Quality Assurance Project Plan (QAPP)

for

Fish and Shellfish Monitoring

Massachusetts Water Resources Authority
Environmental Quality Department
Report 2009-07

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Fish and Shellfish Monitoring

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1.0 PROJECT MANAGEMENT

1.1 Project Organization

Figure 1 presents the project management structure for tissue chemical 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 sample handling, sample analysis, and data loading for the tissue chemical analyses that are part of the MWRA's harbor and outfall monitoring program.

ENQUAD Dr. Andrea Rex is the Director of the Environmental Quality Department. Mr. Maurice Hall is the Project Manager for ENQUAD and is primarily responsible for the fish and shellfish monitoring. 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 management database (EM&MS) and transmitting them to AECOM. Dr. Douglas Hersh is ENQUAD's Database Administrator for the EM&MS database.

DLS Dr. Michael 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. Mr. Jim Fitzgerald is the Supervisor of the Violet team, responsible for sample management. Ms. Polina Epelman is the Section Manager responsible for the Orange and Green Teams. Ms. Patricia Sullivan is the Supervisor of the Orange Team, responsible for metals analyses. Mr. Mark Lambert is the Supervisor of the Green Team, responsible for organics analyses. Ms. Jennifer Prasse 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>
<tr><th colspan="4">Table 1. DLS Reporting Relationships</th></tr>
<tr><td colspan="4">Michael Delaney, Director of Laboratory Services</td></tr>
<tr><td colspan="2">Polina Epelman, Lab Manager
(Operations)</td><td>Steve Rhode, Lab Manager
(Client Services)</td><td rowspan="3">Jennifer Prasse
QA Coordinator</td></tr>
<tr><td rowspan="2">Patricia Sullivan,
Supervisor, Orange
Team</td><td rowspan="2">Mark Lambert,
Supervisor, Green
Team</td><td>Yong Lao,
Project Manager
(Client Services)</td></tr>
<tr><td>Edward Caruso
Client Services Coordinator</td></tr>
<tr><td>Metals</td><td>Organic Contaminants</td><td>Jim Fitzgerald
Supervisor,
Violet Team
Sample Management</td><td>Performance Testing,
Oversight and Document
Control</td></tr>
</table>

AECOM Environment (AECOM) Dr. James Blake is the HOM program manager for AECOM. He is responsible for the overall performance of the HOM project.

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

Figure 1 Organizational Chart for Metals and Organics for the Fish and Shellfish Monitoring Program

