Quality Assurance Project Plan

for

Sediment Chemistry Analyses for Harbor and Outfall Monitoring

Massachusetts Water Resources Authority
Technical Report 2012-10

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Sediment Chemistry Analyses for Harbor and Outfall Monitoring

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1.0 PROJECT MANAGEMENT

1.1 Project Organization

Figure 1 presents the project management structure for sediment chemistry analyses by the MWRA Department of Laboratory Services (DLS) for outfall monitoring. This project is part of the Harbor and Outfall Monitoring (HOM) project of the MWRA Environmental Quality Department (ENQUAD). It includes onshore sample handling, sample analysis, and data loading for the sediment chemistry analyses that are part of the benthic study in the MWRA's outfall ambient monitoring program (bay soft-bottom monitoring study, or BMBSOFT) and harbor monitoring program (harbor soft-bottom monitoring program, or BHSOFT.)

The current HOM contract, currently held by Normandeau Associates, Inc., includes 2011-2013 field sampling.

ENQUAD Dr. Andrea Rex is the Director of the Environmental Quality Department. Mr. Kenneth Keay is the Harbor and Outfall Monitoring Program Manager for ENQUAD. Mr. Keay is also the Benthic Project Manager, and is primarily responsible for benthic/sediment studies within that program. He is responsible for general coordination of monitoring activities and for reviewing monitoring data before it is loaded into the EM & MS database. His responsibility is also to ensure that the data collected as part of the monitoring project satisfies the quality objectives set forth in this QAPP. Ms. Wendy Leo leads the data management group and serves as ENQUAD's Quality Assurance Manager. She is responsible for assigning staff to transfer data from the DLS Laboratory Information Management System (LIMS) into the ENQUAD environmental monitoring and mapping database (EM&MS) and transmitting them to Normandeau. Dr. Douglas Hersh is ENQUAD's Database Administrator for the EM&MS database.

DLS Dr. Michael F. Delaney is the Director of Laboratory Services. Dr. Yong Lao is the Laboratory's Project Manager and is DLS' primary point of contact for this project. Mr. Steve Rhode is the Section Manager responsible for Client Services and the Violet Team. Mr. Edward Caruso is the Client Services Coordinator and is responsible for handling client requests and assisting with Violet Team responsibilities. Ms. Polina Epelman is the Section Manager responsible for the Red, Orange, and Green Teams. Mr. Jim Fitzgerald is the Supervisor of the Violet team, responsible for sample management. Ms. Nancy McSweeney is the Supervisor of the Red Team, responsible for solids analyses. Ms. Patricia Sullivan is the Supervisor of the Orange Team, responsible for metals and total organic carbon (TOC) analyses. Mr. Mark Lambert is the Supervisor of the Green Team, responsible for organics analyses. Ms. Jennifer Constantino is the QA Coordinator and is responsible for the DLS Proficiency Testing programs and laboratory oversight/audit programs. The DLS reporting relationships and functional responsibilities are shown in Table 1.

Table 1. DLS Reporting Relationships

Michael Delaney, Director of Laboratory Services				
Polina Epelman, Lab Manager (Operations)			Steve Rhode, Lab Manager (Client Services)	Jennifer Constantino QA Coordinator
Nancy McSweeney, Supervisor, Red Team	Patricia Sullivan, Supervisor, Orange Team	Mark Lambert, Supervisor, Green Team	Yong Lao, Project Manager (Client Services)	
			Edward Caruso Client Services Coordinator	
			Jim Fitzgerld Supervisor, Violet Team	
Total Solids	Metals, TOC	Organic Contaminants	Sample Management	Performance Testing, Oversight and Document Control

Normandeau Associates, Inc. Ms. Ann Pembroke is the HOM Project Manager for Normandeau. She is responsible for the overall performance of the HOM benthic, fish, and shellfish contract. Mr. Eric Nestler is the Normandeau Assistant Project Manager. The Normandeau Quality Assurance Officer for the project is Dr. Robert Hasevlat. For this task, Dr. Hasevlat is responsible for reviewing data reports submitted by ENQUAD and QA Statements submitted by DLS for completeness and adherence to the Benthic QAPP (Nestler *et al.* 2011.)

Azimuth Geo Services Mr. J. Scott Laswell is the Registered Professional Land Surveyor at Azimuth Geo Services, the contract laboratory that performs grain size analysis.

BAL Laboratory Ms. Darlene Capuano is the Laboratory Director for BAL Laboratory, the contract laboratory that performs *Clostridium perfringens* analysis.

The key contacts at MWRA and Normandeau are shown in Figure 1. Addresses, telephone numbers, and email addresses are given in Table 2.

Table 2.	**Contact Information**			
Name	Title/Role	Location	email	Phone
Darlene Capuano	Laboratory Director	BAL[1]	www.ballaboratory.com DCapuano[at]thielsch.com	401-785-0241
Edward Caruso	Client Services Coordinator	DLS[2]	edward.caruso[at]mwra.state.ma.us	617-660-7807
Jennifer Constantino	QA Coordinator (Yellow)	DLS	jennifer.constantino[at]mwra.state.ma.us	617-660-7808
Mike Delaney	Laboratory Director	DLS	mike.delaney[at]mwra.state.ma.us	617-660-7801
Polina Epelman	Laboratory Manager (Red, Orange, Green)	DLS	polina.epelman[at]mwra.state.ma.us	617-660-7802
Erik Fel'Dotto	Field Supervisor	Normandeau[3]	efeldotto[at]normandeau.com	603-926-7661
Jim Fitzgerald	Team Supervisor (Violet)	DLS	james.fitzgerald[at]mwra.state.ma.us	617-660-7851
Robert Hasevlat	QA Director	Normandeau	rhasevlat[at] normandeau.com	603-637-1142
Doug Hersh	EM&MS Database Administrator	ENQUAD[4]	douglas.hersh[at]mwra.state.ma.us	617-788-4945
Kenneth Keay	Program Manager ENQUAD/Operations Benthic Project Manager	ENQUAD	kenneth.keay[at]mwra.state.ma.us	617-788-4947
Mark Lambert	Team Supervisor (Green)	DLS	mark.lambert[at]mwra.state.ma.us	617-660-7817
Yong Lao	Project Manager	DLS	yong.lao[at]mwra.state.ma.us	617-660-7841
J. Scott Laswell	Registered Professional Land Surveyor	Azimuth[5]	scott-baseline[at]austin.rr.com	512-844-7448
Wendy Leo	EM&MS Manager	ENQUAD	wendy.leo[at]mwra.state.ma.us	617-788-4948
Nancy McSweeney	Team Supervisor (Red)	DLS	nancy.mcsweeney[at]mwra.state.ma.us	617-660-7846
Eric Nestler	HOM 8 Assistant Project Manager	Normandeau	enestler[at] normandeau.com	603-637-1146
Ann Pembroke	HOM 8 Project Manager	Normandeau	apembroke[at]normandeau.com	603-637-1169
Steve Rhode	Laboratory Manager (Violet)	DLS	steve.rhode[at]mwra.state.ma.us	617-660-7803
Pat Sullivan	Team Supervisor (Orange)	DLS	patricia.sullivan[at]mwra.state.ma.us	617-660-7838

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[3] Normandeau Associates Inc., 25 Nashua Road, Bedford, NH 03110. 603-472-5191

[4] Environmental Quality Department, MWRA, 100 First Avenue, Boston, MA 02129, 617-788-4941

[5] Azimuth Geo Services, 8333 Cross Park Drive, Austin, TX 78754

1.2 Communication Plan

Mr. Kenneth Keay is the primary contact with the monitoring prime consultant Normandeau on technical issues. Mr. Steve Rhode is DLS' primary contact with ENQUAD. HOM project meetings are held as needed, as conference calls or at MWRA in the Charlestown Navy Yard. Dr. Yong Lao will attend these calls/meetings when sediment chemistry analysis is on the agenda. DLS holds an internal weekly scheduling and coordination meeting on Tuesdays, which are attended by the DLS Lab Managers, Supervisors, and support staff.

Communication between DLS and Normandeau staff at all levels of the team is encouraged and it is important to keep ENQUAD informed. Email is the primary day-to-day communication method (Table 3).

Table 3. Email cc: List

If the subject is...	*Copy the email to...*
Any	**Kenneth Keay, Steve Rhode, Yong Lao**
Transfer of samples	Erik Fel'Dotto, Jim Fitzgerald (Violet)
Data interpretation	Kenneth Keay, Eric Nestler
Laboratory technical issues	Relevant DLS Team Supervisor(s): ▪ M. Lambert (Green-organics) ▪ P. Sullivan (Orange-metals, TOC) ▪ N. McSweeney (Red- solids) Polina Epelman, Steve Rhode Eric Nestler (issues affecting data interpretation or data quality)
Data management/database	Wendy Leo
Cost/schedule	Kenneth Keay, Mike Delaney Ann Pembroke (issues affecting cost/schedule of Normandeau contract)
Quality assurance	Mike Delaney, Jennifer Constantino, Wendy Leo, Robert Hasevlat (issues affecting data quality not resolved internal to DLS)

The individuals listed in Table 3 take responsibility for forwarding the email to any other relevant staff not on the cc: list. If time is of the essence or if emails fail to produce a response, a telephone call is appropriate. Conversations/contacts affecting scope, schedule, or significant technical issues should be documented in email or memoranda summarizing key items discussed, decisions made, and any actions to be taken.

If expected samples are missing, the DLS Violet Team will immediately notify the Normandeau Field Sample Custodian, Mr. Erik Fel'Dotto, as well as Mr. Steve Rhode, and Mr. Kenneth Keay.

Changes to the number of planned samples should be communicated to the Violet Team, Mr. Steve Rhode, and Mr. Kenneth Keay in advance. It may occur that unusual environmental conditions lead to a decision during field sampling to collect extra samples. In this case, the field team should notify the Violet Team before delivering the samples if possible. If this is not possible, the fact that there are extra samples should be clearly indicated on the chain-of-custody forms to avoid sample mix-ups.

DLS staff usual work hours are 7 am – 3 pm.

Plans for sample custody and transfer are described in Section 2.3.

1.3 Project Background

The background of the HOM project can be found in the QAPP for Benthic Monitoring (Nestler *et al.* 2011).

Samples for sediment chemistry analyses are collected by the HOM consultant and analyzed by DLS. This QAPP describes the quality system implemented for analytical procedures that are performed for the HOM project by the MWRA DLS.

In 2006, Azimuth Geo Services and BAL Laboratory were contracted by MWRA to perform grain size and *Clostridium perfringens* analyses, respectively. This QAPP also includes information pertaining to these contract laboratories.

1.4 Project Description and Schedule

Harbor and Outfall Monitoring (HOM) Project benthic surveys have been conducted since 1991 and are scheduled to continue through 2013. The benthic QAPP (Nestler *et al.* 2011) describes activities specific to the benthic surveys of Massachusetts Bay and Cape Cod Bay, and of Boston Harbor, conducted annually.

MWRA's benthic studies include: (1) monitoring the recovery of the benthic communities in Boston Harbor and (2) obtaining data on the communities and sediment quality at sites in Massachusetts Bay and Cape Cod Bay between 1992 and 2013.

The principal aim of the Harbor studies is documentation of continuing recovery of benthic communities in areas of Boston Harbor in response to decreases in wastewater discharges. The Harbor recovery monitoring includes evaluation of local and area-wide changes in the Boston Harbor system that have resulted from (1) improvements in wastewater treatment practices (*e.g.*, cessation of sludge discharge and conversion from primary to full secondary treatment),

(2) diversion of effluent to the new ocean outfall, and (3) improvements to (CSO) control systems.

Outfall studies include monitoring the response of benthic communities in Massachusetts and Cape Cod Bays to effluent discharge that began in September 2000. This monitoring program focuses most intensely on nearfield sites in western Massachusetts Bay (0 to 8 km from the outfall), where changes in water and sediment quality were predicted to occur following initiation of the discharge. Farfield areas (typically >8 km from the outfall), which serve primarily as reference areas for the nearfield, are also examined as part of the monitoring studies. Such sites can become monitoring stations if the discharge is shown to affect sites a distance from the diffuser.

Relevant to this QAPP, the benthic studies include sediment sampling in the Harbor and Bays for analysis of sedimentary organic matter content and chemical contaminant concentrations. Sedimentary physical characteristics and sewage tracer levels are also measured by other parts of the Harbor and Outfall Monitoring Program. The present status and variability of the benthic environmental quality within the Harbor and Massachusetts Bays system is evaluated by examination of the interrelationships among these parameters. Particular importance is placed on the rapid evaluation of benthic data with respect to monitoring thresholds described in the Contingency and Outfall Monitoring Plans (MWRA 2001, 2010).

Eleven stations in the nearfield and three stations in the farfield (see Table 4) will be sampled in August of each year. In 2011 (and every third year subsequently), samples are analyzed for chemical constituents including polyaromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, metals, and total organic carbon (TOC) , grain size, and *Clostridium perfringens*. Harbor samples, and Massachusetts Bay samples in 2012 and 2013, are analyzed for total organic carbon, grain size, and *Clostridium perfringens* only. Samples collected at each location (relevant to this QAPP) are listed in Table 4 and total number of samples for 2011-2013 is listed in Table 5.

