taken into account:

- the nature of damage sustained by the ship;
- failure or breakdown of machinery or equipment which may adversely affect the ability of the ship to manoeuvre, operate pumps, etc.;
- the location of the ship and its proximity to land or other navigational hazards;
- present weather, tide, current and sea state;
- expected weather conditions;
- traffic density;
- morale, health and ability of the crew on board to deal with the situation.

As a general guide the master should make a report in cases of:

- damage, failure or breakdown which affects the safety of the ship or other shipping: examples of such situations are collision, grounding, fire, explosion, structural failure, flooding, cargo shifting;
- failure or breakdown of machinery or equipment which results in impairment of the safety of navigation: examples are breakdown of steering gear, propulsion, electrical generating system, essential shipborne navigational aids.

Follow Up Reports

Once the vessel has transmitted an initial report, further reports should be sent at regular intervals to keep those concerned informed of developments.

Follow up reports to coastal states should always be in the style given in Section 2.2, and should include information about every significant change in the vessel's condition, the rate of the release and spread of oil or noxious liquid substance, weather conditions, and details of agencies notified and clean-up activities.

SECTION 2.2: INFORMATION REQUIRED

This section should contain details of the information required for the initial report and for supplementary or follow up reports. An example report form should be included in the plan.

Content of Reports

The format and content of an initial report are given below. The format is consistent with the General Principles for Ship Reporting Systems and Ship Reporting Requirements, including Guidelines for Reporting Incidents Involving Dangerous Goods, Harmful Substances and/or Marine Pollutants, adopted as Resolution A.851(20), as amended by MEPC.138(53) by the International Maritime Organization (IMO), and should be followed so far as possible. (Note: The reference letters in the listing below do not follow the complete alphabetical sequence as certain letters are allocated to information required for other reporting formats).

The report should contain the following information:

A. Name of ship, call sign and flag.

B. Date and time (UTC, formerly known as GMT) of incident: a 6-digit group giving day of month (first two digits), hours and minutes (last four digits).

Either

C. Ship's position, giving latitude: a 4-digit group in degrees and minutes suffixed with N (North) or S (South); and longitude: a 5-digit group in degrees and minutes suffixed with E (East) or W (West);

Or

D. Ship's position by true bearing (first 3 digits) and distance (stated) from a clearly identified landmark.

E. True course (as a 3-digit group).

F. Speed (in knots and tenths of a knot as a 3-digit group).

L. Route information - details of intended track.

M. Full details of radio stations and frequencies being guarded.

N. Time of next report (a 6-digit group as in B).

O. Draught (a 4-digit group giving draught in metres and centimetres).

P. Types and quantities of cargo and bunkers on board. For ships carrying many different types of bulk liquid cargoes, it may be sufficient for the initial report to indicate those of prime importance, and the total amount. Follow up reports can identify the types and their location on board.

Q. Brief details of defects, damage, deficiencies or other limitations. These must include the condition of the ship and the ability to transfer cargo, ballast, or fuel.

R. Brief details of actual pollution. This should include the type of oil or noxious liquidsubstance, an estimate of the quantity discharged, whether the discharge is continuing, the cause of the discharge and, if possible, an estimate of the movement of the slick.For noxious liquid substances, the UN number (where known) should be given, and the MARPOL pollution category as listed in the IBC Code.

S. Weather and sea condition, including wind force and direction and relevant tidal or current details.

T. Name, address, telex, facsimile and telephone numbers of the ship's owner or representative (manager or operator of the ship, or their agents).

U. Details of length, breadth, tonnage and type of ship.

V. Total number of persons onboard.

W. Miscellaneous - to include relevant details including, as appropriate:

- Brief details of incident.
- Names of other ships involved.
- Action taken with regard to the discharge and movement of the ship.
- Assistance or salvage resources which have been requested or provided.
- Personnel injuries sustained.
- Whether medical assistance is required.

If no outside assistance is required, this should be clearly stated.

Reports should be transmitted by the quickest available means to the responsible authorities of the nearest coastal state or the Rescue Co-ordination Centre (RCC) via the appropriate shore radio station. If the ship is within or near to an area for which a ship reporting system has been established, reports should be transmitted to the designated shore station of that system.

The following additional information should be sent to the owner or operator either at the same time as the initial report or as soon as possible thereafter:

- Further details of damage to ship and equipment.
- Whether damage is still being sustained.
- Assessment of fire risk and precautions taken.
- Disposition of cargo on board and quantities involved.
- Number of casualties.
- Damage to other ships or property.
- Time (UTC) assistance was requested and time (UTC) assistance expected to arrive at the scene.
- Name of salvor and type of salvage equipment.
- Whether further assistance is required.
- Priority requirements for spare parts and other materials.
- Details of outside parties advised or aware of the incident.
- Any other important information.

For ships carrying noxious liquid substances, it may be found useful to report other information such as the soundness of empty tanks or spaces, the nature of any ballast on board, and the reliability of power generation for the cargo containment system, main propulsion and crew environment.

After transmission of the information in an initial report, as much as possible of the information essential for the safeguarding of life and the protection of the ship and the marine environment should be reported in a supplementary report to the coastal state and the owner or operator, in order to keep them informed of the situation as the incident develops. This information should include items P, Q, R, S and X, as appropriate.

Examples of initial reports follow, together with an example of a format which may be found suitable for direct transmission.

EXAMPLE REPORTS

The following is an example of an initial report sent to the government of the coastal State and to the owner or operator:

AA	WHITEGOLD CALL SIGN..XXXX...XX FLAG
BB	291150
CC	2230N 06000E
EE	137
FF	120
LL	BOUND SINGAPORE FROM RAS TANURA
MM	BAHRAIN RADIO 500KHZ, VHF 16, INMARSAT NO. 888888
NN	AS REQUIRED
OO	1700
PP	18,000 TONNES VARIOUS OIL PRODUCTS, MARPOL ANNEX I. 5,300 TONNES BENZENE, MARPOL ANNEX II, UN NUMBER 1114, POLLUTION CATEGORY C. FULL CARGO DISPOSITION WILL FOLLOW.
QQ	COLLISION WITH CARGO SHIP BLUE SKY. TANK 6 PORT BREACHED. SUBSEQUENT FIRE ON MAIN DECK. FIRE NOW EXTINGUISHED. CARGO PUMPS OPERATIVE BUT UNABLE TO TRANSFER CARGO DUE TO FULL TANKS. BALLAST SYSTEM INOPERATIVE.
RR	ESTIMATE 250 TONNES OF MOTOR GASOLINE LOST FROM 6 PORT. OUTFLOW NOW STOPPED APART FROM SEA ACTION. NO NLS LOST. ESTIMATE OF SLICK MMSEMENT AND AREA NOT POSSIBLE.
SS	WEATHER FINE. WIND SE FORCE 3. SEA SLIGHT.
TT	SHIP OPERATOR WHITE GOLD SHIPPING CO., XXXX, XXXXXX, XXXXXX TELEPHONE: +... ... . FAX: +... ... .
UU	LENGTH 160M. BREADTH 24M. TONNAGE 24,000 DWT. TYPE CHEMICAL CARRIER
WW	25
XX	TUG ABC 2 CONTRACTED TO ASSIST ETA 291600. NO SERIOUS INJURIES. DO NOT ANTICIPATE FURTHER ASSISTANCE REQUIRED.

The following is an example of additional information for the owner or operator:

QQ TANK 6 PORT BREACHED FROM DECK TO 1 METRE ABOVE WATER. SHIP LISTED 5 DEGREES STARBOARD. BOILER OUT OF SERVICE. HOWEVER ANTICIPATE WILL RESTORE TO SERVICE APPROX. 8 HOURS. GENERATORS OK.

NO FURTHER DAMAGE. FIRE RISK UNCERTAIN. CONTINUOUS WATER COOLING ON TANK 6 PORT CARGO TANKS INERTED WHERE POSSIBLE.

RR CARGO DISPOSITION MOTOR GASOLINE IN ONE, TWO, FOUR AND SIX ACROSS, THREE AND FIVE WINGS. BENZENE THREE AND FIVE CENTRES. ALL TANKS FULL.

XX THREE NON SERIOUS INJURIES (NAMES). DAMAGE TO COASTER BLUE SKY UNKNOWN. HOWEVER SHE ADVISES NOT IN DANGER OF SINKING.

TUG ABC 2 ETA REMAINS 1600 UTC. LOF 90 AGREED. DO NOT
ANTICIPATE FURTHER ASSISTANCE REQUIRED.
WILL REVERT WITH SPARES/MATERIALS REQUIREMENTS. SELF AND
WHITE SKY HAVE BROADCAST VHF PAN MESSAGE. COASTAL STATE
ADVISED.
UNDERSTAND LOCAL COAST GUARD ARRANGING AERIAL SPRAYING. NO
OTHER INFORMATION.