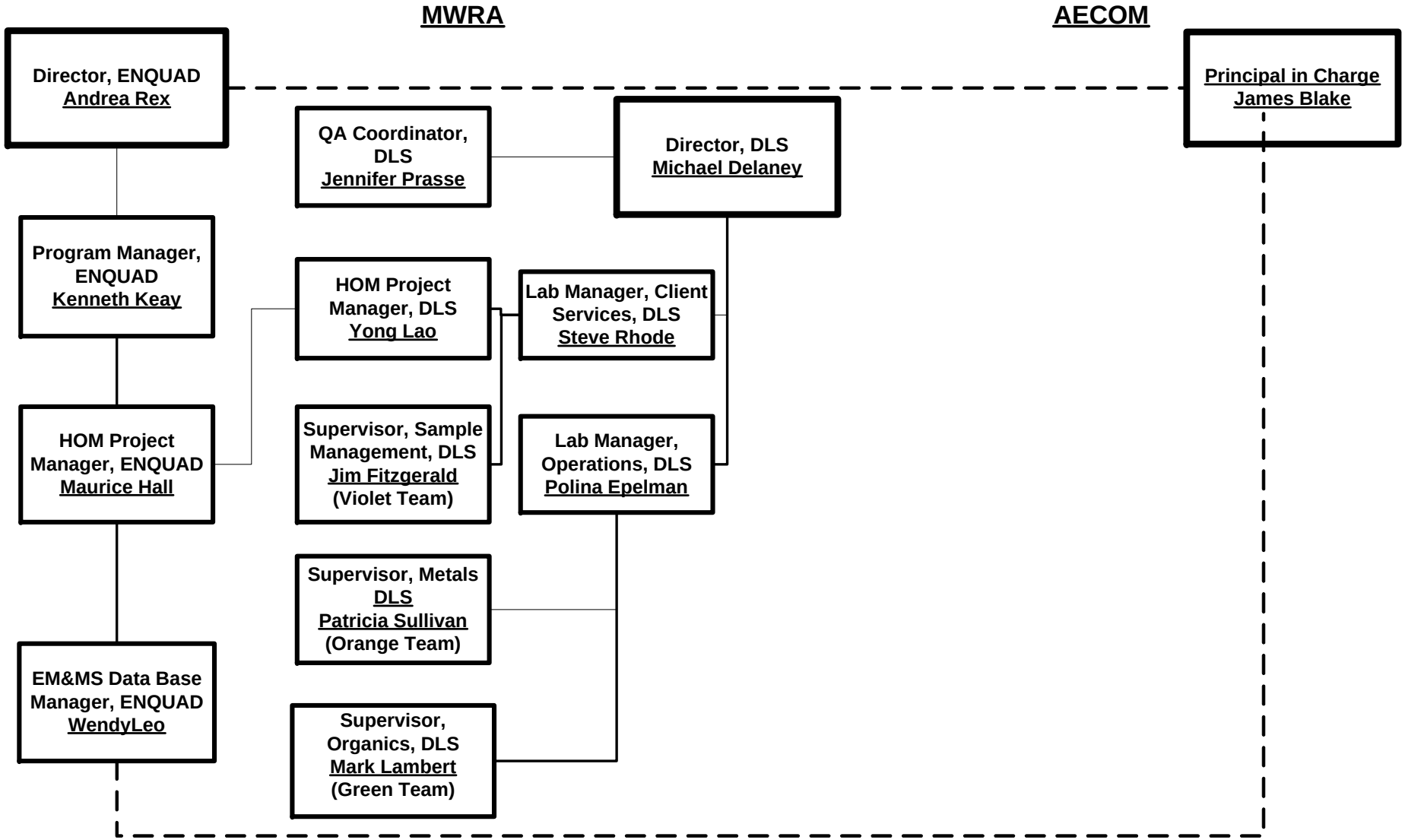

Table 2.		**Contact Information**		
Name	Title/Role	Location	email	Phone
James Blake	HOM6-II Program Manager	AECOM[1]	jblake[at]aecom.com	508-457-7900
Edward Caruso	Client Services Coordinator	DLS[2]	edward.carusojr[at]mwra.state.ma.us	617-660-7807
Mike Delaney	Laboratory Director	DLS	mike.delaney[at]mwra.state.ma.us	617-660-7801
Polina Epelman	Laboratory Manager (Red, Orange, Green)	DLS	polina.epelman[at]mwra.state.ma.us	617-660-7802
Jim Fitzgerald	Team Supervisor (Violet)	DLS	james.fitzgerald[at]mwra.state.ma.us	617-660-7851
Doug Hersh	EM&MS Database Administrator	ENQUAD[3]	douglas.hersh[at]mwra.state.ma.us	617-788-4738
Maurice Hall	Project Manager	ENQUAD	maury.hall[at]mwra.state.ma.us	617-788-4944
Kenneth Keay	Program Manager	ENQUAD	kenneth.keay[at]mwra.state.ma.us	617-488-4947
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
Wendy Leo	EM&MS Manager	ENQUAD	wendy.leo[at]mwra.state.ma.us	617-788-4743
Jennifer Prasse	QA Coordinator (Yellow)	DLS	jprasse[at]mwra.state.ma.us	617-660-7808
Steve Rhode	Laboratory Manager (Violet)	DLS	steve.rhode[at]mwra.state.ma.us	617-660-7803
Debra McGrath Simmons	QA Officer	AECOM	dlsimmons[at]ensr.aecom.com	508-457-7900
Pat Sullivan	Team Supervisor (Orange)	DLS	patricia.sullivan[at]mwra.state.ma.us	617-660-7838