Table 4. Benthic Station Sampling and Replication

Station group name	Stations	Replication: chemistry	Replication: TOC
Harbor traditional (9 stations)	T01, T02, T03, T04, T05A, T06, T07, T08, C019	1	1
Bay (14 stations) (FF01A, FF04, and FF09 are "farfield" stations)	FF01A, FF04, FF09, FF12, NF04, NF10, NF12, NF13, NF14, NF17, NF20, NF21, NF22, NF24	1 (2011 only)	1

Table 5. Parameters Measured, Units, and Number of Samples per Survey

Parameter	Reporting Units	# Samples (2011)	# Samples (2012-2013)
Percent dry weight	%	9x1 + 14x1 = 23	9x1 + 14x1 = 23
TOC	% dry wt.	23	23
Metals	µg/g	14x1 = 14	0
PCBs	µg /kg	14	0
PAHs	µg /kg	14	0
Pesticides	µg/kg	14	0
Grain Size	% dry wt. by class	23	23
Clostridium perfringens	#/100 mL	23	23

1.5 Quality Objectives and Criteria for Measurement Data

The parameters measured, the concentration reporting units, and the number of samples taken per survey is listed in Table 5.

1.5.1 Quality Objectives

Data quality objectives are as follows:

- To ensure that parameters measured adequately describe the effects of effluent and CSO discharge on contamination of Boston Harbor and Massachusetts Bay sediments, and the quality of these sediments as habitat, and

- To ensure that sample results are representative of the location sampled and are accurate.

1.5.2 Measurement Performance Criteria

The objectives are met by examining data collected on BMBSOFT and BHSOFT surveys to quantify chemical concentrations in the sediments of the receiving waters of interest; by analyzing laboratory QC sample to determine precision and accuracy, comparability, representativeness, sensitivity, and completeness; by analyzing laboratory replicates to ensure reproducibility of results; and by repeated measurements collected at the same locations over time to quantify the variability of results at each station. Definitions of quality control samples are provided in Section 2.4.2.

1.5.2.1 Precision and Accuracy

Precision and accuracy of laboratory procedures are ensured by the analysis of quality control (QC) samples including procedural blanks, prepared standards, standard reference materials (SRMs), where available, Laboratory Control Samples (LCS), and laboratory spikes and duplicates, as applicable. Table 6 lists the desired precision, accuracy, and detection limit goals for each parameter being measured. QC samples to be analyzed in the laboratory to assess precision and accuracy are listed in Table 9.

1.5.2.2 Comparability

Data is directly comparable to results obtained previously at the same or similar sites in Massachusetts Bay, Cape Cod Bay, and Boston Harbor (see Nestler *et al.* 2011) because field program design and analytical procedures are similar or identical. In addition, the use of written standardized procedures ensures that sample preparation and analyses are comparable throughout the project and with other projects.

To verify that data generated by DLS are comparable to those generated during earlier years of the HOM project, an inter-comparison study was performed in 2003. The results of the study showed that the data were comparable.

Reporting units for concentrations follow standard convention for most oceanographic studies. (See Table 5). Note however that surrogate recovery values are in percent recovery, but the units shown by LIMS are µg/kg.

1.5.2.3 Representativeness

Representativeness is addressed primarily in sampling design. The sampling practices and laboratory measurements that are performed during the benthic monitoring have already been used in many systems to characterize marine sediment quality and are, therefore, considered to yield data representative of the study area. Representativeness is also ensured by proper handling, storage (including appropriate preservation and holding times), and analysis of samples so that the material analyzed reflects the material collected as accurately as possible.

Deviations from the analytical scheme described in this QAPP are noted in the laboratory records associated with analytical batches and in the QA statements.

1.5.2.4 Sensitivity

Sensitivity is the capability of methodology or instrumentation to discriminate among measurement responses for quantitative differences of a parameter of interest. The method detection limits (MDLs) (Table 6) provide the sensitivity goals for the procedures. With the exception of PAHs, the MDLs listed in Table 6 are comparable to those listed in Nestler *et al.* 2011.

1.5.2.5 Completeness

It is expected that 100% of the samples collected and intended for analysis be analyzed. However, a sample loss of <5% for the entire project does not compromise the objectives of the project.

1.6 Special Training Requirements and Certification

Organic contaminant measurements, metals analysis, and total solids analysis for the HOM Benthic study use routine laboratory analyses (for sediment samples) and data validation. Therefore, specialized training is not required. Metals (except mercury) preparation of sediment samples for the HOM project however, involves a digestion with hydrofluoric acid, which requires specialized training. Once analysts have undergone the proper training in handling, storing, preparing, and analyzing sediment samples as specified in MWRA's Department of Laboratory Services Quality Assurance Management Plan (QAMP, DCN #5000, Section 3.0), they can be certified to perform the analysis.

Table 6. Desired Precision, Accuracy, and MDL for each Parameter based on Quality Objectives

Parameter	Lab Precision[8]	Accuracy[8]	Blank[8] Cleanliness	MDL[1]	RL[4,6]
% dry weight	≤ 10% RPD	89%-115%	< 0.0125%	NA	NA
TOC	≤ 25% RPD	±5% PD	≤ 10% of lowest sample concentration	2.46 mg/L 0.00492%	≤ 10% of lowest sample concentration
Metals (MDL/RL source)	≤ 25% RPD if value > 5*MDL	≤ 20% PD vs. SRM certified values	≤ 10% of the lowest sample concentration		
Aluminum (FAA)				17 µg/g	≤143 µg/g
Iron (FAA)				5 µg/g	≤15 µg/g
Silver (GFA)				0.006 µg/g	≤0.032 µg/g
Cadmium (GFA)				0.002 µg/g	≤0.01 µg/g
Chromium (FAA)				3 µg/g	≤15 µg/g
Copper (FAA)				3 µg/g	≤2.5 µg/g
Mercury (CVA)				0.025 µg/g	≤0.025 µg/g
Nickel (GFA)				0.05 µg/g	≤0.25 µg/g
Lead (GFA)				0.2 µg/g	≤1 µg/g
Zinc (FAA)				1 µg/g	≤4 µg/g
PCBs	≤ 30% RPD	≤ 35% vs. SRM range	≤ RL[4] (0.200 ng/g)		
2,4-Cl_2(8)				0.0299 ng/g	0.2 ng/g
2,2',5-Cl_3(18)				0.0280 ng/g	0.2 ng/g
2,4,4'-Cl_3(28)				0.0288 ng/g	0.2 ng/g
2,2',3,5'-Cl_4(44)				0.0233 ng/g	0.2 ng/g
2,2',5,5'-Cl_4(52)				0.0278 ng/g	0.2 ng/g
2,3',4,4'-Cl_4(66)				0.0301 ng/g	0.2 ng/g
3,3',4,4'-Cl_4(77)				0.0404 ng/g	0.2 ng/g
2,2'4,5,5'-Cl_5(101)				0.0189 ng/g	0.2 ng/g
2,3,3',4,4'-Cl_5(105)				0.0280 ng/g	0.2 ng/g
2,3',4,4'5-Cl_5(118)				0.0335 ng/g	0.2 ng/g
3,3',4,4',5-Cl_5(126)				0.0362 ng/g	0.2 ng/g
2,2',3,3,4,4'-Cl_6(128)				0.0303 ng/g	0.2 ng/g
2,2',3,4,4',5-Cl_6(138)				0.0248 ng/g	0.2 ng/g
2,2'4,4',5,5'-Cl_6(153)				0.0269 ng/g	0.2 ng/g
2,2'3,3,4,4',5-Cl_7(170)				0.0253 ng/g	0.2 ng/g
2,2',3,4,4',5,5'-Cl_7(180)				0.0275 ng/g	0.2 ng/g
2,2',3,4,5,5',6-Cl_7(187)				0.0270 ng/g	0.2 ng/g
2,2',3,3',4,4',5,6-Cl_8(195)				0.0431 ng/g	0.2 ng/g
2,2',3,3'4,4',5,5',6-Cl_9(206)				0.0394 ng/g	0.2 ng/g
Decachlorobiphenyl-Cl_{10}(209)				0.0347 ng/g	0.2 ng/g
PAH[7]	≤ 30% RPD	≤ 35% vs. SRM range	≤ RL[4] (0.500 ng/g)		
Naphthalene				0.190 ng/g	0.5 ng/g
C_1-naphthalenes				0.190ng/g	0.5 ng/g
C_2-naphthalenes				0.190 ng/g	0.5 ng/g
C_3-naphthalenes				0.190 ng/g	0.5 ng/g
C_4-naphthalenes				0.190 ng/g	0.5 ng/g
1-Methylnaphthalene				0.062 ng/g	0.5 ng/g
2-Methylnaphthalene				0.116 ng/g	0.5 ng/g
2,6-Dimethylnaphthalene				0.111 ng/g	0.5 ng/g
Acenaphthylene				0.067 ng/g	0.5 ng/g
Acenaphthene				0.046 ng/g	0.5 ng/g
2,3,5-Trimethylnaphthylene				0.097 ng/g	0.5 ng/g
Fluorene				0.073 ng/g	0.5 ng/g
C_1-fluorenes				0.073 ng/g	0.5 ng/g
C_2-fluorenes				0.073 ng/g	0.5 ng/g
C_3-fluorenes				0.073 ng/g	0.5 ng/g
Anthracene				0.060 ng/g	0.5 ng/g
Phenanthrene				0.079ng/g	0.5 ng/g
C_1-phenanthrenes/anthracene				0.079ng/g	0.5 ng/g
C_2-phenanthrenes/anthracene				0.079ng/g	0.5 ng/g

Table 6. Desired Precision, Accuracy, and MDL for each Parameter based on Quality Objectives

Parameter	Lab Precision[8]	Accuracy[8]	Blank[8] Cleanliness	MDL[1]	RL[4,6]
C_3-phenanthrenes/anthracene				0.079 ng/g	0.5 ng/g
C_4-phenanthrenes/anthracene				0.079 ng/g	0.5 ng/g
1-Methylphenanthrene				0.124 ng/g	0.5 ng/g
Dibenzothiophene				0.097 ng/g	0.5 ng/g
C_1-dibenzothiophenes				0.097ng/g	0.5 ng/g
C_2-dibenzothiophenes				0.097ng/g	0.5 ng/g
C_3-dibenzothiophenes				0.097 ng/g	0.5 ng/g
Fluoranthene				0.055 ng/g	0.5 ng/g
Pyrene				0.044 ng/g	0.5 ng/g
C_1-fluoranthenes/pyrenes				0.044 ng/g	0.5 ng/g
C_2-fluoranthenes/pyrenes				0.044 ng/g	0.5 ng/g
C_3-fluoranthenes/pyrenes				0.044 ng/g	0.5 ng/g
Benzo(a)anthracene				0.062 ng/g	0.5 ng/g
Chrysene				0.055 ng/g	0.5 ng/g
C_1-chrysene				0.055 ng/g	0.5 ng/g
C_2-chrysene				0.055 ng/g	0.5 ng/g
C_3-chrysene				0.055 ng/g	0.5 ng/g
C_4-chrysene				0.055 ng/g	0.5 ng/g
benzo(b)fluoranthene				0.029 ng/g	0.5 ng/g
benzo(k)fluoranthene				0.083 ng/g	0.5 ng/g
benzo(a)pyrene				0.033 ng/g	0.5 ng/g
dibenzo(a,h)anthracene				0.074 ng/g	0.5 ng/g
benzo(g,h,i)perylene				0.061 ng/g	0.5 ng/g
Indeno(1,2,3-c,d)pyrene				0.044 ng/g	0.5 ng/g
Perylene				0.037 ng/g	0.5 ng/g
Biphenyl				0.050 ng/g	0.5 ng/g
benzo(e)pyrene				0.072 ng/g	0.5 ng/g
Dibenzofuran				0.036 ng/g	0.5 ng/g
Benzothiazole				0.129 ng/g	0.5 ng/g
Pesticides	≤ 30% RPD	≤ 35% vs. SRM range	≤ RL[4]		
Hexachlorobenzene				0.0920 ng/g	0.2 ng/g
Lindane (gamma- BHC)				0.0839 ng/g	0.2 ng/g
Heptachlor				0.1631 ng/g	0.2 ng/g
Aldrin				0.0803 ng/g	0.2 ng/g
Heptachlorepoxide				0.0366 ng/g	0.2 ng/g
Alpha-chlordane				0.0158 ng/g	0.2 ng/g
Trans-Nonachlor				0.0213 ng/g	0.2 ng/g
Dieldrin				0.1845 ng/g	0.2 ng/g
Endrin				0.0612 ng/g	0.2 ng/g
Mirex				0.0226 ng/g	0.2 ng/g
2,4’-DDD				0.0322 ng/g	0.2 ng/g
4,4’-DDD				0.0266 ng/g	0.2 ng/g
2,4’-DDE				0.0253 ng/g	0.2 ng/g
4,4’-DDE				0.0294 ng/g	0.2 ng/g
2,4’-DDT				0.0303 ng/g	0.2 ng/g
4,4’-DDT				0.0277 ng/g	0.2 ng/g
DDMU				0.0250 ng/g	0.2 ng/g
Gamma-Chlordane				0.0325 ng/g	0.2 ng/g
Cis-Nonachlor				0.0131 ng/g	0.2 ng/g
Oxychlordane				0.0790 ng/g	0.2 ng/g
Grain Size	≤25% RPD	NA	NA	2%	
Clostridium perfringens	5%	NA	NA	NA	NA

[1] MDL = method detection limit. The actual MDL may be updated periodically. Contact the MWRA Central Laboratory for the most current MDL information.