EXAMPLE FORMAT FOR INITIAL REPORT BY FACSIMILE

AA Name

Call sign and Flag:

BB Date and time (UTC) __ __ __ __ __ __
D D H H M M

Either:

CC. Position (Latitude and longitude)

__ __ __ __ __ __ __ __ __ __ __
d d m m N/S d d d m m E/W

or:

DD Position (Bearing and distance from landmark)

Brg. __ __ __ºT, Dist. __ __ __ miles from ……………………………………….

EE Course. __ __ __ ºT **FF** Speed. __ __ **.** __ Knots

LL Route information.

MM

NN Time of next report __ __ __ __ __ __
D D H H M M

OO Draught __ __ **.** __ __
M M cm cm

PP Cargo and bunkers.

QQ Defects, damage, deficiencies, limitations.

RR Actual pollution.

SS Weather and sea conditions.

Wind direction: ………….. force …………….

Swell height: ……………. Tide data: ……………………………………….

TT Owner's contact details.

UU Ship details.

Length: (m); Breadth: (m); Tonnage: Ship type:

XX Other details.

SECTION 2.3: WHOM TO CONTACT

NATIONAL CONTACTS FOR COASTAL STATES (Coastal State Contacts)

In order to expedite response and minimise damage from a pollution incident, it is essential that appropriate coastal states are notified without delay. This process is begun with the initial report. Guidelines for compiling reports are provided in Section 2.2.

This plan includes as Appendix 1 a list of agencies or officials of administrations responsible for receiving and processing reports. In the absence of a listed focal point, or where the responsible authority cannot be contacted by direct means without delay, the master should contact the nearest coast radio station, designated ship movement reporting station or Rescue Co-ordination Centre (RCC) by the quickest available means.

The latest list of contact points, can be obtained from the IMO Internet site*:* *"http://www.imo.org". On the Home Page, select "Circulars" from the left column and on the Circulars Page select "Contact Points" from the right column.* ***MEPC.6/Circ.7****"*

Whenever the ship is in United States' waters, an incident must be reported immediately to the US National Response Centre (NRC), regardless of whether or not the incident has been or will be reported to the US Coast Guard. 24-hour telephone numbers for the NRC are:

1 800 424 8802 or 202 267 2675.

It will be necessary for actual telephone, according to the coast earth station used.

Name	Address	Telephone Nos.
FLAG STATE CONTACT POINTS FOR PORT STATE CONTROL		
As a Flag State for PSC matters	The Norwegian Maritime Directorate Inspection Department P.O. Box 2222 Haugesund 5509	Tel: +47 52745000 Fax : +47 52745001 Email:postmottak@sjofartsdir.no
Ship Inspection Head Office	The Norwegian Maritime Directorate Inspection Department P.O. Box 2222 Haugesund 5509	Tel: +47 52745000 Fax : +47 52745001 Email:knut.kristiansen@sjofartsdir.no
NATIONAL OPERATIONAL CONTACT POINTS FROM SHIP TO COASTAL STATES		
-	Kystverket/Norwegian Coastal Administration Department of Emergency and Response Po Box 125, N-3191 Horten	Tel: +47 33034800 Fax : +4733034949 Email: vakt@kystverkt.no
CONTACT ADDRESSES OF RESPONSIBLE NATIONAL AUTHORITIES		
-	The Norwegian Maritime Directorate Inspection Department P.O. Box 2222 Haugesund 5509	Tel: +47 52745000 Fax : +47 52745001 Email:knut.kristiansen@sjofartsdir.no

PORT CONTACTS

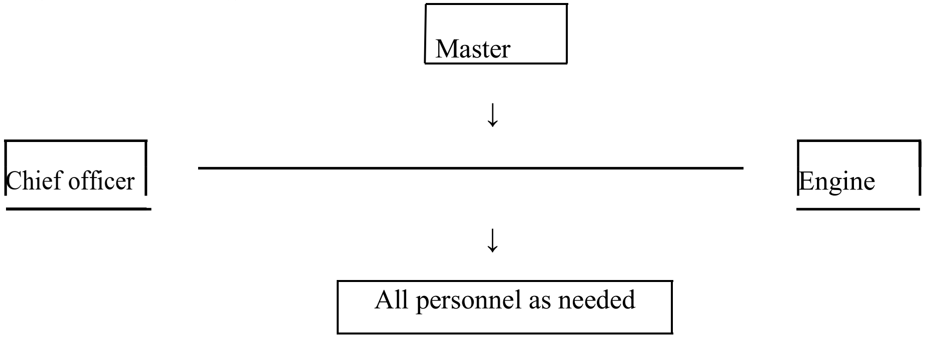
Notification of local agencies will speed response. Information on regularly visited ports is included in **Appendix 2**. The master had to obtain details concerning local reporting procedures upon arriving in port.

If a spill occurs when the vessel is in port, whatever the cause, it is the master's duty immediately to activate the vessel's Pollution Prevention Team and report the incident. Precise details of whom to notify locally should be obtained on arrival, but the following is a guide:

- Terminal/loading master
- Local fire department (in case of explosion and / or fire)
- Agent
- Port Authority
- The vessel's local P&I representative (P&I Club List of Correspondents is filed in.....).
- Owner/manager.
- Charterer.
- Clean-up contractor.

(A pro-forma for listing port contacts is shown at Appendix 2.)

SHIP INTEREST CONTACTS



In the event of an oil spill, the team should be called out immediately.

The team should be given the necessary training in the use of such equipment or oil absorbents as the vessel may carry. All members of the Oil Pollution Prevention Team should be aware of their duties should an oil spill occur.

PROPOSED INSTRUCTIONS TO OIL POLLUTION PREVENTION TEAM

Master: In overall charge.
Inform terminal authorities of incidents.
Inform local agent and request agent to inform the local P& I Club representative.
Advise the company's head office of the situation. Keep everyone updated at
regular intervals and advise any changes in status of the emergency.
Request assistance as deemed necessary.

Chief Officer: In charge of deck operation. Keep master informed and updated on the situation and of officer: the results of steps taken to limit outflow.

Engineer: In charge of bunker operations.
Organize distribution of oil spill detergent.

A list of ship interest contacts is detailed in Appendix 3, which also indicates the order of priority for notifying those concerned. Out-of-hours contacts are included.

All further reports and copies of messages sent to coastal states and/or port authorities should be sent to the company. If required, the company's office will be staffed as soon as possible after receipt of an initial report.

Once initial reports have been made, the company's corporate plan will ensure that other interests such as flag State authorities, P & I Club and classification society are notified and kept up to date on the incident.

SECTION 3 - STEPS TO CONTROL DISCHARGE

WHENEVER A SPILL OF OIL OR A NOXIOUS LIQUID SUBSTANCE OCCURS IT IS THE DUTY OF THE PERSON FINDING THE SPILL TO IMMEDIATELY INFORM THE MASTER OR RESPONSIBLE OFFICER, WHO SHOULD CALL OUT THE VESSEL'S POLLUTION PREVENTION TEAM (See Appendix 4). REMEMBER THAT AN OIL SPILL MAY CREATE A FIRE OR EXPLOSION HAZARD, REQUIRING SAFETY PRECAUTIONS TO BE OBSERVED. SPILLAGE OF A NOXIOUS LIQUID SUBSTANCE MAY CREATE THE SAME HAZARD, OR ADDITIONAL HAZARDS OF TOXICITY, CORROSIVENESS OR REACTIVITY, REQUIRING APPROPRIATE PRECAUTIONS.

THIS PLAN IS DIRECTED AT POLLUTION CONTROL, AND IT IS NOT APPROPRIATE TO BURDEN IT UNNECESSARILY BY REPEATING GENERAL SHIP OR COMPANY SAFETY PROCEDURES.

STEPS TO CONTROL DISCHARGE

This section is intended to ensure that the provisions of Regulation 37 of MARPOL 73/78 Annex I regarding steps to control any discharge of oil are complied with.

The ships personnel will almost always be in the best position to take quick action to mitigate or control the discharge of oil from the ship. This Plan provides the master with clear guidance on how to accomplish this mitigation for a variety of situations. Reference should also be made to Section 6 for response team duties and checklists for use in emergencies.