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[3] Environmental Quality Department, MWRA, 100 First Avenue, Boston, MA 02129, 617-788-4601

1.2 Communication Plan

Mr. Maurice Hall is the primary contact with the monitoring prime consultant AECOM on technical issues. Dr. Yong Lao is DLS' primary contact with ENQUAD, and attends selected HOM project meetings. 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 AECOM and Battelle 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	**Maurice Hall, Yong Lao**
Transfer of samples	James Blake, Jim Fitzgerald (Violet)
Data interpretation	Maurice Hall
Laboratory technical issues	Relevant DLS Team Supervisor(s): ▪ M. Lambert (Green-organics) ▪ P. Sullivan (Orange-metals) Polina Epelman, Steve Rhode
Data management/database	Wendy Leo
Cost/schedule	Kenneth Keay, Mike Delaney James Blake (issues affecting cost/schedule of AECOM contract)
Quality assurance	Mike Delaney, Jennifer Prasse, Wendy Leo, James Blake (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, Mr. Jim Fitzgerald immediately notifies the AECOM Field Sample Custodian, Mr. James Blake as well as Dr. Yong Lao, and Mr. Maurice Hall.

Changes to the number of planned samples should be communicated to the Violet Team, Dr. Yong Lao and Mr. Maurice Hall 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 2.2.

1.3 Project Background

The background of the fish and shellfish project can be found in the CWQAPP for Fish and Shellfish Monitoring (Pembroke et al., 2006, Maciolek et al., 2008).

Previously, the tissue chemical analyses were conducted by subcontractor laboratories to the HOM consultant. This QAPP reflects a change in analytical laboratories and describes the quality system implemented for analytical procedures that are performed for the HOM project by the MWRA DLS.

1.4 Project Description and Schedule

1.4.1 Objectives and Scope

The Massachusetts Water Resources Authority (MWRA) is continuing a long-term biomonitoring program for fish and shellfish for the MWRA effluent outfall that is located in Massachusetts Bay. The goal of the biomonitoring is to provide data that may be used to assess potential environmental impact of effluent discharge into Massachusetts Bay. These data will be used to ensure that discharge from the new outfall does not result in adverse impacts by comparing values with established thresholds (MWRA, 2001a) and between potentially-impacted and reference stations (MWRA, 2004a).

The overall objective of the fish and shellfish monitoring is to define the condition of fish and shellfish health in terms of the presence of disease (external and internal), and organic and inorganic (metal) contaminant concentrations in the liver (winter flounder), hepatopancreas (lobster), and edible tissue (winter flounder, lobster and mussel) of these selected organisms.

The fish and shellfish monitoring program includes three surveys: (1) a flounder survey that is to obtain specimens of winter flounder *(Pseudopleuronectes americanus)* from four sampling sites in Boston Harbor and offshore for gross examination, histology, aging, and chemical analyses of tissue to determine sublethal effects of contaminant exposure and tissue burden; (2) a lobster survey that is to obtain specimens of lobster *(Homarus americanus)* from three sampling sites in Boston Harbor and offshore for gross examination and chemical analyses of tissues to determine health and tissue burden of contaminants; and (3) a mussel bioaccumulation survey that is to obtain, deploy, and recover blue mussels *(Mytilus edulis)* for determination of short-term accumulation of anthropogenic contaminants in mussel tissue (see Table 4).

1.4.2 Sampling Plan

The sampling sites and requirements are given in Table 4 (Pembroke et al. 2006). There are four sites for the flounder survey, three sites for the lobster survey and four sites for the mussel survey.