MDL ADOCs: PES/PCB/PAH: 2004-29; FAA:2010-26; GFA: 96-05, 96-06, 97-07, MDL study 1995-96 for Pb (conversion to dry weight based on 0.05 Lfinal volume, 0.7 g initial weight, and 100% solids); CVA: 95-27 (conversion to dry weight based on 0.1 L final volume, 0.2 g initial weight, and 100% solids); TOC: 2008-62.

[2] Relative Percent Difference (RPD)% = $\left| \text{(replicate 1 - replicate 2)/(replicate 1 + replicate 2)/2} \right|$ x 100.

[3] Percent Difference (PD)% = [(true concentration – measured concentration)/true concentration] x 100.

[4] Reporting Limit (RL): The RL is the typical reporting limit, which is based on the low point of the calibration curve. Concentrations below the RL are reported, so long as all identification criteria are met.

[5] For organics SRM: If the detected value falls within the SRM certified range, then PD=0. If the detected value falls outside the SRM certified range, then the PD is determined against either the upper or lower limit of the range.

[6] Metals RLs for FAA are documented in ADOC 2010-26. Metals RLs for GFA are derived from ADOCs 96-05, 96-06, 97-07, MDL study 1995-96. These MDL values are converted to dry weight values based on 0.7 g initial weight, 100% solids, and 50 mL final volume (except mercury, which has a final volume of 100 mL and an initial weight of 0.2 g). These converted values are then multiplied by 5 to arrive at the reporting limit. The Hg RL is equal to the MDL (ADOC 95-27). PES/PCB RLs are documented in ADOC 2010-27. PAH RLs are documented in ADOC 2010-28.

[7] MDL concentrations for alkyl homologues are based on the MDL of the unsubstituted, parent compound.

[8] ADOCS for QC Limits: Metals (FAA): 2010-26; Metals (GFA): 2010-26; Metals (CVA): 2010-26; PES/PCB Congeners: 2010-27; PAH: 2010-28; TS: 2001-81; TOC: 2003-21

1.7 Documentation and Records

Documents and records are created and maintained according to the guidance and requirements found in the following DLS documents: QAMP, Section 12.0 (DCN #5000), SOP (DCN #5006), “Guidance for Writing, Revising and Approving Standard Operating Procedures”, and SOP (DCN #5007), “Procedures and Guidelines for the Handling, Storage and Archiving of Hardcopy and Electronic Records.”

1.7.1 Document Control

MWRA DLS maintains documents relevant to laboratory analysis activities and entry of data into LIMS. The DLS document retention system includes all logbooks, raw data, instrument reports, calculated data, and COC forms.

The pertinent documents applicable to the HOM analyses are this QAPP, the DLS QAMP (DCN #5000) and the analysis SOPs (See Table 8). The guidance for the control of DLS’ SOPs is set forth in the DLS SOP DCN: 5006, “Guidance for Writing, Revising, and Approving Standard Operating Procedures”. After revision and approval, all SOPs are available electronically to the respective Team/Supervisor/Analyst. A copy of the most current analysis SOP is kept in the lab area where the analysis is being performed and on the MWRA Intranet.

1.7.2 Analysis Records

All data are recorded initially into bound laboratory logbooks, onto established data forms or onto electronic file, where applicable. Sampling logs associated with custody and tracking are held in the custody of the Violet Team Supervisor responsible for sample management. Field measurements and laboratory analytical results are subsequently entered into LIMS.

1.7.3 Records Retention and Storage

All hardcopy records are stored, secured, and protected in appropriate locations either in the Team areas, the QA File area, or in the DLS Record Retention Room. Subsequently, hard copy records are sent and archived at MWRA's Central Record Storage location. All records are kept for a period of fifteen years. The guidance for record handling is set forth in the DLS SOP DCN: 5007, "Procedures and Guidance for the Handling, Storage, and Archiving of Hardcopy and Electronic Records".

1.7.4 LIMS Electronic Records

All records and data stored in LIMS are backed up daily (Monday through Friday) by MWRA's MIS department. Backups are sent to an off-site location where they are kept for the appropriate retention period. Daily backups are kept for a five week rotating cycle. Monthly backups are kept for a period of two years, and every year-end a backup is done which is performed and retained for a period of 15 years.

1.7.5 Records Managed by ENQUAD

ENQUAD maintains all documents relevant to data loading into EM&MS, and to data reviews.

2.0 MEASUREMENT/DATA ACQUISITION

2.1 Sampling Process Design (Experimental Design)

2.1.1 Scheduled Project Activities, Including Measurement Activities

The BMBSOFT and BHSOFT studies are performed on an ongoing basis as specified in Nestler *et al.* 2011. They have been ongoing, with some changes in sampling frequency and sampling locations, since 1991 or 1992, thus including about nineteen years of monitoring. Each currently includes one sampling event per year.

2.1.2 Design Rationale

The objective of the BMBSOFT and BHSOFT studies is to measure sediment quality changes after wastewater discharges were transferred offshore to Massachusetts Bay. The evaluation of sediment quality changes due to the transfer of discharges offshore is assessed through measurement of organic carbon content and of chemical contaminant concentrations, among others. Outfall effects are most likely at the nearfield stations. Farfield stations serve as reference stations as well as documenting the spatial extent of any change due to the outfall. Harbor stations show recovery from past effluent and sludge discharges to the harbor, and changes due to changes in other sources (e.g. CSOs).

2.1.3 Design Assumptions

Because sediment properties in the nearfield are known to be spatially heterogeneous, stations close to one another are assumed to be replicates of one another. Conversely, Boston Harbor stations are located in distinct areas of the harbor and were selected to be representative of sediment quality for each of these areas. It is assumed that the sediment properties change only gradually over time, except in response to large storms, so annual or biennial sampling is sufficient to characterize the distribution of contaminants and the quality of the sediment as benthic habitat.

2.1.4 Procedures for Locating and Selecting Environmental Samples

The choice of sampling locations is discussed in the Ambient Monitoring Plan (MWRA 2010) and in the QAPP for Benthic Monitoring (Nestler *et al.* 2011).

2.1.5 Classification of Measurements as Critical or Non-critical

All measurements collected as part of the BWQM surveys are considered critical due to the requirement in MWRA's discharge permit to conduct the measurements described in the Ambient Monitoring Plan (MWRA 2010).

2.2 Sampling Methods Requirements

2.2.1 Sample Collection, Preparation, Presevation Procedures

Samples for each suite of analytes are collected in Kynar coated Ted Young-modified Van Veen grab samplers as described in Nestler *et al.* 2011. The upper 0-2 cm is subsampled with a Kynar-coated scoop. The sample bottles and the associated analytes are shown in Table 7, along with field preservation method and holding time. DLS provides all sample containers containers and bar-coded sample labels. All other field supplies are provided by Normandeau.

2.2.2 Sampling/Measurement System Failure Response and Corrective Action Process

Corrective action in the field is covered in Nestler *et al.* 2011.

From time to time, circumstances/conditions (e.g., broken or contaminated sample containers,) may be identified prior to check-in or prior to analysis, which, in turn, may dictate that a corrective action be initiated. The corrective action process/procedures are summarized in Section 3.0 of this document and Section 11.0 of the DLS QAMP (DCN #5000). If an anomaly is identified after analysis (e.g. samples were matched incorrectly with identifying information) but prior to approval in LIMS, changes to the data in LIMS may be made by a supervisor or analyst with validation privileges and a corrective action may be initiated. If an anomaly is noticed after approval in LIMS a DAIR (Data Anomaly Investigation Request) must be initiated. See Section 2.8.7 for the DAIR process. Again, a corrective action may be initiated.

Table 7. Sample Collection and Storage

Parameter	Sample Mass (Target) (g)[a]	Sample Containers[b]	Shipboard Processing/ Preservation	Maximum Holding Time to Analysis
TOC, % dry weight	50	Clean, labeled glass jar	freeze (-20° C)	28 days from thawing
Metals	100	Clean, tared and labeled I-CHEM container	freeze (-20° C)	6 months to preparation and analysis; Hg holding time is 28 days from thawing
Organic contaminants	125	Clean, labeled glass jar with Teflon-lined cap	freeze (-20° C)	1 year to extract (if samples frozen); 40 days from extraction to analysis
Grain Size	500	Clean plastic bag	Cool (4° C)	28 days
Clostridium perfringens	25	Sterile sample bottles	Cool (4° C)	Not defined.

[a] Sample mass processed for analysis.
[b] Name brand items (*e.g*., I-CHEM) may be substituted with comparable items from a different manufacturer.

2.3 Sample Handling and Custody Requirements

2.3.1 Sampling Equipment, Preservation, and Holding Times Requirements

Samples collected for laboratory analysis are stored on ice in coolers or frozen and holding times (Table 7) are met to ensure the accuracy of results. The temperatures of sample storage units are monitored to verify that holding temperatures are met.

2.3.2 Sample Custody Procedure

The QAPP for benthic studies (*Nestler et al. 2011*) describes sample tracking in the field. Field samples will be assigned IDs by Normandeau. LIMS IDs for analysis will be provided by DLS in advance.

The Chief Scientist is responsible for verifying each Sample ID vs. the COC forms generated prior to delivering the samples to the laboratory. All samples are delivered to the Normandeau Field Sample Custodian, who distributes them to the appropriate laboratory personnel by hand or by Federal Express. Hand-delivery may include direct transfer of samples to DLS personnel at the boat, dock, or lab. All frozen samples that must be shipped are placed on dry ice with protective layers of foam or bubble wrap to ensure samples remain intact and frozen during shipment.

Normandeau field staff will drive the samples to Deer Island a day or two after the survey or ship via FedEx. Coordinating with the DLS HOM Project Manager, the samples can be dropped off or picked up first thing in the morning (0700) during a multi-day survey.

2.3.3 Sample Receipt and Check-in

Upon receipt of the samples, the MWRA DLS Laboratory Sample Management Team (Violet):

- Inspects the samples to verify that:

  (1) integrity is intact (containers are sealed and intact),
  (2) the sample label and custody forms agree, and
  (3) all shipped samples have been received.

- Completes the Normandeau COC forms, and signs the COC form so that transfer of custody of the samples is complete. Any discrepancies between sample labels and the custody forms, and unusual events or deviations from the project QAPP are documented in detail on the COC, and are communicated to the DLS Project Manager who notifies the Normandeau Field Supervisor within 24 hours of receipt. **Note:** The original COC forms are sent to ENQUAD to be forwarded to Normandeau along with the data set and other associated documentation; copies are kept at the DLS Laboratory.

- Checks the samples into LIMS to provide a permanent laboratory record. **Note:** This is accomplished by matching up the BOTTLE_ID with the Labware LIMS text ID. The LIMS text IDs are used throughout the laboratory analysis.

After the samples are received by the DLS laboratory:

- Samples are stored in the secure Sample Bank or a secure freezer at the temperature conditions specified in Table 7. Access to the samples is only allowed to lab personnel, using their electronic pass card, key, or combination lock.

- TOC and metals containers are subsampled for TS analysis, and PAH containers are subsampled for PCB, pesticide, and TS analysis.

- Samples are sent to contract labs for grain size and *Clostridium perfringens* analysis.