SECTION 3.1: OPERATIONAL SPILLS OF OIL OR NOXIOUS LIQUID SUBSTANCES

The most likely operational spill will result from:

- 3.1.1. Pipeline leakages, including transfer hoses
- 3.1.2. Cargo tank or bunker tank overflows
- 3.1.3. Hull leakages

3.1.1 Pipeline Leakage During Discharging or Loading of Oil or Noxious Liquid Substance Cargoes, or During Bunkering

1. Have all bunker operations been stopped?

2. Have all valves including isolating valves been closed?

3. Has agreed reporting procedure been followed?

4. Has the effected bunker line been drained to an available empty or slack tank?

5. Resist temptations to drop lines back to pump room bilges and anly resort to this course of action if all other spaces are full. In this instance full consideration should be given to safety implications.

6. Has oil pollution equipment been utilized and operated in accordance with the manufacturer's instructions, to prevent oil on deck being spilled overboard?

SPILLED OIL SHOULD NEVER BE WASHED OVERBOARD, NOR SHOULD DISPERSANTS OR DEGREASER BE USED ON OIL SPILLED OVERBOARD WITHOUT THE PERMISSION OF THE HARBOUR AUTHORITIES.

7. If the leakage is suspected from the pump room sea valves:

   7.1 Has any pressure in the relevant section of pipe been relieved?

   7.2 Have the sea valves and isolating valves been firmly closed?

   7.3 Have sections of pipe to the sea valve been stripped by drainage, purging or use of a stripping pump or eductor and transferred to a slop tank?

   7.4 Has water been introduced into the section of pipe leading to the sea valve in order to evacuate oil?

8. Has oil remaining on deck been removed using all means available?

9. If necessary, has use of the services of a clean-up company, if available, been made?

NO ACTION SHOULD BE TAKEN WHICH COULD IN ANY WAY JEOPARDIZE THE SAFETY OF THE SHIP OR PERSONNEL EITHER ONBOARD OR ASHORE.

The use of a simple check list is recommended.
Example check lists are included in Appendix 5.

After dealing with the cause of the spill it may be necessary to obtain permission from local authorities or the terminal (or both) to continue normal operations.

3.1.2 Tank overflow During Loading Or Bunkering

1. Have all cargo or bunker operations been stopped?

2. Have all valves been closed to prevent further ingress of oil to the overflowing tank?

3. Has agreed reporting procedure been followed?

4 Has the level of the affected tank been reduced by gravitating to a slack or empty tank or by pumping back ashore?

5. Has oil pollution equipment been utilised and operated in accordance with the manufacturer’s instructions, to prevent oil on deck being spilled overboard?

SPILLED OIL SHOULD NEVER BE WASHED OVERBOARD, NOR SHOULD DISPERSANTS OR DEGREASER BE USED ON OIL SPILLED OVERBOARD WITHOUT THE PERMISSION OF THE HARBOUR AUTHORITIES.

6. Has oil remaining on deck been removed using all means available?
7. If necessary, has use of the services of a clean-up company, if available, been made?

NO ACTIONS SHOULD BE TAKEN WHICH COULD IN ANY WAY JEOPARDISE THE SAFETY OF THE SHIP OR PERSONNEL EITHER ONBOARD OR ASHORE.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

After dealing with the cause of the spill it may be necessary to obtain permission from local authorities or the terminal (or both) to continue normal operations.

3.1.3 Hull Leakage

Unless timely corrective action is taken, oil will continue to rapidly escape from the tank until there is a hydrostatic balance between the sea and the liquid in the tank.. After hydrostatic balance has been reached, oil will escape from the affected tank more slowly as it is displaced by the ingress if water.

1. Has agreed reporting procedure been followed?
2. Has the leaking tank been identified and isolated from all other tanks?
3. Has the head of cargo or bunkers in the leaking tank been reduced, either by internal transfer or discharge ashore?

DUE REGARD MUST BE MADE OF OVERALL STRESS AND STABILITY CONSIDERATIONS BEFORE UNDERTAKING ANY TRANSFER OF CARGO OR BALLAST.

4. If it is not possible to identify the specific tank from which the leakage has occurred, have levels in the tanks in the vicinity of the leakage been reduced by internal transfer or discharge ashore?
5. If suspected that the leakage is from a fracture in the bottom plating or lower shell plating, has the level of the tank been reduced if full, or emptied if space is available elsewhere or ashore, and a water cushion then introduced to the damaged tank to prevent further oil spillage?
6. Has assistance of divers been requested to locate and reduce or stop the leak?

7. In every case where action in taken to prevent or minimize oil spillage, cargo segregation and quality concerns must be secondary to preventative measures.
8. If necessary, has use of the services of a clean-up company if available been made?

NO ACTION SHOULD BE TAKEN WHICH COULD IN ANY WAY JEOPARDISE THE SAFETY OF THE SHIP OR PERSONNEL EITHER ONBOARD OR ASHORE.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

After dealing with the cause of the spill it may be necessary to obtain permission from local authorities or the terminal (or both) to continue normal operations.

It is possible that failure of machinery, such as the oily water separating equipment or the oil discharge monitor, can cause an operational discharge while at sea in excess of that permitted. In such an incident the discharge should be stopped immediately and the correct report made. It is generally acknowledged that no clean up is possible by the ship, but the shore authorities can often respond more effectively to a spill in its early stages.

SECTION 3.2: SPILLS RESULTING FROM CASUALTIES

In the event of a casualty the master's first priority is to ensure the safety of the ship's personnel and to initiate action to prevent the incident from getting worse. Mitigating activities that can be considered are transfer of liquid from damaged compartments, containment of on-board deck spills, and preparation for dispersal of spills overboard.

If the casualty involves grounding, breaching of the outer hull, or other structural damage for which calculations of stability and damaged longitudinal strength are beyond the ship's resources, assistance must be sought from shore.

> An appendix can be referred to, which could include guidance on information that would be needed by an agency such as the ship's classification society to enable it to make damage stability and survival calculations. To allow prompt and worthwhile advantage to be obtained from such a service, pre-modelling of the ship on the agency's computer would be essential. Details of the damage condition can then be fed in and proposed corrective action rapidly assessed.

It may be necessary to transfer all or part of the cargo to another ship. The ICS/OCIMF publication "Ship to Ship Transfer Guide (Petroleum)" describes procedures to be followed in such a case. The advice is supplemented for noxious liquid cargoes by advice in the ICS "Tanker Safety Guide (Chemicals)". [For liquefied gas carriers it is appropriate to refer to "Ship to Ship Transfer Guide (Liquefied Gases)"]. Copies are held on board, and the master should encourage officers to familiarise themselves with the contents. When arranging a rendezvous, the master should ensure that the lightering vessel will also follow the same procedures.

The following casualty situations are dealt with:

- Grounding
- Fire/Explosion
- Collision (with a fixed or a moving object)
- Hull Failure
- Excessive List
- Containment System Failure
- Submerged or Foundered
- Wrecked or Stranded
- Hazardous Vapour Release
- Dangerous Reactions of Cargo
- Other Dangerous Cargo Release
- Loss of Tank Environmental Control
- Cargo Contamination Yielding A Hazardous Condition

There is much repetition in the following sub-sections, but this is deemed to be acceptable. The intent is that, in an emergency, the initial guidance given is as appropriate as possible. It is probable that, once the extent of a casualty has been assessed, the first essential steps taken and the situation stabilized, careful consideration will allow departure from or variation of pre-prepared plans. Then cross referral would be appropriate.

3.2.1 Grounding and Stranding

If the ship runs aground, the following steps should be taken immediately:

- Sound the emergency alarm and initiate emergency response procedures
- Eliminate all avoidable sources of ignition and ban all smoking on board
- Consider whether to stop air intake to accommodation and non-essential air intake to the engine-room
- In the case of a noxious liquid substance, consider what protection from vapour or liquid contact is necessary for the response team and for other crew members.
- Reduce the inert gas pressure to zero

Further action

- Carry out a visual inspection of the ship to determine the severity of the situation
- Take soundings around the ship to determine the nature and gradient of the seabed
- Check difference in the tidal ranges at the grounding site
- Evaluate tidal current in the grounding area
- Take soundings of all cargo, ballast and bunker tanks and check all other compartments adjacent to the hull. Ullage plugs should not be opened indiscriminately as loss of buoyancy could result
- Compare present tank soundings against departure soundings
- Evaluate the probability of additional release of oil or a noxious liquid substance.

Further information on the action to be taken when a ship is aground is contained in the ICS/OCIMF publication "Peril at Sea and Salvage - A Guide for Masters".

Having assessed the damage that the vessel has sustained, and taking into account the effects of hull stress and stability, the master should decide whether or not any action can be taken to avoid further spillage, such as:

- Transfer of cargo and bunkers internally. If the damage is limited, for example to one or two tanks, consideration should be given to transfer of liquid from damaged to intact tanks.
- Isolate all cargo and bunker tanks to reduce further loss due to hydrostatic pressure during tidal changes.
- Review existing and forecast weather conditions, and whether the ship will be adversely affected by them.
- Evaluate the possibility of transferring cargo to barges or other ships, and request such assistance accordingly.
- Trimming or lightening the vessel sufficiently to avoid damage to intact tanks, thereby avoiding additional pollution from spillage of oil or noxious liquid substances.