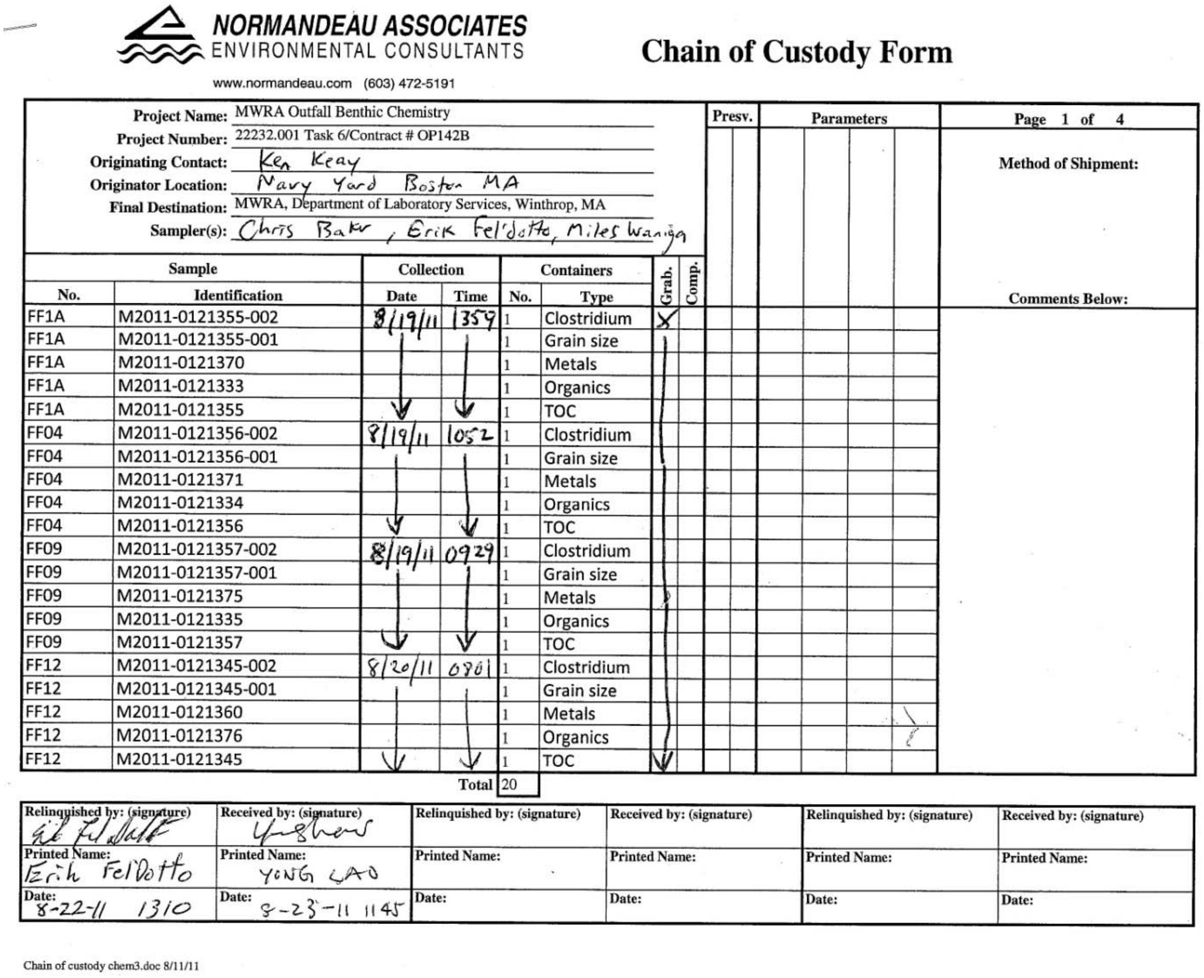
- Samples are stored in the secure Sample Bank or freezer until they are transferred to a member of laboratory staff for analysis. The internal chain of custody is documented in LIMS.

- Internal laboratory documentation in LIMS tracks sample custody and location throughout processing and analysis. Transfer of samples is documented in LIMS, using a password-protected program to document both the person relinquishing the samples as well as the recipient. A copy of the DLS internal LIMS Chain-of-Custody is shown in Figure 3.

- When an analyst is finished with a sample, custody is transferred in LIMS either back to the Sample Bank for archival and or to sample disposal.

- All samples covered by this QAPP are analyzed by the DLS Central Laboratory, BAL Laboratory, and Azimuth Geo Services. The analyses performed by the DLS follow the procedures listed in the various DLS SOPs (Table 8).

- When the results are transferred to the EM&MS database (see Section 4.1.2), ENQUAD EM&MS personnel maps Normandeau's SAMPLE_ID into the SAMPLE_ID field, and the LIMS text_id into the BOTTLE_ID and the LIMS sample_number into the LAB_SAMPLE_ID field.



NORMANDEAU ASSOCIATES
ENVIRONMENTAL CONSULTANTS
www.normandeau.com (603) 472-5191

Chain of Custody Form

Project Name: MWRA Outfall Benthic Chemistry
Project Number: 22232.001 Task 6/Contract # OP142B
Originating Contact: Ken Keay
Originator Location: Navy Yard Boston MA
Final Destination: MWRA, Department of Laboratory Services, Winthrop, MA
Sampler(s): Chris Baker, Erik Fel'dotto, Miles Waniga

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Method of Shipment:

Comments Below:

Sample		Collection		Containers				Presv.	Parameters
No.	**Identification**	**Date**	**Time**	**No.**	**Type**	**Grab.**	**Comp.**		
FF1A	M2011-0121355-002	8/19/11	1359	1	Clostridium	X			
FF1A	M2011-0121355-001	↓	↓	1	Grain size	↓			
FF1A	M2011-0121370	↓	↓	1	Metals	↓			
FF1A	M2011-0121333	↓	↓	1	Organics	↓			
FF1A	M2011-0121355	↓	↓	1	TOC	↓			
FF04	M2011-0121356-002	8/19/11	1052	1	Clostridium	↓			
FF04	M2011-0121356-001	↓	↓	1	Grain size	↓			
FF04	M2011-0121371	↓	↓	1	Metals	↓			
FF04	M2011-0121334	↓	↓	1	Organics	↓			
FF04	M2011-0121356	↓	↓	1	TOC	↓			
FF09	M2011-0121357-002	8/19/11	0929	1	Clostridium	↓			
FF09	M2011-0121357-001	↓	↓	1	Grain size	↓			
FF09	M2011-0121375	↓	↓	1	Metals	↓			
FF09	M2011-0121335	↓	↓	1	Organics	↓			
FF09	M2011-0121357	↓	↓	1	TOC	↓			
FF12	M2011-0121345-002	8/20/11	0801	1	Clostridium	↓			
FF12	M2011-0121345-001	↓	↓	1	Grain size	↓			
FF12	M2011-0121360	↓	↓	1	Metals	↓			
FF12	M2011-0121376	↓	↓	1	Organics	↓			
FF12	M2011-0121345	↓	↓	1	TOC	↓			
			Total	20					

Relinquished by: (signature)	**Received by: (signature)**	**Relinquished by: (signature)**	**Received by: (signature)**	**Relinquished by: (signature)**	**Received by: (signature)**
Printed Name: Erik FelDotto	**Printed Name:** YONG CAO	**Printed Name:**	**Printed Name:**	**Printed Name:**	**Printed Name:**
Date: 8-22-11 1310	**Date:** 8-23-11 1145	**Date:**	**Date:**	**Date:**	**Date:**

Chain of custody chem3.doc 8/11/11

Figure 2 **Normandeau Chain-of-Custody Form**

Internal Chain of Custody Record

Data Date and Time: 12-Feb-2010 10:23

Sample: M2010-0003033-008

Field Name:	X_CUST_SET_(	New Field Value:	2010-02-10 14:32:02
Audit Timestamp (GMT):	10-Feb-2010 19:32:13	Old Field Value:	2010-02-10 10:20:16
Reason:	Internal Chain Of Custody	Changed By:	Charles Blodget

Field Name:	X_CUST_SET_I	New Field Value:	CBLODGET
Audit Timestamp (GMT):	10-Feb-2010 19:32:13	Old Field Value:	Z_LARISSA
Reason:	Internal Chain Of Custody	Changed By:	Charles Blodget

Field Name:	X_CUSTODY_C	New Field Value:	CBLODGET
Audit Timestamp (GMT):	10-Feb-2010 19:32:13	Old Field Value:	Z_LARISSA
Reason:	Internal Chain Of Custody	Changed By:	Charles Blodget

Field Name:	LOCATION	New Field Value:	147-SAMPLE BANK
Audit Timestamp (GMT):	10-Feb-2010 19:32:13	Old Field Value:	316A-WET CHEM NU
Reason:	Internal Chain Of Custody	Changed By:	Charles Blodget

Field Name:	X_CUST_SET_(	New Field Value:	2010-02-10 10:20:16
Audit Timestamp (GMT):	10-Feb-2010 15:21:54	Old Field Value:	
Reason:	Internal Chain Of Custody	Changed By:	Larissa M. Zilitinkevitch

Field Name:	X_CUST_SET_I	New Field Value:	Z_LARISSA
Audit Timestamp (GMT):	10-Feb-2010 15:21:54	Old Field Value:	
Reason:	Internal Chain Of Custody	Changed By:	Larissa M. Zilitinkevitch

Field Name:	X_CUSTODY_C	New Field Value:	Z_LARISSA
Audit Timestamp (GMT):	10-Feb-2010 15:21:54	Old Field Value:	
Reason:	Internal Chain Of Custody	Changed By:	Larissa M. Zilitinkevitch

Field Name:	LOCATION	New Field Value:	316A-WET CHEM NU
Audit Timestamp (GMT):	10-Feb-2010 15:21:54	Old Field Value:	147-SAMPLE BANK
Reason:	Internal Chain Of Custody	Changed By:	Larissa M. Zilitinkevitch

Field Name:	RECEIVED_BY	New Field Value:	CSEAMAN
Audit Timestamp (GMT):	08-Feb-2010 12:49:30	Old Field Value:	
Reason:	No reason specified	Changed By:	Cara Seaman

Field Name:	RECD_DATE	New Field Value:	2010-02-08 07:49:29
Audit Timestamp (GMT):	08-Feb-2010 12:49:30	Old Field Value:	
Reason:	No reason specified	Changed By:	Cara Seaman

Field Name:	LOCATION	New Field Value:	147-SAMPLE BANK
Audit Timestamp (GMT):	08-Feb-2010 12:49:30	Old Field Value:	
Reason:	No reason specified	Changed By:	Cara Seaman

Figure 3 **DLS LIMS Internal Chain-of-Custody Form**

2.4 Analytical Requirements

2.4.1 Analytical Methods

Table 8 summarizes the methods used for sample analysis. The analyses are conducted as described in the DLS SOPs listed, which are based on literature references or EPA methods as indicated in the SOP. Also analyses for grain size are conducted as described in the Azimuth Geo Services method and analyses for *Clostridium perfringens* are conducted as described in the BAL Laboratory method (Section 2.4.1.5 and 2.4.1.6).

The preparation and analysis of samples are described in detail in the DLS Standard Operating Procedures. The comprehensive QA/QC program is described in the DLS' QAMP (DCN #5000). All sample aliquots are taken by mixing in any superficial liquid with the solid portion of the sample before an aliquot is taken.

Calibration procedures for laboratory instruments are summarized in Table 10. All laboratory calibration records are reviewed by analysts and maintained in the laboratory document retention system.

Table 8. Methods for Sediment Sample Analyses to be Conducted by DLS

Parameter	LIMS test code	Units	Instrument	DLS SOP DCN[1] (Based on Reference)
Dry weight	TS--SOGRV	%	NA	#1094
TOC	TOC-SOCIR	% dry wt.	DC-190	#1233
Metals				
Aluminum	AL--SOFAA	mg/kg	FAA	#1193/#1199
Iron	FE--SOFAA		FAA	#1193/#1199
Silver	GFA-SOABS		GFA	#1193/#1150/#1199
Cadmium	GFA-SOABS		GFA	#1193/#1150/#1199
Chromium	CR--SOFAA		FAA	#1193/#1199/#1150
Copper	CU--SOFAA		FAA	#1193/#1199/#1150
Mercury	HG--SOABS		CVA	#1027/ #1049
Nickel	GFA-SOABS		GFA	#1193/#1150/#1199
Lead	GFA-SOABS		GFA	#1193/#1150/#1199
Zinc	ZN--SOFAA		FAA	#1193/#1199
PCBs	PES-SOSIM	µg/kg	GC/MS	#1188/#1173
Polynuclear Aromatic Hydrocarbons (PAH)	PAH-SOSIM	µg/kg	GC/MS	#1188/ #1030
Pesticides	PES-SOSIM	µg/kg	GC/MS	#1188/#1173

[1] DCN= Document Control Number. The SOP revision number is not included in the DCN. Contact the MWRA Central Laboratory for the most current revision number.

2.4.1.1 Organic Chemical Analysis

The MWRA Central Laboratory performs all organic sediment chemistry analyses. Sediment samples are extracted for PAH, chlorinated pesticides, and PCB congeners by following MWRA SOP #1188, *Combined Sediment Sample Extraction by Sonication for PAH, Pesticides, and PCB Congener Analyses*. This extraction method utilizes sonication, and is based on EPA Method 3550B (USEPA, 1997). Approximately 10 g - 20 g of sediment is mixed with sodium sulfate and is serially extracted with methylene chloride (DCM) using sonication techniques. (Approximately 25 g of the original sample is also taken for dry weight determination by the Red Team.) The sample is weighed into an extraction vessel, mixed with the appropriate amount of sodium sulfate to achieve a free-flowing consistency, and spiked with the surrogate compounds. Methylene chloride is added and the sample is sonicated using the ultrasonic disruptor. The extract is decanted into an Erlenmeyer flask through a powder funnel containing glass wool and sodium sulfate to remove any water and sediment particles. After each extraction (total of three solvent additions) the filtered solvent is combined in the flask. The combined extracts are processed through a silica gel cartridge and concentrated to 1 mL using the DryVap automatic concentrator technique. The extract may then be additionally cleaned using activated copper to remove elemental sulfur, if present. The post-cleanup extracts are concentrated to 1.0 mL - 2.0 mL and split 50:50 for analysis by the PAH and pesticide/congener methods.