If the risk of additional damage to the ship by attempting to refloat it by its own means is assessed to be greater than by remaining aground until assistance has been obtained, the master should try to prevent the ship from moving from its present position by;

- Using anchors
- Taking in ballast in empty tanks (if possible)
- Reducing longitudinal stress on the hull by transferring cargo internally. Attention should be given to hull stress and damage stability information, referring to the classification society if necessary. Care must be taken over the compatibility of noxious liquid substances with tank type, material of construction and tank coating.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.2 Fire and Explosion

If an explosion or a fire occurs on board:
- Sound the emergency alarm, deploy the ship's fire emergency team(s) and follow the emergency procedures
- Determine the extent of the damage, and decide what damage control measures can be taken
- Determine whether there are casualties
- Request assistance as deemed necessary
- Assess the possibility of pollution from leakage of oil or noxious liquid substances
- If there is a spill of oil or noxious liquid substances in connection with the fire or explosion, inform appropriate parties in accordance with Section 2 of this plan.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.3 Collision with a fixed or moving object

If a collision occurs:
- Sound the emergency alarm and initiate emergency procedures.
- Determine whether there are casualties.

The master should assess the situation for pollution purposes as follows, taking action where appropriate:
- Decide whether separation of the ships may cause or increase the spillage of oil or noxious liquid substances
- If any cargo or bunker tanks are penetrated, reduce the risk of further spillage by isolating penetrated tanks or transferring liquid to slack or empty tanks. Care must be taken over the compatibility of noxious liquid substances with tank type, material of construction and tank coating.
- If there is a spill of oil or noxious liquid substances in connection with the collision, inform the appropriate parties in accordance with Section 2 of this plan.

Having assessed the damage and dealt with imminent danger, consideration of further action for repair or cargo transhipment, or mitigating the effect of liquid spilt, should be done in conjunction with appropriate authorities, in order to facilitate pollution control.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.4 Hull Failure

If the ship suffers severe structural hull failure:
- Sound the emergency alarm and muster the crew
- Reduce speed or stop to minimise stress on the hull
- Assess the immediate danger of sinking or capsize
- Initiate damage control measures
- Reduce the inert gas pressure to zero.

The master should then assess the situation for pollution purposes as follows:

- If oil or noxious liquid substances has spilled, inform the appropriate parties in accordance with Section 2 of this plan
- If immediate action is necessary to jettison cargo, inform the appropriate parties in accordance with Section 2 of this plan
- Consider whether offloading of oil or noxious liquid substances that is necessary in order to maintain stability can wait until another ship or a barge is available
- If the change in stability and stress cannot be calculated on board, contact the classification society and arrange for the necessary calculations to be carried out
- Consider the forecast weather conditions and the effect they may have on the situation.

Having assessed the damage and dealt with imminent danger, consideration of further action for repair or cargo transhipment, or mitigating the effect of liquid spilt, should be done in conjunction with appropriate authorities, in order to facilitate pollution control.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.5 Excessive List

If excessive list occurs rapidly and unexpectedly it may be due to:

- Failure of the hull plating
- Failure of an internal bulkhead between compartments
- Shift of cargo
- Flooding of a large space such as the engine room, where free surface can cause a list
- Damage through grounding or collision
- Incorrect operational procedures.

Steps to be taken immediately:

- Stop any cargo, bunkering or ballast operations in progress, and close all tank valves and pipeline master valves
- Sound the emergency alarm and muster the crew
- If under way, reduce speed or stop
- Establish the reason for the list.

Further measures

- Sound all tanks and compare soundings with departure soundings
- If oil or a noxious liquid substance has spilled, inform the appropriate parties in accordance with Section 2 of this plan
- If immediate action is necessary to jettison cargo, inform the appropriate parties in accordance with Section 2 of this plan
- Consider whether offloading of oil or noxious liquid substances that is necessary in order to maintain stability can wait until another ship or a barge is available
- If possible, take corrective action to rectify the situation.

Having assessed the damage and dealt with imminent danger, consideration of further action for repair or cargo transhipment, or to mitigate the effects of liquid spilt, should be done in conjunction with appropriate authorities, in order to facilitate pollution control.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.6 Containment System Failure

If there has been an internal failure of the bunker oil or cargo containment system, other than pipeline leakage, it is likely that it will be detected by another symptom such as an excessive list, a tank overflow or external hull leakage, often preceded or accompanied by a loud or unusual noise. Advice on initial reaction in each case will be described under other sections. However, once a failure of the internal containment system has been identified, there may be additional responses that can be taken to avoid or mitigate a spill of oil or a noxious liquid substance.

Steps to be taken immediately:
- Stop any cargo, bunkering or ballasting operations in progress, and close all tank valves and pipeline master valves
- If under way, consider reducing speed or stopping
- If in port, consider evacuation of non-essential personnel

Further measures:
- Determine the extent of the damage, and decide what damage control measures can be taken.
- If the failure has occurred to a system containing a noxious liquid substance, consider what safety precautions are necessary to protect the crew from vapours or contact with the liquid
- Assess the possibility of pollution from leakage of oil or noxious liquid substances
- If oil or a noxious liquid substance has spilled, inform the appropriate parties in accordance with Section 2 of this plan
- If immediate action is necessary to jettison cargo, inform the appropriate parties in accordance with Section 2 of this plan
- Consider whether offloading of oil or a noxious liquid substance that is necessary in order to maintain stability can wait until another ship or a barge is available
- Consider whether the level of liquid in the tanks associated with the system failure should be reduced. Remember to consider the effect on hull stress and stability of the vessel and the compatibility of noxious liquid substances with tank type, material of construction and tank coating.

If the spilled liquid is contained on board and can be handled by the Pollution Prevention Team then:
- Use sorbents and permissible solvents to clean up the liquid spilled on board.
- Ensure that any residues collected, and any contaminated absorbent materials used in the clean up operation are stored carefully prior to disposal.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.7 Submerged, Foundered or Wrecked

If the ship is wrecked to the extent that it is in imminent danger of foundering or being completely or partially submerged, safety of the lives of the crew will take priority over preventing pollution. It is likely that the event which caused the sinking will have led to some surface pollution already. However, if time allows, it may be possible to take some measures, which will limit subsequent spillage.

The following actions may be considered, if there is no risk to the safety of the crew, and time allows:

- Inform the appropriate parties in accordance with Section 2 of this plan
- Close all tank valves and pipeline master valves
- Screw down or lock shut any tank vent valves
- Close vent flaps and watertight openings in the cargo area
- Alert other ships and navigational authorities to the presence of pollutants.

The use of a simple check list is recommended.
Example check lists are included in Appendix 5.

3.2.8 Hazardous Vapour Release

For oil tankers and cargo ships at sea, it is unlikely that a significant marine pollution hazard will be created solely by vapour release. In port the main problem with such an event is safety of the crew and nearby shore personnel in a flammable or toxic atmosphere.

Steps to take immediately:

- Stop any cargo, bunkering or ballasting operations in progress, and close all tank valves and pipeline master valves
- Eliminate possible sources of ignition
- If under way, consider altering course to create the best wind flow, or reducing speed or stopping
- If in port, consider evacuation of non-essential personnel
- If in port, alert shore and terminal personnel, and the crew of craft alongside

Further measures

- Establish the reason for the hazardous vapour release
- If possible, take corrective action to rectify the situation.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.9 Dangerous Reactions of Cargoes, and Cargo Contamination Yielding a Hazardous Condition

This is an inherent danger associated with the carriage of chemical cargoes and noxious liquid substances. If a dangerous cargo reaction or contamination of a cargo yielding a hazardous condition occurs, the consequences to the safety of the ship, its crew and nearby shore personnel will be paramount, and must take priority. However, a result of the occurrence may be a spill into the sea, and mitigation of the risk of pollution or actual pollution can be addressed by consideration of the following measures:

Steps to take immediately:

- Stop any cargo, bunkering or ballasting operations in progress, and close all tank valves and pipeline master valves
- Eliminate possible sources of ignition
- If oil or a noxious liquid substance has spilled, inform the appropriate parties in accordance with Section 2 of this plan
- If immediate action is necessary to jettison cargo, inform the appropriate parties in accordance with Section 2 of this plan
- If in port, alert shore and terminal personnel, and the crew of any craft alongside

Further measures

- Establish the reason for the reaction or contamination
- If possible, take corrective action to rectify the situation. This may involve reducing the liquid level in a tank, cooling or diluting a mixture where a reaction is in progress, or containing the process by smothering with inert gas or foam.
- Consider whether offloading of oil or a noxious liquid substance that is necessary for safety or in order to maintain stability can wait until another ship or a barge is available
- If the change in stability and stress cannot be calculated on board, contact the classification society and arrange for the necessary calculations to be carried out
- Consider whether external salvage assistance may be necessary for control and mitigation of a probable spill. An early warning that proves to be over-cautious and must be cancelled may be better than a late call that cannot be met.