Sample extracts are analyzed for PAH compounds by gas chromatography/mass spectrometry (GC/MS) operating in the selected-ion-monitoring (SIM) mode, using a 30m Rtx-5 column (or equivalent) and an Agilent 5973 detector (or equivalent), according to MWRA SOP #1030, *Trace Level Polynuclear Aromatic Hydrocarbon Analysis by Gas Chromatography/Mass Spectrometry using Selected Ion Monitoring (GC/MS SIM)*. The PAH compounds are quantified using the internal standard method. Sample data are not surrogate corrected prior to entry into the LIMS system, but guidance regarding the surrogate compounds is provided so that the client may later perform surrogate correction if desired. Concentrations of the substituted PAH homologues are determined by summing the total area of each homologue and using the response factor of the parent PAH compound.

Pesticides and PCB congeners are analyzed by gas chromatography/mass spectrometry (GC/MS) operating in the selected-ion-monitoring (SIM) mode, using a 60m Rtx-5 column (or equivalent) and an Agilent 5973 detector (or equivalent), according to MWRA SOP #1173, *Trace Level PCB Congener and Pesticide Analysis by Gas Chromatography/Mass Spectrometry using Selected Ion Monitoring (GC/MS SIM)*. Two separate analyses are performed, one to determine the pesticide compounds and one for the PCB congeners. Concentrations for all target analytes are determined using the internal standard method. Sample data are not surrogate-corrected prior to entry into the LIMS system, but guidance regarding the surrogate compounds is provided so that the client may later perform surrogate correction if desired.

All PAH, PCB congener, and pesticide results are reported in micrograms per kilogram (µg/kg) on a dry weight basis.

2.4.1.2 Metal Analysis

The MWRA Central Laboratory performs metals digestions and analyses for Ag, Al, Cd, Cr, Cu, Fe, Ni, Pb, and Zn. Sediment samples are digested using a hydrochloric/nitric/hydrofluoric digestion according to DLS SOP #1193, *Preparation for Analysis of Total Elements in Sediment Samples by Microwave Digestion*. A 0.7 g of solid sample is combined with 5 mL HNO_3, 2.5 mL HCl, and 4 mL HF in a Teflon microwave vessel. Samples are cold-digested in this acid mixture overnight. Samples are microwave digested for approximately 30 minutes. After heating and cooling, samples are filtered through Whatman #541 filters and rinsed with Milli-Q (Barnstead DI) water (final volume is 50 mL). After rinsing, filter paper is transferred back to the digestion vessel and the digestion is repeated (an additional 5 mL HNO_3, 2.5 mL HCl, and 4 mL HF is added and samples are filtered again) to further digest the sample. This extract is filtered through a Whatman #541 filter and rinsed with Milli-Q water to a final volume of 50 mL. Then 5 mL concentrated HNO3 and 2.5 mL concentrated HCL is added to the filter papers and swirled to mix, 0.5 g solid boric acid and 15 mL deionized water are added, and samples are microwaved again and allowed to cool. The extract is filtered through a Whatman #541 filter and rinsed to a final volume of 50 mL.Digestates are analyzed either by GFA (DLS SOP #1150, *Graphite Furnace Atomic Absorption Spectroscopy*) or FAA (DLS SOP 1199, *Analysis of Sediments and Tissues by Flame AA*). Acceptance criteria for the calibration are listed in Table 10. Results are reported as µg/g dry-weight.

CVAA Analysis of Hg- Samples are digested and analyzed by the MWRA Central Laboratory for Hg using cold-vapor atomic absorption spectroscopy (CVAA) according to DLS SOP #1027, *Digestion of Solid Samples for Mercury Analysis* and DLS SOP #1049, *Mercury analysis by Cold Vapor Atomic Absorption Spectroscopy (CETAC M6000A).* A 200 mg aliquot is leached with 15 mL dilute HNO_3 and HCl in a waterbath for 2 minutes. Cooled samples are diluted to 50 mL and oxidized with $KMnO_4$ in a waterbath at 95º C for 30 minutes. Deionized water is added to bring the final sample volume to 100 mL. The digested sample is mixed with a reducing agent in-line to release elemental Hg vapor. Hg is quantified by atomic absorption at 254 nm. Acceptance criteria for the calibration are listed in Table 10. Results are reported as µg/g dry-weight.

2.4.1.3 Total Organic Carbon Analysis

Samples are processed and analyzed by the MWRA Central Laboratory according to DLS SOP #1233, *Total Organic Carbon in Sediment by Combustion with Infrared Detection (Teledyne Apollo 9000)*. Sediment samples for TOC analysis are thawed in the refrigerator. A portion of the wet sample (approximately 250 mg) is transferred to a scintillation vial. The sample is treated with 5 mL of 10% HCl to remove inorganic carbon and the sample is heated in a water bath at 70° C for 10 minutes. The analyzer operates through the high-temperature conversion of all carbon in the treated sample to carbon dioxide in the presence of oxygen. The carbon dioxide is quantified by infrared detection. Acceptance criteria for the calibration are listed in Table 10. Results are reported as %C dry-weight.

2.4.1.4 Total Solids (% Dry Weight)

Percent dry weight is performed by the MWRA Central Laboratory. For this analysis, percent dry weight is determined following SOP #1094, *Percent Total, Volatile, and Fixed Solids in Solid and Semisolid Samples*. A well-mixed representative sample is evaporated to dryness in a pre-weighed dish and dried to constant weight in an oven at 103° to 105° C. The weight of the residue remaining in the dish is the amount of total solids in the sample. Percentages of total solids are calculated according to the formula in SOP #1094. This result is used to determine metals, TOC, and organics results.

2.4.1.5 Grain Size

Grain size is performed by Azimuth Geo Services in Austin, Texas using the following procedures.

2.4.1.5.1 Marine sediment samples are analyzed for grain size by a sequence of wet sieving and dry sieving. Samples will be prepared by first splitting the individual sediment samples into the appropriate size for analysis. If sufficient sample material is available, optimal sample size will be 30 dry grams of mud and at least 70 dry grams of sand. The sample will be mixed by hand in 200 mL of a 5% solution of dispersant (sodium hexametaphosphate) to loosen clays. The mixture will be left for at least 12 hours and mixed by hand a second time. A 3% hydrogen peroxide solution will be mixed and left at least 12 hours. This procedure will be repeated if necessary. The sample will then be washed through a 0.063 millimeter sieve (US standard sieve mesh #230).

The wash load, which contains the silt and clay fractions, will be transferred to a 1000-mL cylinder, topped to 1000 mL with deionized water, and covered. The material retained on the sieve is the sand and gravel fractions. This coarse load will be transferred to a 200-mL beaker, decanted, and dried overnight at 95° C.

The dried sand and gravel fraction will be mixed by hand to disaggregate the material, and then dry-sieved using the following six sieve sizes:

Millimeters	2	1	0.5	0.25	0.125	0.0635
Phi Units	-1.0	0.0	1.0	2.0	3.0	4.0
U.S. Standard Sieve Mesh #	10	18	35	60	120	230

Stacked sieves will be placed on a Fritsch Analysette vibrating table for 10 minutes. Material retained on the -1.0 phi sieve will be considered the gravel fraction. Material retained on the 0.0, 1.0, 2.0, 3.0, and 4.0 phi sieves will be considered the sand fraction. Particles smaller than 4 phi will be analyzed using the pipette method described below. Each size class will be weighed to the nearest 0.1 mg on a top-loading balance.

The mud (silt + clay) fraction will be analyzed using the pipette method. The procedure is based on Stokes Law, which computes sediment settling velocity. The sample in the cylinder will be mixed to fully and uniformly suspend the sediment in the cylinder. When the mixing stops, settling of mud will begin and the time will be recorded. Within the first 20 seconds of settling, a 25-mL aliquot will be removed by pipette from a depth of 20 cm and emptied into a pre-weighed (based on an average of at least three weighings) 50-mL beaker. Twenty-five milliliters of deionized water then will be drawn into the pipette and emptied into the beaker to wash out any sediment inside the pipette. This sample will represent the total mud fraction of the sample. The beaker will be dried overnight at 95^{o} C and weighed to the nearest 0.1 mg. The total mud weight will be determined by subtracting the beaker weight and multiplying by 40 (25 mL × 40 = 1000 mL, total sample volume). A second withdrawal will be made at the time when all silt-sized (coarser than 8 phi) material has settled below the depth of the pipette. This withdrawal can be made at any depth, as long as the settling times are properly computed according to Stokes Law. According to calculations based on Stokes Law, at 10-cm depth this withdrawal time should occur at 2 hours, 3 minutes after mixing stops, and at 20-cm depth at about 4 hours, 5 minutes after mixing stops (Folk, 1974). Data will be presented in weight percent by size class. In addition, the gravel:sand:silt:clay ratio and a numerical approximation of mean size and sorting (standard deviation) will be calculated. A cumulative frequency curve of the data may be prepared using phi units.

Grain size is reported as dry weight percentages of sediments from minus 1 phi to 4 phi, as well as dry weight percentages of gravel, silts, and clays. This analysis is performed by a contract laboratory and the results are provided to the DLS for manual data entry into LabWare.

2.4.1.5.2 Quality control (QC) measures will include: method detection limit based on the analytical sensitivity of the balance used, and precision of laboratory triplicates analysis at a frequency of 5% of the samples analyzed.

2.4.1.6 *Clostridium Perfringens*

Clostridium perfringens is performed by BAL Laboratory in Cranston, Rhode Island using the following procedures.

2.4.1.6.1 Marine sediment samples are analyzed for *Clostridium perfringens* by determining the abundance on a dry weight basis of spores of the bacterium *Clostridium perfringens.* This is done by homogenizing samples and an aliquot of known weight transferred to a sterile 50-mL polypropylene centrifuge tube. .Sterile sodium hexametaphosphate solution will be added to the sample, and the tube will be capped and mixed thoroughly for 10-15 seconds. Sterile deionized water will be added, the sample remixed, and allowed to settle for 10 minutes. The supernatant will be removed from the tube with a sterile pipette and placed in a sterile test tube. The tubes will be stored on ice and analyzed within 30 minutes.

Analysis of the supernatant will be performed by membrane filtration. Enumeration of *C. perfringens* spores will follow the method of Bisson and Cabelli (1979). The extract will be filtered through a sterile, 0.45-*u*m pore size, gridded membrane filter that retains the bacteria. After filtration, the membrane containing the bacterial cells will be placed on a selective-differential medium and incubated.

The filters for enumeration of *C. perfringens* spores will be incubated anaerobically at 44.5^{o}C for 24 hours. Following incubation, the filter will be exposed to ammonium hydroxide for 15-30 seconds. Yellowish colonies that turn red to dark pink upon exposure will be counted as *C. perfringens*. Results are reported as # colonies/100 mL. This analysis is performed by a contract laboratory and the results are provided to the DLS for manual data entry into LabWare.

2.4.1.6.2 Quality control requirements include positive and negative controls, and laboratory duplicates at 5% of the samples analyzed.

2.4.2 Quality Control Requirements

Quality Control (QC) samples are run with every analytical batch of 20 samples or fewer. The suite of QC samples specified for a particular analytical batch depends on the parameters being analyzed. Table 9 lists the quality control samples and data quality acceptance limits for each measurement according to the particular parameter(s) being analyzed. Other QC samples (e.g., instrument QC) may be dictated by the analytical method and are described in Section 8.0 of DLS' QAMP (DCN #5000) and the specific SOP.

The definitions of the QC samples are as follows:

- **Laboratory Control Sample:** A sample of deionized water free from the analytes of interest and interferences, spiked with verified known amounts of analytes. It is processed simultaneously with and under the same conditions as samples through all steps of the preparatory and analytical procedures. The purpose of the LCS is to establish intra-laboratory or analyst specific recovery, precision, and bias and to assess the performance of the entire measurement process. These standards are purchased either from NIST (National Institute of Standards) or from a qualified commercial vendor.

- **Standard Reference Material:** A reference material, which is sufficiently well established for the calibration of procedures and development of methods. Certified values are generally based on the results of determinations by at least two independent methods of analysis. These standards are purchased either from NIST (National Institute of Standards) or NRC (National Research Council Canada).

- **Laboratory Duplicate (Processing):** A second aliquot of a sample taken from the same container as the first aliquot under laboratory conditions and processed and analyzed independently.

- **Method (Procedural) Blanks:** A sample of deionized water that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the preparatory and analytical procedures. The purpose of the Method Blank is to demonstrate that the analytical system is free of target analytes and interferences, or assess any possible contamination.

- **Field Duplicates/Triplicates:** Two/Three subsamples taken from one field sample (grab sample) and processed in the field as two/three separate samples, resulting in two/three sample containers.

- **Matrix Spike:** 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. The purpose of the matrix spike is to determine the effect of the matrix on a method's recovery efficiency.

- **Matrix Spike Duplicate:** A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

2.5 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

All equipment associated with sediment analyses (GC/MS, ICP, GFA, FAA, Hg analyzer, TOC analyzer, analytical balances, thermometers, and incubators) are calibrated and maintained according to manufacturer's specifications. This is done or checked on each day of use as described in Section 10.0 of DLS' QAMP (DCN #5000) or the pertinent SOP. An equipment logbook is maintained to document periodic maintenance and repairs of major equipment.