Having assessed the situation and dealt with imminent danger, consideration of further action or cargo transhipment, or to mitigate the effects of liquid spilt, should be done in conjunction with appropriate authorities, in order to facilitate pollution control.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.10 Other Dangerous Cargo Release

Danger to personnel, the ship or shore installations should be given priority over the potential for pollution. However, release of noxious liquid substances into the sea can create dangerous situations through reaction between the liquid and water.

Steps to take immediately:

- Stop any cargo, bunkering or ballasting operations in progress, and close all tank valves and pipeline master valves
- Eliminate possible sources of ignition
- By reference to the cargo information sheet, determine whether a dangerous reaction with water is likely
- If oil or a noxious liquid substance has spilled, inform the appropriate parties in accordance with Section 2 of this plan
- If in port, alert shore and terminal personnel, and the crew of any craft alongside

Further measures

- Establish the reason for the cargo release
- If possible, take corrective action to stop the release. This may involve reducing the liquid level in a tank by transferring it to another tank. Remember to consider the effect on hull stress and stability of the vessel and the compatibility of noxious liquid substances with tank type, material of construction and tank coating.
- If the change in stability and stress cannot be calculated on board, contact the classification society and arrange for the necessary calculations to be carried out
- If it is necessary to offload a noxious liquid substance, consider whether it can wait until another ship or a barge is available
- Consider whether external salvage assistance may be necessary for control and mitigation of a probable spill. An early warning that proves to be over-cautious and must be cancelled may be better than a late call that cannot be met.

Having assessed the situation and dealt with imminent danger, consideration of further action or cargo transhipment, or to mitigate the effects of liquid spilt, should be done in conjunction with appropriate authorities, in order to facilitate pollution control.

The use of a simple checklist is recommended.
Example checklists are included in Appendix 5.

3.2.11 Loss of Tank Environmental Control

Many noxious liquid substances carried as cargo by sea are required to have the atmosphere above the liquid controlled in order to ensure safety. Loss of this control can result in a hazardous situation that may lead to marine pollution. If loss of control occurs in tanks containing such cargoes, the following measures can avoid or mitigate the effect and the possibility of a spill.

Steps to take immediately:

- Ensure necessary precautionary safety measures for crew, ship and shore are taken
- Establish the reason for the loss of tank environmental control, and if possible take corrective action
- Establish a continual monitoring process to compare liquid levels, tank pressures or cargo temperatures.
- By reference to the cargo information sheet, determine whether a dangerous situation is likely to arise. If a spill is probable, or jettison of the noxious liquid substance may become necessary, inform the appropriate parties in accordance with Section 2 of this plan

The use of a simple check list is recommended.
Example checklists are included in Appendix 5.

SECTION 4 - NATIONAL AND LOCAL CO-ORDINATION

Quick, efficient co-ordination between the ship and coastal state or other involved parties becomes vital in mitigating the effects of an oil or NLS pollution incident.

In most countries it is accepted that a spillage of oil or a noxious liquid substance can be tackled most effectively from the shore and there is normally no requirement on the part of the shipowner or the ship's crew to organise the clean-up response in respect of a pollutant lost overboard. Operational spills usually occur in port at an oil or chemical cargo terminal or at a bunkering facility, and tend to be cleaned up by the facility operator. In the case of casualties, the responsibility for organising and controlling the clean-up response is usually assumed by an agency of government. In both cases the spiller would be expected to co-operate fully, and pay the reasonable costs of clean-up and any damages caused, up to a specified limit of liability based on the tonnage of the ship.

SECTION 5 - ADDITIONAL INFORMATION

This is the section where company advice and instructions, and any pollution avoidance procedures developed in-house,are likely to be placed. Text in this section does not require approval by the Flag Administration, therefore it can be revised as necessary to ensure the guidance given reflects the best industry practice.

1. **Company policy** may require other matters to be covered in the plan such as diagrams and drawings, spill response equipment carried on board, public affairs, record-keeping and reference materials**.**
2. **Diagrams and drawings:** In addition to the plans referred to in Section 1, other details concerning the ship's design and construction may be appended to the plan or their location identified (See Appendix 7).
3. **Response equipment:** Some ships may carry equipment on board to assist in pollution response. The plan should provide an inventory of such equipment. It should also provide directions for safe use, and guidelines to assist the master in determining when use is warranted. Care should be exercised to ensure that the use of such equipment by the crew is practical and consistent with safety considerations. When specialized equipment is carried, the plan should establish personnel responsibilities for its deployment and maintenance (see Appendix 8 which includes an example of a maintenance schedule), and also provide for crew training in its use. The plan should include a provision that no chemical agent should be used for response to pollution outside the ship without the approval of the appropriate coastal state, and that authorization may need to be requested, when use of containment or recovery equipment is necessary.
4. **Public affairs:** The company may wish to include in the plan guidance for the master in dealing with the distribution of information to the news media. Such guidance should take into account the need to minimise the burden on the master who will already be busy with the emergency at hand.
5. **Record-keeping:** As with any other incident that may give rise to questions of liability, compensation and reimbursement, the company may wish to include guidance on the keeping of appropriate records of the incident. Apart from detailing all actions taken on board, records might include communications with outside authorities, the company, and other parties, as well as a brief summary of decisions and information passed and received. Guidance on collecting samples of spilt oil or noxious liquid substances as well as that carried on board may also be provided.
6. **Plan review**: Regular review of the plan by the master is recommended to ensure that it remains current. A system should be employed which will allow quick revision of the plan, both on board and by the company as follows:

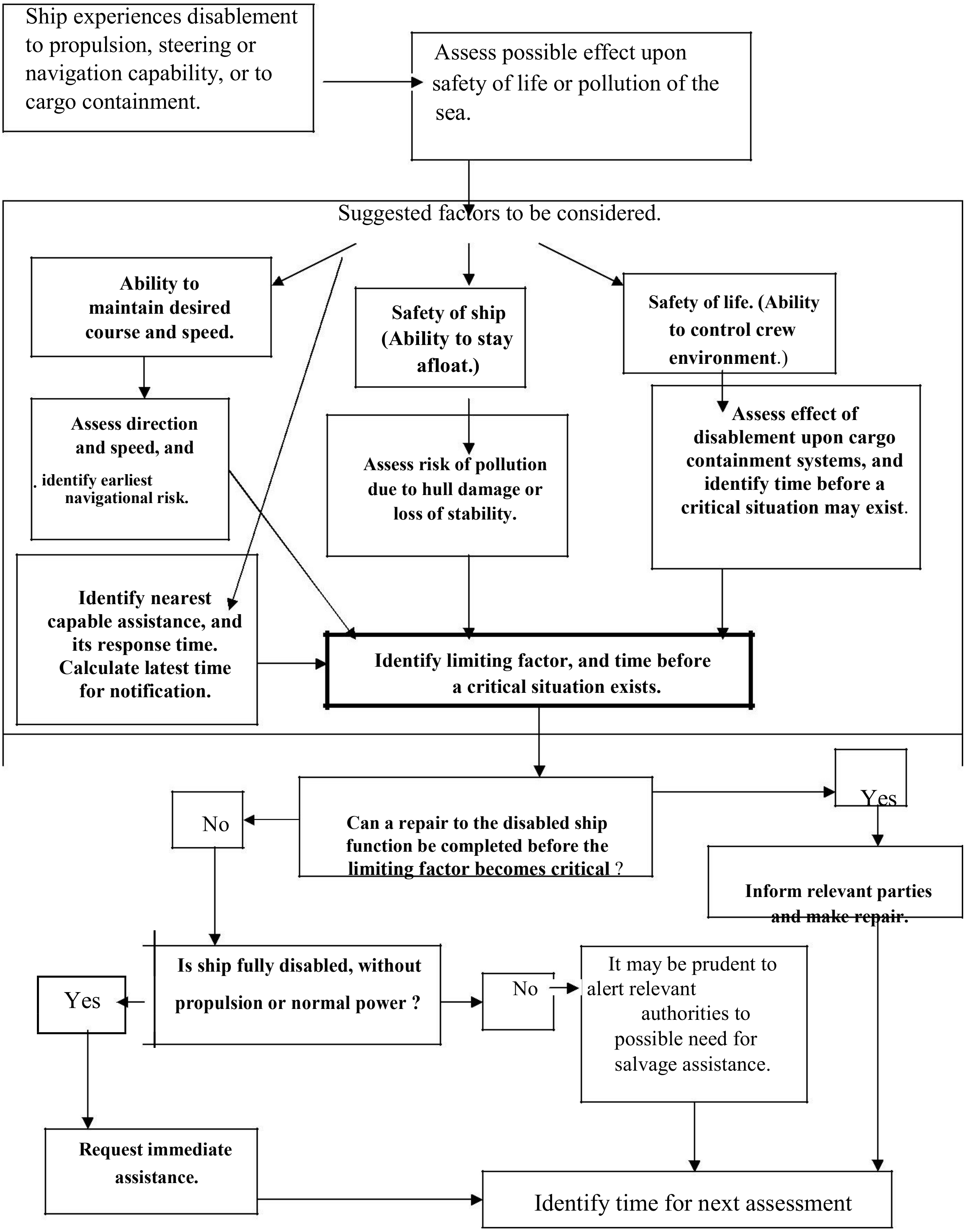
   6.1 Periodic review: The plan should be reviewed by the Company at regular intervals to take account of changes in local law or policy, contact names and numbers, ship characteristics, or company policy;

   6.2 Event review: After any use of the plan in response to an incident, its effectiveness should be evaluated by the Company and modifications made accordingly.

7. **Plan testing:** The plan will be of little value if it is not familiar to the personnel who will use it. Regular exercises should be held to confirm that the plan functions effectively. Such exercises may be held in conjunction with other shipboard exercises and appropriately recorded (see Appendix 9). Where ships carry response equipment, hands-on experience of it on the part of crew members will greatly enhance effectiveness in an emergency situation.
8. **Salvage:** The Plan should contain information on what the crew's responsibilities are in a casualty where a ship is partially or fully disabled, and what constitutes dangerous conditions. A decision process should be outlined in the Plan that will aid the master in determining when salvage assistance should be obtained. The decision process should address the nearest land or hazard to navigation, the ship's set and drift, the location and time of impact with a hazard based on ship's set and drift, the estimated time of completing rectification of a disabling defect, and determination of the nearest capable assistance and its response time (i.e., for tug assistance, the time it will take to get on scene and secure the tow).

   When a casualty occurs to a ship under way that reduces its maneuverability, the master needs to determine his window of opportunity considering the response time of assistance, regardless of the estimated time of repair. It would not be prudent to hesitate in calling for assistance when the time needed to repair something goes beyond the window of opportunity. The following flow chart may be found useful as a basis for providing guidance to a master or to the shore management in assessing the need and urgency of calling for salvage assistance when a casualty occurs.

MODEL FLOWCHART TO ASSIST MASTER TO DETERMINE WHEN SALVAGE ASSISTANCE SHOULD BE OBTAINED.

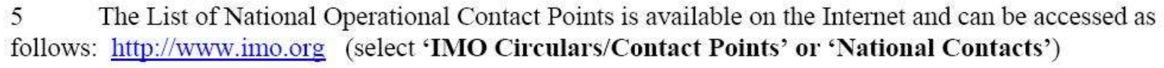


APPENDIX OVERVIEW

Appendix 1	List of coastal state contacts
Appendix 2	List of port contacts
Appendix 3	List of ship interest contacts
Appendix 4	Pollution prevention team
Appendix 5	Check lists for use in emergencies
Appendix 6	Advice for immediate information needed by a shore agency to assess damaged stress & stability calculations
Appendix 7	Plans and Drawing
Appendix 8	List of oil spill response equipment carried on board, and maintenance schedule.
Appendix 9	Record of oil pollution prevention drills.
Appendix 10	References.

APPENDIX 1

LIST OF COASTAL STATE CONTACTS



5 The List of National Operational Contact Points is available on the Internet and can be accessed as follows: http://www.imo.org (select **'IMO Circulars/Contact Points'** or **'National Contacts'**)

The attached list should be updated regularly.

In the absence of a listed focal point, or should any undue delay be experienced in contacting the responsible authority by direct means, the master should be advised to contact the nearest coastal radio station, designated ship movement reporting station or rescue co-ordination center (RCC) by the quickest available means.

APPENDIX 2

LIST OF PORT CONTACTS

PORT	INSTITUTION AND PERSON TO BE CONTACTED	MEANS OF CONTACT	REMARKS
Mention terminal or berth if appropriate.		Tel. office: ……… Tel. mobile: ……. Fax.: ………… Email: …………	Date of visit

APPENDIX 3

LIST OF SHIP INTEREST CONTACTS

Institution and person to be contacted	**Address**	**Means of contact**	**Remarks**
Owner /Operator Designated person (DP) After hours watch (24/7)	Amar Shipping 3 AS Haugesund 123, 4262 Avaldsnes Norway		Operation
Charterer			No info yet
Local agent			To be logged on bridge
P&I Club and Correspondents GARD P&I, Bergen	Gard AS Skipsbyggerhallen Solheimsgaten 11 NO-5058 Bergen Norway	Tel.+4737019100	
Emergency contact		Mob.+4790524100	24/7
Norwegian Hull Club H&M	Olav Kyrresgate 11 NO-5014 Bergen Postal address: P.O. Box 75 Sentrum NO-5803 Bergen	Tel.+4755559500 Fax.+4755559555	
Emergency contact	Norway	Tel.+4722428844	24/7
Classification Society Bureau Veritas AS			
Norwegian Maritime Authority	Postboks 2222 N-5509 Haugesund, Norway	Tel.+4752745000 Fax.+4752745001	
Emergency contact		Tel.+4752745000	24/7

INSTITUTION AND PERSON TO BE CONTACTED	ADDRESS	MEANS OF CONTACT	REMARKS
Owner / operator. Designated person.			
Charterer			
Local agent			
P&I Club and correspondents Bergen, Gard A.S	Skipsbyggerhallen Solheimsgaten 11 5088 Bergen Norway	Tel. + 47 37 01 91 00 Fax. + 47 55 17 40 01	
Classification society			

APPENDIX 4

POLLUTION PREVENTION TEAM

The master of the ship should appoint a pollution prevention team on board. The primary function should be to initiate immediate recovery or clean-up procedures if an incident occurs during cargo operations or bunker transfer. The company's spill response plan should be brought to the attention of everyone in the team, so that they understand their own part in the broader picture.

In the event of a spillage of oil or a noxious liquid substance the team should be called out immediately.

The team should be given the necessary training in the use of spill containment equipment or absorbents carried on the ship. All members of the Pollution Prevention Team should be aware of their duties should a spill occur.

Suggested instructions to a Pollution Prevention Team

Master **In overall charge**

Inform terminal authorities of incident.
Inform local agent and request agent to inform the local P&I Club representative.
Advise company's head office.
Keep everyone updated at regular intervals.
Advise of any changes in status of the emergency.
Request assistance as deemed necessary.

Chief Officer **In charge of deck operation.**

Keep master informed and updated on the situation.
Ensure event log is maintained.
Report results of steps taken to limit liquid outflow.

Chief Officer **In charge of bunker operations.**

If bunkering in progress, stop operation.
Organise distribution of oil spill detergent or appropriate treatment.
Organise starting of foam pump if required.

Deck officer on duty **Tank spillage:**

Open an empty or slack tank.
Stop pumping of that cargo; consider stopping cargo operations.
Alert and inform chief officer and master of the situation.
Alert shore staff.

Deck officer on duty **Prepare for fire fighting.**

Assist chief engineer.

Deck officer on duty **If a leakage is detected,**

Alert duty officer immediately.

APPENDIX 5

A. Checklist for response to operational spill of oil or noxious liquid substance:

This checklist is intended for response guidance when dealing with a spill of oil or a noxious liquid substance during cargo or bunkering operations. Responsibility for action to deal with other emergencies which result from the liquid spill will be as laid down in existing plans, such as the Emergency Muster List.