2.6 Instrumentation Calibration and Frequency

Calibration procedures for laboratory instruments are summarized in Table 10. All laboratory calibration records are reviewed by the Team Supervisor as part of the validation process and filed.

DLS policy on calibration standards is described in Section 6.0 of the QAMP (DCN #5000). Specific details are included in the pertinent analytical SOPs.

2.7 Tracking and Quality Verification of Supplies and Consumables

All supplies and consumables are ordered and when received, checked/verified by the analysts according to the requirements of the respective analysis SOP. All reagents and chemicals are Analytical Reagent Grade or higher. Standards are purchased according to the requirements of the respective analysis SOP and all information concerning the standards (purchased or prepared) is kept in the Standards Logbook. Certificates are kept in the team's Standards Certificate File. Expiration dates are assigned by the analyst either according to the manufacturer's specification or according to the requirements given in the respective analysis SOP. Additional information concerning standards and reagents can be found in Section 6.0 of DLS' QAMP (DCN #5000).

Table 9. Quality Control Samples and Data Quality Objectives for Sediment Chemistry

QC Type	Frequency	Acceptance Criteria	Corrective Action
Procedural Blanks			
Organics	1 per 20 samples	< RL[1]	Results examined by project manager, team supervisor, or lab manager. Corrective action (*e.g.*, re-extraction, reanalysis, data qualifier) is documented in LIMS flags and test comments and/or a Corrective Action Report. If appropriate, flag as 'B' (Not blank corrected, blank >5x MDL.)
Metals	1 per 20 samples	≤ 10% of the lowest sample concentration	
TOC	1 per day or 1 per 20 samples, whichever comes first	≤ 10% of the lowest sample concentration (total carbon)	
% dry weight	1 per 20 samples	≤ 0.0125%	
Grain Size	NA		
Clostridium perfringens	NA		
Accuracy			
Matrix Spike			
Organics	1 per 20 samples	50-150% recovery[2]	Document, justify deviations. Corrective action (*e.g.*, re-extraction, reanalysis, data qualifier) is documented in LIMS flags and comments and/or a Corrective Action Report. Flag as 'Q' (accuracy does not meet DQO.)
Metals	1 per 20 samples	70-130% recovery[2]	
TOC	NA	NA	
% dry weight	NA	NA	
Grain Size	NA	NA	
Clostridium perfringens	NA	NA	
Surrogate standards			
Organics only	Every sample	50-150% recovery[3] (40-150% for Naphthalene-d8)	Document, justify deviations. Corrective action (*e.g.*, re-extraction, reanalysis, data qualifier) is documented in LIMS flags and comments and/or a Corrective Action Report. Flag as 'Q' (accuracy does not meet DQO.)
SRMs			
Organics	1 per 20 samples	PD ≤ 35% vs. SRM range[4]	Results examined by project manager, team supervisor, or lab manager. Corrective action (*e.g.*, re-extraction, reanalysis, data qualifier) is documented in LIMS flags and comments and/or a Corrective Action
Metals	1 per 20 samples	PD ≤ 20% vs. SRM certified values[5]	
TOC	1 per 20 samples (digested); Run once a day with each batch	± 5% of certified value	

Table 9. Quality Control Samples and Data Quality Objectives for Sediment Chemistry			
QC Type	**Frequency**	**Acceptance Criteria**	**Corrective Action**
% dry weight	1 per 20 samples	89%-115% of certified value	Report. Flag as 'Q' (accuracy does not meet DQO.)
Grain Size	NA		
Clostridium perfringens	positive and negative control per 20 samples		
Precision			
Duplicates			
Organics (MS/MSD)	1 per 20 samples	≤ 30% RPD[6]	Document, justify deviations. Corrective action (*e.g.*, re-extraction, reanalysis, data qualifier) is documented in LIMS flags and comments and/or a Corrective Action Report. Flag with 'R' (precision does not meet DQO).
Metals	1 per 20 samples	≤ 25% RPD if value is >5 X MDL	
TOC	1 per 20 samples	≤ 25% RPD	
% dry weight	1 per 20 samples	≤ 10%	
Grain Size	2 per 20 samples (triplicates)	≤ 25% RPD	Reanalyze all samples in the batch.
Clostridium perfringens	1 per 10 samples	≤ 5% RPD	Rerun both samples.

[1] Reporting Limit (RL): The RL is the typical reporting limit, which is based on the low point of the calibration curve. (For PCBs and Pesticides this is 0.200 ng/g and for PAHs this is 0.500 ng/g.) Concentrations below the RL are reported only if all identification criteria are met.

[2] For matrix spike and matrix spike duplicates: Percent Recovery =([spiked sample result – unspiked sample result] ÷ spike amount) × 100.

[3] For surrogate standards: Percent Recovery = [(measured concentration)/(true or nominal concentration)] x 100%.

[4] For organics SRM: If the detected value falls within the SRM certified range, then percent difference (PD)=0. If the detected value falls outside the SRM certified range, then the PD is determined against either the upper or lower limit of the range.

[5] Percent Difference = [(SRM Certified value – Laboratory SRM result) ÷ SRM Certified value)]× 100

[6] Relative Percent Difference (RPD) = $\left|\text{(replicate 1 - replicate 2) x 2/(replicate 1 + replicate 2)}\right|$ x 100%.

Table 10. Calibration Procedures for Laboratory Instruments

Parameter	Instrument Type	Initial Calibration			Continuing Calibration		Corrective Action
		No. Stds.	Acceptance Criteria	Frequency	Acceptance Criteria	Frequency	
PCB	GC/MS (SIM)	5	RSD ≤ 20%	Prior to analytical run	PD from initial ≤ 25%	Every 24 hours	Document, justify deviations. Remedial maintenance, new initial calibration, or reanalyze samples as needed.
Pesticides	GC/MS (SIM)	5	RSD ≤ 20%	Prior to analytical run	PD from initial ≤ 25%	Every 24 hours	Document, justify deviations. Remedial maintenance, new initial calibration, or reanalyze samples as needed.
PAH	GC/MS (SIM)	5	RSD ≤ 25%	Prior to analytical run	PD from initial ≤ 25%	Every 24 hours	Document, justify deviations. Remedial maintenance, new initial calibration, or reanalyze samples as needed.
Metals	CVAA (Hg)	3	R ≥ 0.995 [1]	Prior to analytical run	± 15 % Rec.	Every 10 samples	Document, justify deviations. Remedial maintenance, new initial calibration, or reanalyze samples as needed.
	GFAA [2] (as required)	3	R ≥ 0.995 [1]	Prior to analytical run	± 10 % Rec.	Every 10 samples	
	FAA [2] (as required)	3	R ≥ 0.995 [1]	Prior to analytical run	± 10 % Rec.	Every 10 samples	
TOC	Combustion/ Infrared Carbon Analyzer	1 (Check std.)	See footnote 3	Weekly	5 % Rec.	Every 20 samples	Document, justify deviations. Remedial maintenance, new initial calibration, or reanalyze samples as needed.

[1] Instrument Performance Check standard (IPC =±5%), Independent Calibration Verification (ICV = ±10%), and Instrument Calibration Blank (ICB=<MDL) precede each run.
[2] Samples are screened by the ICP but may be analyzed by other methods as required.
[3] Three IPCs are checked after calibration. The mean of the standards must be between 95-105% recovery of the true value.

2.8 Data Management

2.8.1 Acquisition of Non-Direct Measurement Data

Field sample locations are pre-loaded in LIMS as Location IDs. A listing of Location IDs and corresponding sample numbers is sent to Normandeau in advance of the survey for loading into their navigation/sample tracking system. When samples are checked in, the Normandeau Bottle ID is scanned in to match the LIMS text ID. The Location ID for that container should match the Station ID printed on the sample label. Except for date and time, no field measurements are entered in LIMS. Station IDs are given in Table 11. The LIMS Location_ID, is equivalent to the Station_ID (EM&MS STAT_ID) or an abbreviation of the Station_ID in the case of station FF01A. Where there are multiple grabs per station, different Sample IDs will be assigned to each replicate, however the location codes will be the same and replicates can be matched using the collection time, which is recorded on the sample bottle at the time of collection.

Table 11. Station Identifiers

FACILITY_ID	LOCATION_ID	EM&MS STAT_ID	Location Description
HARBBNTH	C019	C019	42-21.56, 71-2.72, FORT POINT CHANNEL
HARBBNTH	T01	T01	42-20.95, 70-57.81, OFF DEER ISLAND WEST SIDE
HARBBNTH	T02	T02	42-20.57, 70-60.12, PRESIDENT ROADS
HARBBNTH	T03	T03	42-19.81, 70-57.72, OFF NORTH EAST TIP OF LONG ISLAND
HARBBNTH	T04	T04	42-18.6, 70-62.49, DORCHESTER BAY
HARBBNTH	T05A	T05A	42-20.38, 70-57.64, PRESIDENT ROADS
HARBBNTH	T06	T06	42-17.61, 70-56.66, NANTASKET ROADS
HARBBNTH	T07	T07	42-17.36, 70-58.71, QUINCY BAY
HARBBNTH	T08	T08	42-17.12, 70-54.75, HINGHAM BAY
MASSBNTH	FF1A	FF01A	42-33.84, 70-40.56, SOUTH OF CAPE ANN
MASSBNTH	FF04	FF04	42-17.28, 70-25.50, STELLWAGEN BASIN
MASSBNTH	FF09	FF09	42-18.78, 70-39.42, MASS BAY, WEST OF STELLWAGEN BASIN
MASSBNTH	FF12	FF12	42-23.40, 70-54.00, MASSACHUSETTS BAY NEAR NAHANT
MASSBNTH	NF04	NF04	42-24.96, 70-48.42, NORTH OF OUTFALL SITE
MASSBNTH	NF10	NF10	42-23.58, 70-50.28, WEST OF OUTFALL SITE
MASSBNTH	NF12	NF12	42-23.40, 70-49.86, WEST OF OUTFALL SITE
MASSBNTH	NF13	NF13	42-23.40, 70-49.38, WEST OF OUTFALL SITE
MASSBNTH	NF14	NF14	42-23.22, 70-49.38, WEST OF OUTFALL SITE
MASSBNTH	NF17	NF17	42-22.86, 70-48.90, WEST OF OUTFALL SITE
MASSBNTH	NF20	NF20	42-22.68, 70-50.70, WEST OF OUTFALL SITE
MASSBNTH	NF21	NF21	42-24.18, 70-50.22, NORTHWEST OF OUTFALL SITE
MASSBNTH	NF22	NF22	42-20.88, 70-48.90, SOUTH OF OUTFALL SITE
MASSBNTH	NF24	NF24	42-22.86, 70-48.12, SOUTH OF OUTFALL SITE

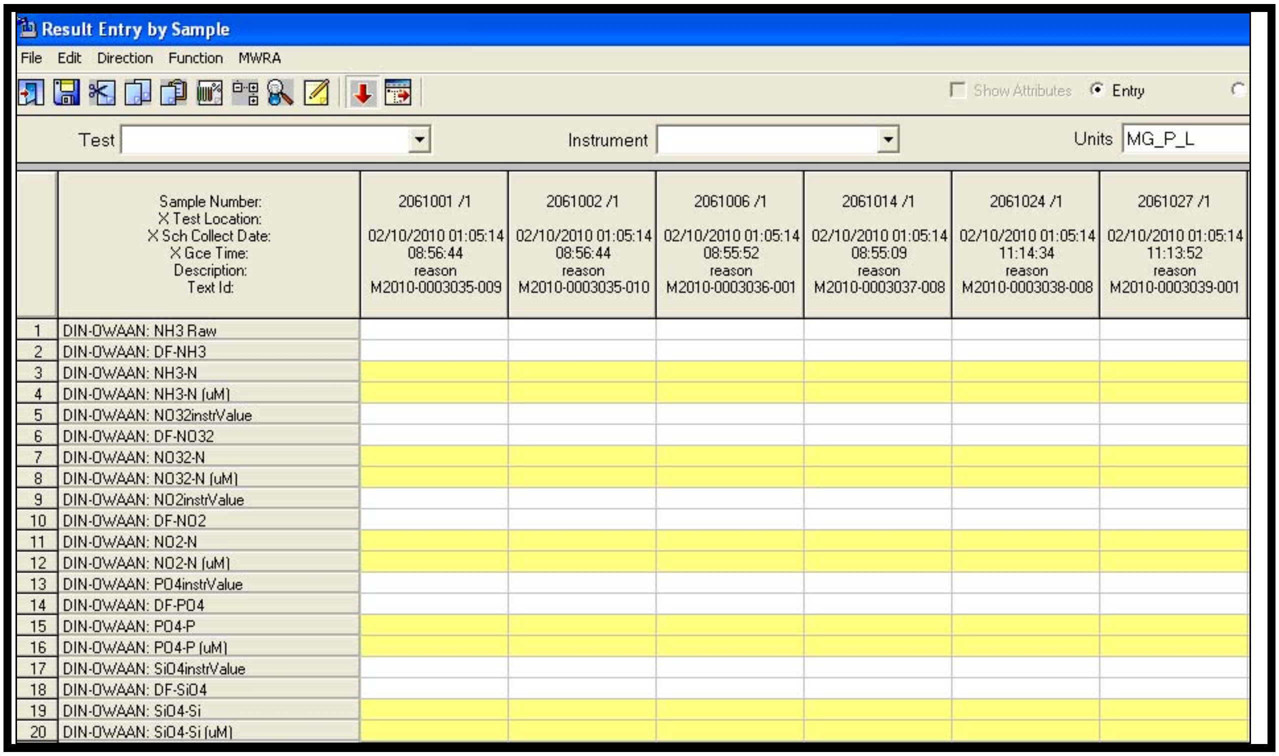
2.8.2 Data Recording

All documentation conforms to the DLS QAMP (DCN #5000), including:

- All original data are recorded in permanent ink in a bound notebook, on standardized forms, or, where applicable, in electronic files.
- Corrections are made by placing a single line through the incorrect entry.
- Corrections are initialed and dated at the time the correction is made.
- All QC data (precision, accuracy) are recorded in laboratory notebooks and in LIMS.