ACTION TO BE CONSIDERED	ACTION TAKEN		PERSON RESPONSIBLE
Immediate Action	**Yes**	**No**	
Sound Emergency Alarm	…	…	Person discovering incident
Initiate ship's emergency response procedure	…	…	Officer on duty
Initial Response			
Stop all cargo and bunkering operations	…	…	Officer on duty
Close manifold valves	…	…	Officer on duty
Stop air intake to accommodation	…	…	Officer on duty
Stop non-essential air intake to machinery spaces	…	…	Officer on duty
Locate source of leakage	…	…	Officer on duty
Close all tank valves and pipeline master valves	…	…	Officer on duty
Commence clean-up procedures using absorbents and permitted solvents.	…	…	Officer on duty
Comply with reporting procedures	…	…	Master
Secondary Response			
Assess fire risk from release of flammable liquids or vapour	…	…	Chief Officer
Reduce liquid level in relevant tank by dropping into an empty or slack tank	…	…	Chief Officer
Reduce liquid levels in tanks in suspect area	…	…	Chief Officer
Drain affected pipeline to empty or slack tank	…	…	Chief Officer
Reduce inert gas pressure to zero	…	…	Chief Officer
If leakage is at pumproom seavalve, relieve pipeline pressure	…	…	Chief Officer
Prepare pumps for transfer of liquid to other tanks or to shore or to lighter	…	…	Chief Officer
Prepare portable pumps for transfer of spilt liquid to empty tank	…	…	Chief Officer
Further response			
Consider mitigating activities to reduce effect of spilt liquid	…	…	Master
Pump water into leaking tank to create water cushion under oil or light chemical to prevent further loss	…	…	Chief Officer
If leakage is below waterline, arrange divers to investigate	…	…	Master
Calculate stresses and stability, requesting shore assistance if necessary	…	…	Chief Officer
Transfer cargo or bunkers to alleviate high stresses	…	…	Chief Officer
Designate stowage for residues from clean-up prior to disposal	…	…	Officer on duty

B. Checklist for response to spill of oil or noxious liquid substance after a casualty:

This checklist is intended for response guidance when dealing with a spill of oil or a noxious liquid substance following a casualty. Responsibility for action to deal with the casualty itself will be as laid down in existing plans, such as the Emergency Muster list.
The term "Navigator" refers to the officer responsible for passage planning and voyage analysis, usually the second officer.

ACTION TO BE CONSIDERED	ACTION TAKEN		PERSON RESPONSIBLE
Immediate Action	**Yes**	**No**	
Sound Emergency Alarm	…	…	Person discovering incident
Initiate ship's emergency response procedure	…	…	Officer on duty
Initial Response			
Stop air intake to accommodation	…	…	Officer on duty
Stop non-essential air intake to machinery spaces	…	…	Officer on duty
Assess further danger to ship or personnel by such as capsize or immediate sinking	…	…	Master
Stop all cargo and ballasting operations	…	…	Officer on duty
Close all tank valves and pipeline master valves	…	…	Officer on duty
Assess whether oil or NLS has actually been spilt	…	…	Officer on duty
Assess whether oil or NLS will probably be spilt	…	…	Master
Assess security of tank environmental control systems	…	…	Officer on duty
Assess risk of complex chemical reaction in NLS cargo	…	…	Officer on duty
Comply with reporting procedures	…	…	Master
Sound all compartments	…	…	Officer on duty
Sound around ship if it is aground	…	…	Officer on duty
Request outside assistance	…	…	Master
Stop or reduce outflow of oil or NLS	…	…	Officer on duty
Counter excessive list	…	…	Officer on duty
Contain spilt liquid still on deck	…	…	Officer on duty
Commence clean-up procedures using absorbents and permitted solvents.	…	…	Officer on duty
Further response			
Reduce inert gas pressure to zero	…	…	Officer on duty
Assess fire risk from release of flammable liquids or vapour	…	…	Officer on duty
Consider evacuation of non-essential crew	…	…	Master
Assess likelihood of further damage to ship or cargo	…	…	Master
Calculate stresses and stability, requesting shore assistance if necessary	…	…	Officer on duty
Transfer cargo or bunkers to alleviate high stresses	…	…	Officer on duty
Request assistance or escort to place of refuge	…	…	Master
Manoeuvre upwind of spill and / or away from land	…	…	Master
Assess effect of tide and current, on ship and spilt liquid	…	…	Officer on duty r
Obtain weather forecast and assess effect on ship	…	…	Master
Prepare pumps for transfer of liquid to other tanks or to shore or to lighter	…	…	Officer on duty
Reduce liquid levels in tanks in suspect area	…	…	Officer on duty
Designate stowage for residues from clean-up prior to disposal	…	…	Officer on duty

APPENDIX 6

EMERGENCY STRESS AND STABILITY CALCULATIONS

When taking steps to mitigate the spillage of oil or noxious liquid substances, great care must be taken to consider stability and strength of the ship, especially if the hull is damaged due to collision or grounding. When the damage sustained is extensive, the impact of internal transfers on stress and stability may be impossible for the ship to assess. Contact may have to be made with shore entity with the necessary capability.

The master should contact the owners' head office in order to gain access to these facilities.

Before the transfer of noxious liquid substances in bulk, there must be consideration of the compatibility of all substances involved, such as other cargoes, bunkers, tank materials and coatings, pipelines, etc.

-o0o-

INSTITUTION AND PERSON TO BE CONTACTED

MEANS OF CONTACT

ADDRESS:

INFORMATION TO HAVE READY FOR TRANSMISSION IN INITIAL CONTACT.

Identity of ship	Fishfarming support vessel
Type of casualty	…………………………….
Present and expected weather	…………………………….
Details of damage	…………………………….

APPENDIX 7

PLANS, DRAWINGS AND SHIP-SPECIFIC DETAILS

It is particularly important that the appended plans and information receive attention during any overall review of the plan, and after the ship has undergone refit or repairs in a shipyard.

APPENDIX 8

LIST OF SPILL RESPONSE EQUIPMENT CARRIED ON BOARD, AND PLANNED MAINTENANCE SCHEDULE

Response equipment

Type	Quantity	Location
Heavy duty Neoprene gloves	1 pr	Coffe shop
Green Nitrile gloves	1 pr	Coffe shop
Safety chemical goggles	1 pr	Coffe shop
Tyvek disposable overalls	1 pr	Coffe shop
P2 Dust/Mist respirator	1	Coffe shop
Dust pan and brush	1	Coffe shop
Plastic scoop (for spreading or collecting absorbent material)	1	Coffe shop
100 um Plastic Waste Disposal bags	5	Coffe shop
Caution – Spill Area barricade tape	1 roll	Coffe shop
Absorbent boom/snake (long) 2m	1	Coffe shop
Absorbent boom/snake (long) 1.2m	2	Coffe shop
General Absorbent pads	50	Coffe shop
Oil & fuel Absorbent e.g.: Global Peat	1	Coffe shop

PLANNED MAINTENANCE SCHEDULE:

1. Prevention Equipment

Tank Lids, check that:

- tank lids sit squarely on the coamings
- packing is in good condition
- cleats have sufficient movement
- sighting ports are sitting properly
- packing of sighting ports

Butterworth Plates, check that:

- plates sit squarely on aperture
- packing is properly fitted with no gaps
- all studs have good threads
- retaining nuts screw down tightly

Deck Pipelines (cargo, bunker and hydraulic), check:

- the condition of deck lines, ensuring that there is no apparent leakage
- couplings for signs of leakage
- deck valves for tightness
- that blanks are available for all manifolds, and that all fit well with bolts in each hole
- that sample cocks are fitted tightly with no leakage from either the sampling end or the end connected to the pipework

Hull Plating, check the condition of hull for damage or possible weak spots, and notify Head Office of areas of concern.

If necessary, make temporary repairs to ensure tightness and ensure that Head Office is informed.

2. Containment Equipment

Check that:

- drip trays are sound with no obvious cracks or holes
- save-alls around bunker vent pipes (where fitted) are sound
- scupper plugs are in good condition and that they are a good fit in the scuppers
- there are sufficient spare scupper plugs on board, and their location is known
- portable pumps and educators are working satisfactorily
- all drain plugs in drip trays and save-alls can be shut tight
- there are sufficient quantities of detergent on board, and its location is known
- there are sufficient quantities of absorbent material on board, and its location is known
- there are sufficient scoops, buckets and squeegees on board for mopping up operations, and their location is known
- that pipework and gauges associated with deep well cargo pumps are tight.

3. Spillage Equipment

Check that:

- detergent or treatment fluid is in containers which would make it readily available for use
- foam branch pipes and portable spraying equipment is readily available and in good working order
- all methods of communication can be operated effectively

4. Permanent equipment

Check that:

- eductors are in good working order
- all components of the engine room bilge oily water system work satisfactorily, and there is a sign in the vicinity of associated overboard discharge(s), indicating the need for them to be shut and lashed in port
- overboard discharge valves are lashed shut when not in use
- all components of the oil discharge monitoring equipment in the ballast system work satisfactorily
- the MARPOL interface detector is readily available and in good condition

APPENDIX 9

RECORD OF POLLUTION PREVENTION DRILLS

DATE	TYPE OF SPILL CONTINGENCY	LOCATION OF SHIP	PARTICIPANTS

APPENDIX 10

REFERENCES

The following publications will provide additional assistance to the preparation of ships pollution emergency plans:

"Guidelines for the Development of Shipboard Marine Pollution Emergency Plans. 2001 Edition".
International Maritime Organization (IMO)
Available in English, French and Spanish from IMO, Publications Section, 4 Albert Embankment, London SE1 7SR.
(This booklet contains guidelines for the Shipboard Oil Pollution Emergency Plan (SOPEP) required by ships carrying oil as cargo or as bunkers, and guidelines for the Shipboard Marine Pollution Emergency Plan (SMPEP) required by the above ships that are also certified to carry noxious liquid substances.)

"Provisions Concerning the Reporting of Incidents Involving Harmful Substances under MARPOL 73/78".
International Maritime Organization (IMO)
Available in English, French and Spanish from IMO, as above.

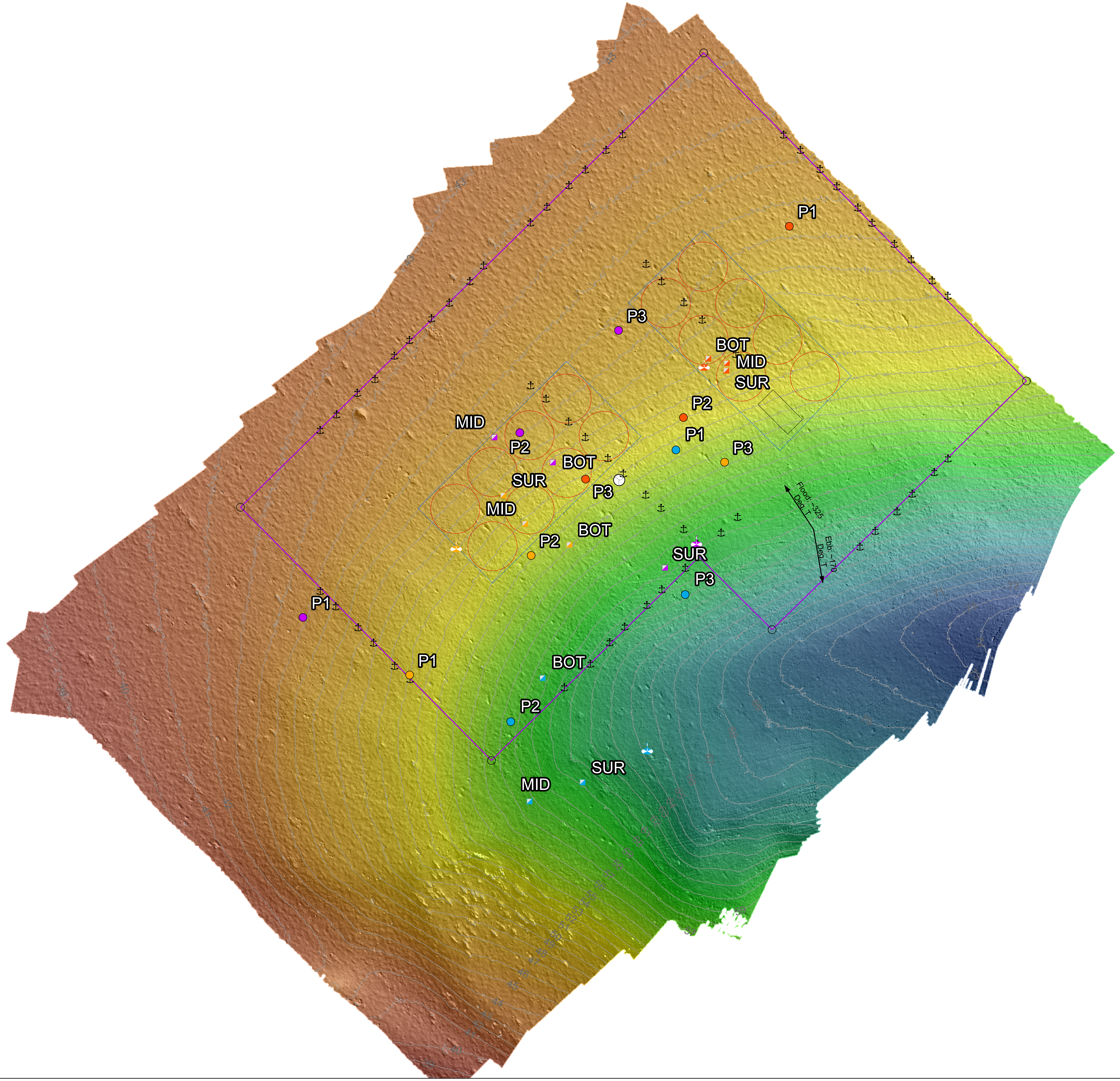
"Peril at Sea and Salvage - A Guide for Masters"
International Chamber of Shipping and Oil Companies International Marine Forum (ICS/OCIMF)
Available from Witherby & Co. Ltd., London 32-36 Aylesbury Street, London EC1R OET

“Tanker Safety Guide (Chemicals)” and “Tanker Safety Guide (Liquefied Gas)”
International Chamber of Shipping (ICS)
Available from ICS, London
12 Carthusian Street, London EC1M 6EZ

"Ship to Ship Transfer Guide (Petroleum)”
International Chamber of Shipping and Oil Companies International Marine Forum (ICS/OCIMF)
“Ship to Ship Transfer (Liquefied Gases)”
International Chamber of Shipping, Oil Companies International Marine Forum, and Society of Liquefied Gas Tanker and Terminal Operators (ICS / OCIMF / SIGTTO))
Available from Witherby & Co. Ltd., London

"International Safety Guide for Oil Tankers and Terminals"
International Chamber of Shipping, Oil Companies International Marine Forum, and International Association of Ports and Harbors (ICS / OCIMF / IAPH)
Available from Witherby & Co. Ltd., London

"Response to Marine Oil Spills"
International Tanker Owners Pollution Federation (ITOPF)
Available in English, French, and Spanish from Witherby & Co. Ltd., London



Sample Month and ID	Easting (UTM m)	Northing (UTM m)
Jul, P1	566,903	4,919,001
Jul, P2	566,745	4,918,741
Jul, P3	566,912	4,918,862
Jul, SECCHI	566,875	4,918,715
Jul, BOT	566,775	4,918,783
Jul, MID	566,763	4,918,665
Jul, SUR	566,813	4,918,683
Aug, BOT	566,801	4,918,910
Aug, MID	566,758	4,918,930
Aug, SUR	566,738	4,918,957
Aug, P1	566,648	4,918,786
Aug, P2	566,764	4,918,900
Aug, P3	566,949	4,918,989
Aug, SECCHI	566,693	4,918,908
Sep, BOT	566,785	4,918,989
Sep, MID	566,729	4,919,013
Sep, P1	566,546	4,918,841
Sep, P2	566,754	4,919,017
Sep, P3	566,848	4,919,115
Sep, SUR	566,892	4,918,888
Sep, SECCHI	566,922	4,918,913
Oct, SECCHI	566,930	4,919,082
Oct, BOT	566,933	4,919,088
Oct, MID	566,951	4,919,083
Oct, P1	567,011	4,919,214
Oct, P2	566,910	4,919,031
Oct, P3	566,816	4,918,973
Oct, SUR	566,951	4,919,076

RANSOM
Consulting
Engineers
and Scientists

Legend and Notes

Jul 2020 Sampling
Other
● Profile
◪ Sample
✛ Secchi

Aug 2020 Sampling
● Profile
◪ Sample
✛ Secchi

Sep 2020 Sampling
● Profile
◪ Sample
✛ Secchi

Oct 2020 Sampling
● Profile
◪ Sample
✛ Secchi

Lease Features
☐ Lease Boundary
☐ Mooring Grid Boundary
☐ Pen Outline
☐ Barge Outline
○ Lease Corner
⚓ Anchor
○ ADCP Location
— Bathy. Contour (m MLLW)

General Notes:

1. Basemap from NOAA RNC Server.
2. Some features shown are approximate in location and scale.
3. GIS data accessed and downloaded from the Maine Office of GIS, NOAA Digital Coast, and/or The National Map.
4. Pen layout and intake/discharge structure locations based on plans prepared by Aqua Knowledge AS.
5. Bathymetry from multibeam survey completed by Substructure in Sept, 2020.

Prepared For

American Aquafarms
68 Commercial Street
Portland, Maine

Site Address

Long Porcupine
Frenchman Bay
Gouldsboro, Maine

Orientation and Scale

0 75 150 300 Ft
1 inch = 150 feet

201.05012 *Apr 2021*

Supplemental Figure 1
Lease Layout and
Sample Locations

NOAA / NOS Special Projects / Office of Coast Survey