For this project, all test results are either entered manually into LIMS from laboratory logbooks, spreadsheets, or instrument data system printouts, or are electronically transferred from instrument data systems. In the LIMS system, the LIMS batch module (Batch Manager) is used to create sample/test fields for routine internal laboratory QC parameters (method blanks, laboratory control samples, and laboratory duplicates). These QC tests are programmed in LIMS with test-specific warning and control limits. As results are entered, the field and QC tests are checked against limits, and the analyst is informed of any parameter that exceeds a warning or control limit. This allows gross typographical errors to be detected and serves as an early notification of any limit exceedance.

Completed data forms or other types of hand-entered data are signed and dated by the individual entering the data. Direct-entry and electronic data entries identify the person collecting or entering the data. An example LIMS data entry screen for this project is shown in Figure 4. It is the responsibility of the validator to ensure that all data entries and hand calculations are verified in accordance with procedures described in Section 2.8.5.



Test
Instrument
Units MG_P_L

	Sample Number: X Test Location: X Sch Collect Date: X Gce Time: Description: Text Id:	2061001 /1 02/10/2010 01:05:14 08:56:44 reason M2010-0003035-009	2061002 /1 02/10/2010 01:05:14 08:56:44 reason M2010-0003035-010	2061006 /1 02/10/2010 01:05:14 08:55:52 reason M2010-0003036-001	2061014 /1 02/10/2010 01:05:14 08:55:09 reason M2010-0003037-008	2061024 /1 02/10/2010 01:05:14 11:14:34 reason M2010-0003038-008	2061027 /1 02/10/2010 01:05:14 11:13:52 reason M2010-0003039-001
1	DIN-OWAAN: NH3 Raw						
2	DIN-OWAAN: DF-NH3						
3	DIN-OWAAN: NH3-N						
4	DIN-OWAAN: NH3-N (uM)						
5	DIN-OWAAN: NO32instrValue						
6	DIN-OWAAN: DF-NO32						
7	DIN-OWAAN: NO32-N						
8	DIN-OWAAN: NO32-N (uM)						
9	DIN-OWAAN: NO2instrValue						
10	DIN-OWAAN: DF-NO2						
11	DIN-OWAAN: NO2-N						
12	DIN-OWAAN: NO2-N (uM)						
13	DIN-OWAAN: PO4instrValue						
14	DIN-OWAAN: DF-PO4						
15	DIN-OWAAN: PO4-P						
16	DIN-OWAAN: PO4-P (uM)						
17	DIN-OWAAN: SiO4instrValue						
18	DIN-OWAAN: DF-SiO4						
19	DIN-OWAAN: SiO4-Si						
20	DIN-OWAAN: SiO4-Si (uM)						

Figure 4: LIMS Data Entry Screen

2.8.3 Analysis Comments

Flags and comments, where necessary and appropriate, are made in LIMS for sample measured/non-measured information to provide the data validator/reviewer with an explanation or description of the test results or sample characteristics. All LIMS entered comments associated with a sample/test/result are part of the LIMS database record for the analysis of the respective sample.

2.8.3.1 Flag Types

Flags are the preferred type of annotation. Flags can be applied at the sample, test, and result levels using a pre-defined list of flags, including those in Table 12.

2.8.3.2 Comment Types

Comments are entered as free-flowing text. Comments can be applied at the sample or test level. When pre-defined text is used, it should not be altered. Comments should be used to augment the flags or as a substitute to pre-defined text when there is no appropriate existing flag. Further, test comments for HOM analyses are only used to augment flags and are entered either by the analyst or validator, or to document a DAIR.

2.8.3.3 Sample Notepad Comments

If there is a situation for which flags or comments are inappropriate, the Sample Notepad is available for entry of free-flowing text. The Sample Notepad should not be needed routinely and should be regarded as a last resort. Non-routine sample receipt information can be recorded in the Receiving Notes field.

2.8.3.4 Test and Result Flags

From time to time, a test result is reported as invalid or is qualified by the DLS. When such a situation occurs, the analyst/validator/approver annotates the reason for the invalidation or qualification by entering an appropriate sample, test, or result flag, and explanatory text into the appropriate test comment field. The pre-defined flags are listed in Table 12, below.

To alert the data user to results that may be affected by low-level laboratory bias, the following flagging procedure is used with regard to method procedural blanks. If the method procedural blank is >5 times the MDL, all tests and QC in the batch are flagged with “B”. Note that tests are also flagged with “J” (“estimated value”) when the result is below the lowest calibration standard. However, when a J flag is used, no other flags are needed on that test because the J flag already indicates that the result is an “estimated value”.

Also note the following:
- “Q”, accuracy does not meet data quality objectives, is used for all tests in a batch when the LCS recovery is outside limits.
- “R”, precision does not meet data quality objectives, is used only on a test used for duplicate analysis when the duplicate RPD is outside limits.
- “W”, use with caution, is only used for exceptional situations. It will no longer be routinely used when a blank is >MDL and the sample is <5x the blank.
- “L”, analytical concentration reported from dilution, will be applied to the results for those components analyzed on dilution, rather than on the whole test.

LabWare LIMS allows multiple result flags (or test flags) to be used; these will be concatenated in the data warehouse and parsed into multiple value qualifiers by ENQUAD automated routines.

Note: The EM&MS qualifiers, which are used for reporting data to Normandeau, are not the same as the pre-defined LIMS flags used to qualify analytical results.

Table 12. Test Comments and Qualifiers for Qualifying/Annotating Sample Test Results

LIMS Flag	Description
A	Not detected - value reported as negative or missing
B	Not blank corrected, blank ≥5x MDL
E1	Calibration level exceeded
E2	Results not reported, value given is NULL, see comments field
F	Value reported <MDL, See Sample Notepad
G1	Recovery outside data quality objectives
G2	Co-eluting compound interferes with peak of interest
J	Estimated value [1]
K	Matrix interference
L	Analytical concentration reported from dilution
P	Lab sample bottles mislabeled - caution data use
Q	Accuracy does not meet data quality objectives
R	Precision does not meet data quality objectives
S	Suspect/Invalid. Not fit for use
T	Holding time exceeded
W	This datum should be used with caution, see comment field
X	See Sample Notepad for multiple qualifiers

[1] A value reported between the MDL and the lowest calibration standard is considered to be an estimate.

In order to ensure that all samples are accounted for when transferring the results from LIMS to EM&MS, if a rejected (invalid) result is not superseded by a retest, it must include a flag or comment indicating why the result was rejected and could not be retested. A rejected sample will appear to the LIMS user on the screen in italics, not bold and not red.

2.8.4 Data Reduction

Data reduction procedures and formulae are defined in laboratory SOPs and in Section 7.0 of the QAMP (DCN #5000). This is performed electronically either by the instrument software or in a spreadsheet and is validated according to procedures described in Section 2.8.5.

2.8.5 Data Validation

Data validation, a two-step process, is a standardized process for judging the quality and usefulness of a discrete set of chemical data. The first data validation step for HOM data produced by the DLS involves the review of analytical results of both HOM samples and QC samples against the Data Quality Objectives (Table 9) and the quality standards in Section 7.0 of DLS' QAMP (DCN #5000). The completion of the validation process and the approval process is documented in LIMS. Until a sample is approved, the results are regarded as preliminary. Subsequent to the approval of a sample test result, data can only be changed through the DAIR process described in Section 2.8.7, below.

The second step in the process is the review of the results by the ENQUAD HOM Project Manager and is detailed in Section 4.0 below**.**

The veracity and validity of analytical results are assessed throughout the analytical data result Analyst Review, Validation and Approval process, which includes, but is not limited to:

- **Analyst Review** (result review)**:** An assessment of the components of the analytical method (reagents, glassware cleanliness, standard expiration dates, instrument operation, etc.), QC, calculations, and data entry by the analyst;
- **Validation** (test review)**:** Performance of QC sample results against established limits, holding times calculation cross-checking, etc. by the Team Supervisor or his/her delegated validator; and;
- **Approval** (sample review)**:** Comparability and test consistency of the sample, etc. by a Lab Manager or his/her delegated approver.

Data specified in the QAMP or specified in this plan are not to be marked as rejected (invalid) in LIMS unless the data validator has provided an explanation with a flag and comment. Data that do not meet the Data Quality Objectives of this plan are annotated (See Section 2.8.3 above). When all samples from a survey are approved in LIMS, the DLS HOM Project Manager notifies the ENQUAD Benthic Project Manager and EM&MS Manager.

2.8.6 Reporting of Results

All data are reported electronically to the ENQUAD Benthic Project Manager as approved results in LIMS. Also, a QA Package (see 2.8.6.4, below) is to be forwarded to the ENQUAD Benthic Project Manager immediately subsequent to the completion of the analyses of all survey samples.

2.8.6.1 Turnaround Times

In order to meet the reporting deadlines to Normandeau, the sample turnaround time for benthic parameters is 42 calendar days from receipt of the last sample. This is the deadline for samples to be approved in LIMS.

2.8.6.2 Results Data Entry

Organics: For organics, "non-detects" are reported as <RL, where the RL is based on the concentration of the low standard in the ICAL (see Table 6). However, all "detects" are reported, regardless of the RL or MDL, as long as they meet the following identification criteria:

- The peak must be at the correct retention time.
- The signal-to-noise ratio of the quantitation ion must be ≥ 3.
- The secondary ion ratio criteria must be met.

If the ion ratio criteria are not met but it is the analyst's professional judgment that the compound is present, the compound can be reported with an "S" flag. The reasons for including a compound that fails the ion ratio criteria include: suspected interferences, if its presence is consistent with other compounds (such as Fluoranthene/Pyrene, DDE/DDT, etc.), or based on historical data.

Whenever a compound is reported at a concentration below either the MDL or RL, the data must be flagged using the Result Flag in LIMS and the Test Comments and/or Sample Notepad (where necessary) to provide information regarding component-specific qualifiers. All sample data must be clearly marked on the data summary sheet, so that the appropriate comments can be added by the data validator.

Metals, TOC, and Solids: Results for metals and TOC are reported down to the Instrument Blank. In most cases, the Instrument Blank is equal to the MDL. In instances when the Instrument Blank exceeds the MDL, blank and sample results are reported down to the RL. For solids, results are reported down to 0.0125%. Results are expressed in the units listed in Table 8.

2.8.6.3 Traceability

Reported results must be traceable. Traceability is the characteristic of data that allows a final result to be verified by review of its associated documentation. All laboratory results for a given sample must be traceable throughout the entire analytical process applied to the sample. Traceability is maintained through LIMS (which stores all of the pertinent data associated with the sample and keeps an audit trail of all record transactions) and by the utilization of various logbooks (preparation, analytical, and instrumental), instrument raw data printouts, electronic files, and spreadsheets. Traceability in EM&MS is documented through the use of Standard Query Language (SQL) scripts to make any corrections to the data; electronic records of scripts and their output files are maintained by ENQUAD.

2.8.6.4 QA Package

Upon completing the chemical analyses, DLS forwards to the ENQUAD Benthic Project Manager a QA Package consisting of:

- QA results vs. acceptance ranges.
- Any descriptive QA information relevant to the delivered data (i.e. sample notepad comments).
- **Audit Reports:** Copies of the quarterly rolling compliance audit and any audits that may have been specifically performed on HOM items.
- **Missing Samples Report:** A Missing Samples Report is generated by DLS and forwarded as part of the QA Package along with an explanation of why the samples are missing.

- **Corrective Action Report:** Report of corrective actions associated with HOM benthic survey sample analyses.

- **DAIR (Data Anomaly Investigation Report) Report:** Report of DAIRs associated with HOM benthic survey sample analyses.

- **Normandeau Chain-of-Custody forms:** All signed originals.

- **QA Statement:** A QA Statement (see Figure 5) based on the Precision, Accuracy, and Representativeness (where applicable), Custody, and Comparability is compiled and forwarded to the ENQUAD Benthic Project Manager. Deviations and unusual circumstances will be noted in the comments. The QA Statement is signed by the DLS HOM Project Manager and Lab Manager.

All information, including the signed QA Statement, is forwarded by inter-office mail to the ENQUAD Benthic Project Manager.

2.8.7 Changes to Approved Data

Once a LIMS result has been approved and released to the client, it can only be modified through the DAIR (Data Anomaly Investigation Report) process. The DAIR process is detailed in the DLS SOP DCN: 5004, "Procedures for the Response to Discoveries of Anomalies in the Department of Laboratory Services' Data Records". A DAIR is initiated by anyone who wants a data anomaly to be researched and, if possible, rectified. For example, this may result from a discovery that wasn't known when the samples were being processed (e.g. a sample was collected at the wrong location) or when results appear suspect (e.g. significantly higher or lower than previous results). The DAIR process documents the review of the suspect results, the decisions that were reached, and any changes that were made to the LIMS results. Ultimately, the client's approval (ENQUAD) is obtained before results are changed in LIMS. The client is notified when the corrections are complete.

In the event that apparently anomalous data needs to reviewed and, if necessary, changed after approval but before it is released by ENQUAD, the "Fast Track" DAIR process should be used.

In LabWare LIMS, all DAIRs are processed electronically. Client-initiated DAIRs should be communicated via email to the QA Coordinator. She will initiate the electronic DAIR or designate to the appropriate personnel. The initiator is to include any comments or information received from the client. The results of a completed DAIR will be communicated back to the client.

Figure 5: Quality Assurance Statement

MWRA DEPARTMENT OF LABORATORY SERVICES

MWRA Harbor and Outfall Monitoring Project

Quality Assurance Statement

Description of Data Set or Deliverable:____________________________________

1.0 Sample Analyses

All samples were handled, analyzed and reported according to the procedures and requirements specified in the QAPP **(**Constantino *et al.*, 2012), except as noted in the comments. Specifically:

- The custody of all samples were transferred properly and maintained. **Yes** **No**
- All of the samples on the COC were received and all required tests performed. **Yes** **No**
- QC samples were analyzed and all acceptance criteria in accordance with the DLS QAMP (DCN #5000) and the QAPP (Constantino, *et al.,* 2012) were met. **Yes** **No**
- 100% of the data entry and 20% of manually-calculated data were checked for accuracy. **Yes** **No**
- Test/Sample Comments were assigned properly. **Yes** **No**
- All tests were validated and approved. **Yes** **No**

2.0 Attached Documentation

The following documentation, when applicable, is included in the QA Package:

☐ Audit Reports
☐ Corrective Actions
☐ DAIRs
☐ Normandeau COC Forms (Originals)

Comments:

3.0 CERTIFICATION

We, the undersigned, attest that the material contained in this analytical report is, to the best of our knowledge and belief, accurate and complete.

DLS Project Manager *(date)*

DLS Section Manager *(date)*

3.0 ASSESSMENT/OVERSIGHT

3.1 Department of Laboratory Services

3.1.1 Performance and system audits

The DLS’ audit procedures are documented in Section 9.0 of the QAMP (DCN #5000). A performance audit provides a quantitative assessment of the analytical measurement process. It provides a direct and independent, point-in-time evaluation of the accuracy of the various measurements systems and methods. This is accomplished by challenging each analytical system (method/procedure) with an accepted reference standard for the analyte(s) of interest. The DLS annually participates in Discharge Monitoring Report (DMR) Performance Testing (PT) studies and in the Water Pollution (WP) and Water Supply (WS) Performance Testing studies. The applicable parameters found in the PT samples are: TOC, Pesticides, PCBs, and metals. Acceptable performance on these PT samples is required for NPDES self-monitoring analyses and Massachusetts DEP Certification, respectively.

In addition, internally administered performance evaluation samples may be submitted to the laboratory sections on a random, as required, basis and for those analytes not present in the PT samples.

Quarterly rolling compliance audits are performed to review laboratory operations to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data. Each quarter a different aspect of the laboratory operation is audited. This process identifies the strengths and weaknesses of the DLS Laboratory and indicates areas that need improvement. Rolling audits are performed by the QA Coordinator. Any significant deviations from accepted practices result in Corrective Actions.

All data must be reviewed by the ENQUAD Benthic Project Manager prior to incorporation in the ENQUAD environmental monitoring database and must be accompanied by a signed QA statement that describes the types of audits and reviews conducted and any outstanding issues that could affect data quality and a QC narrative of activities, as described in Section 2.8.6.4, above.

Performance audits, procedures used to determine quantitatively the accuracy of the total measurement system, or its components, are the responsibility of DLS as described above.

3.1.2 Corrective Action

Section 11.0 of DLS’ QAMP (DCN #5000) details the situations that require corrective action, how corrective actions are initiated, investigated, resolved, and documented to ensure a complete and systematic response to each corrective action request. Examples of situations requiring initiation of the corrective action process include mishandling of a sample or its documentation, deficiencies discovered during an internal audit, or use of unapproved modifications to an analytical method. The occurrence of a practice or incident that is inconsistent with the established quality assurance and quality control procedures of the laboratory must be formally addressed with a corrective action response. Any laboratory employee may request corrective actions when necessary.

Upon the initiation of a corrective action, the problem is documented, and a corrective action plan is developed. After required corrective action has been taken, the information is documented by the team and approved by the Laboratory Director.

In LabWare LIMS all Corrective Actions are processed electronically. Client-initiated Corrective Actions should be communicated via email to the QA Coordinator. She will initiate the electronic Corrective Action or designate to the appropriate personnel. The initiator is to include any comments or information received from the client. The results of a completed Corrective Action will be communicated back to the client.

3.1.3 Reports to Management

Information concerning any activity or situation relating to the QA of this project is reported quarterly to DLS managers and supervisors as part of DLS' quarterly QA (Quality Assurance) Report and Rolling Audit Report. The QA Coordinator prepares these reports. Specific information resulting from any oversight activities is included in the QA Package (2.8.6.4) accompanying the survey results. Guidance for QA reporting can be found in Section 13.0 of DLS' QAMP (DCN #5000).

3.2 Normandeau

3.2.1 Performance and System Audits

The Normandeau QA Officer for the Harbor and Outfall Monitoring Project conducts Field Sampling Technical System Audits of the field program, and Data Technical System Audits of the sample collection data, as described in the Benthic Monitoring QAPP (Nestler *et al.* 2011). Like other "subcontractor" laboratories on the HOM project, DLS is fully responsible for the QA of the data it submits. Data must be submitted in QAPP-prescribed formats; no other is acceptable.

3.2.2 Corrective Action

As defined in Normandeau's QAPP (Nestler *et al.*, 2011),

> All technical personnel share responsibility for identifying and resolving problems encountered in the routine performance of their duties. Issues that affect the schedule, cost, or performance of project tasks will be reported to Ms. Ann Pembroke, Normandeau's Project Manager. She will be accountable to MWRA and to Normandeau management for overall conduct of the Harbor and Outfall Benthic Monitoring Project, including the schedule, costs, and technical performance. Ms. Pembroke will be responsible for identifying and resolving problems that (1) have not been addressed in a

timely manner or successfully at a lower level, (2) influence multiple components of the project, or (3) require consultation with Normandeau management or with MWRA. She will be responsible for evaluating the overall impact of the problem on the project and for discussing corrective actions with the MWRA Benthic Monitoring Project Area Manager. She will also identify and resolve problems that necessitate changes to this QAPP. Problems identified by the Normandeau QA Officer, Mr. Robert Hasevlat, will be reported to Ms. Pembroke and corrected as described in Section C2 [of Nestler *et al.* 2011].

Identification of problems and corrective action at the laboratory level (such as meeting data quality requirements) is resolved by DLS staff and/or by ENQUAD staff. Issues that affect schedule, cost, or performance of the sediment monitoring tasks, and any issues affecting data quality, are reported to the ENQUAD Benthic Project Manager and to the Normandeau Project Manager. The DLS HOM Project Manager and the ENQUAD Benthic Project Manager are responsible for addressing these issues and for evaluating the overall impact of the problem on the project and for discussing corrective actions with Normandeau Project Management. Problems identified by the Normandeau QA Officer are reported and corrected as described in Section C.1.2 of the Benthic QAPP (Nestler *et al.* 2011).

3.3 Work Stoppage for Cause

The ENQUAD Program Manager, Water Quality, in consultation and conjunction with the Director of DLS, has the authority to stop any and all work for cause.

4.0 DATA VALIDATION AND USABILITY BY ENQUAD

This section addresses the review of data for fitness-for-use subsequent to their being approved and validated by DLS, and prior to their loading into the MWRA EM&MS database, inclusion in a data report, and use by Normandeau in synthesis reports.

4.1 Data Reduction and Transfer

4.1.1 Data Reduction and Processing

The requirements for data reduction and processing are described in the DLS QAMP (DCN # 5000), applicable laboratory SOPs, and Section 2.8 above.

4.1.2 Data Transfer

- Only approved data are transferred to EM&MS, including those marked as invalid by DLS. The data is transferred after the QA Package is received. Data is transferred every 15 minutes from LIMS automatically to the WWQ data warehouse by tested automated routines. Transfer of data from WWQ to EM&MS work tables is done by tested automated routines.

- Application of qualifiers in EM&MS is done by automated routines that parse flags applied by the laboratory, or by the ENQUAD Benthic Project Manager based on review of the data and associated comments.

- Generally, invalid data are given an EM&MS qualifier of ‘s’. Invalid data may be accepted into EM&MS with a qualifier other than ‘s’ at the discretion of the ENQUAD Benthic Project Manager, provided another appropriate qualifier is used and an explanatory comment is included in the database record.

- Any manual additions or changes to qualifiers and comments directed by the ENQUAD Benthic Project Manager are documented in an Oracle table in the HOM Review application.

4.1.3 Change and Corrections in the EM&MS Database

The guidance for changing and correcting data in the EM&MS database is as follows:

- Corrections to data in EM&MS work or production tables are done only through the use of SQL scripts, which must include the following:

  - Indication of whether the script is to be run on work or production tables
  - Comments including the name of script, author, date, and purpose of script
  - Record of date run in spool file
  - List out records to be changed

  - Demonstrate that problem has been fixed (*e.g.* by listing changed records.)

- Changes may be made only by the EM&MS Database Administrator (Dr. Douglas Hersh) or his designee. These changes are also documented in the DB_TASKS table within the EM&MS database.

4.1.4 Data Review, Validation, and Fitness-for-Use

4.1.4.1 Data Review

The ENQUAD Benthic Project Manager uses a spreadsheet prepared from an Oracle Discoverer workbook to review the analytical results, flags, and comments. Standard LIMS flags are parsed into EM&MS qualifiers. In order to review and assess the HOM results, the ENQUAD Benthic Project Manager:

- Reviews all data for technical reasonableness and completeness. Reviews include all rejected samples, deleted and invalid tests, and out of range results. The ENQUAD Benthic Project Manager reviews documentation in LIMS and the QA Package, and compares results to historical data distributions to check for reasonableness.

- Corrects or adds to qualifiers and comments as appropriate based on review of the data. If there are questions that cannot be resolved by examining the comments, he initiates a DAIR (see 2.8.7).

The ENQUAD Database Manager:

- Makes available for the ENQUAD Benthic Project Manager's review: the QA statement from DLS,a Notepad comments Report if provided by DLS, QC results, a comparison of results to the range of historical data, and a spreadsheet of the results and qualifiers.

- Adds qualifiers and comments as directed by the ENQUAD Benthic Project Manager.

- Calculates descriptive statistics such as sample size, mean, standard deviation, minimum, and maximum after the survey results are transferred from LIMS to EM&MS via WWQ.

- Ensures that the data loaded into the EM&MS database meet all applicable constraints (*i.e.* on the BOTTLE and ANALYTICAL_RESULTS tables.)

- Produces a data report for DLS review, containing the statistics, a list of non-detects, and pertinent information from the flags, test comments, sample notepad comments and ENQUAD Benthic Project Manager along with the data.

4.1.4.2 Data Validation/Fitness-for-Use

The ENQUAD Benthic Project Manager determines whether the results are Fit-for-Use and can be incorporated into the synthesis reports.

In accordance with the DLS' QAMP (DCN #5000) 20% of manual calculations are performed by a second staff member to verify that calculations are accurate and appropriate.

Data from the laboratories receive an additional review by ENQUAD staff after the data has been synthesized into a data report. Any issues are corrected in the database and documented in scripts and list files maintained by MWRA data management.

4.1.4.3 Sampling Design

All sampling is performed by Normandeau. This QAPP does not address sampling design, which is described in the Benthic Monitoring QAPP (Nestler *et al.* 2011.)

4.1.4.4 Data Transmittal to Normandeau

After review of the data report by DLS and incorporation of any corrections, the ENQUAD Database Manager can export the data from the EM&MS database as needed for synthesis, in a format agreed upon between ENQUAD and Normandeau.

4.1.4.5 Data Analysis

Data is analyzed and reported by Normandeau as part of the synthesis reporting under the HOM contract (see Nestler *et al.* 2011).

5.0 REFERENCES

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