Table 4. Sampling Locations and Requirements for the Surveys

Survey	Sites	Sampling time	Number of field samples	Number of composite samples
Flounder	(1) Deer Island Flats (Boston Harbor) (2) Off Nantasket Beach (3) Offshore Effluent Outfall Site (4) Eastern Cape Cod Bay	late April	50 flounders at each site (Sexually mature winter flounder)	3 reps/site @ 15/ea: meat: 4x3=12 composites liver: 4x3=12 composites
Lobster	(1) Deer Island Flats (Boston Harbor) (2) Off Nantasket Beach (3) Eastern Cape Cod Bay	July	21 at each site (Commercially harvestable)	3 reps/site @ 7/ea: meat: 3x3=9 composites hepato: 3x3=9 composites
Mussels	Collect mussels from Stover's Point, Maine for both baseline and deployment studies. Then deploy the mussels in cage at 4 sites: (1) Boston Inner Harbor (2 deployments) [1] (2) "B" Buoy site (2 deployments) [1] (3) Off Deer Island Light (3 deployments) [1] (4) Outfall site (5 deployments) [2]	Jun-Aug (deploy for 45-60 days)	Baseline: (100 mussels) 110 mussels at each deployment (All mussels are ~ 6cm in length)	Baseline chemistry: 4 reps @ 25/ea Sites (1), (2), (3): 4 reps/site@25/ea. Site (4): 8 reps @25/ea Total = 24 composites

[1] Note: Extra deployments to account for possible losses of live mussels.
[2] Note: Four replicates are planned from the middle of the Outfall diffuser line and 2 replicates each from the east and west side of the diffuser line.

1.4.3 Tissue Chemical Analyses

The objective of tissue chemical analyses is to determine the body burdens of toxic substances and potential elevations of these body burdens caused by relocation of the outfall. Relevant to this QAPP, the tissue samples are collected and composited by Battelle Ocean Sciences (subconstultant to AECOM) and are analyzed by the DLS Central Lab. Flounder samples will consist of fillets and liver tissues which are dissected and composited (3 replicates of 15 flounder composited at each site). Lobster samples (meat and hepatopancreas) will also be composited (3 replicates of 7 lobsters composited at each site). After the collection of 1,200 mussel samples from a "clean" location in Maine, 4 replicates of 25 mussels (randomly chosen) are composited for baseline chemistry. The remaining mussels are deployed into four locations and from these

deployments 4 replicates of 25 mussels are collected and composited for Boston Inner Harbor, "B" Buoy, and Off Deer Island Light, and 8 replicates of 25 mussels are collected and composited for the Outfall site. The number of field samples collected (flounder and lobster) and mussels deployed are given in Table 4. The last column in Table 4 lists the number of replicates planned for each survey site. Upon compositing, a new sample ID number will be generated by AECOM to track the composite, maintaining a record of which specific fish and shellfish are included in each composite. The composite samples are shipped by AECOM on ice to DLS for chemical analysis.

The metals and organic compounds to be analyzed for each type of the tissue samples are given in Table 5. The detailed lists of metals and organic compounds are given in Table 6.

Table 5. Parameters to be analyzed in composited samples

Composite sample	Metals, other than Hg and Pb	Hg	Pb	PCBs	PAHs	Pesticides	% Lipids
Flounder meat		√		√		√	√
Flounder liver	√	√	√	√	√	√	√
Lobster meat		√		√		√	√
Lobster hepatopancreas	√	√	√	√	√	√	√
Mussels		√	√	√	√	√	√

1.5 Quality Objectives and Criteria for Measurement Data

The parameters measured, the precision, accuracy, and blank requirements, and the MDLs and RLs are listed in Table 6.

1.5.1 Quality Objectives

Data quality objectives are as follows:

- To ensure that parameters measured adequately describe the effects of effluent on fish and shellfish and their ecological environment, 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 analyzing samples collected on the fish and shellfish surveys to quantify chemical concentrations in the specimens of the receiving waters of interest; by analyzing laboratory QC sample to determine precision and accuracy, 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 2.3.2.

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 Representativeness

Representativeness is addressed primarily in sampling design. The sampling practices and laboratory measurements that are performed during the fish and shellfish monitoring have already been used in many systems to characterize marine tissue 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.

1.5.2.3 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.

1.5.2.4 Completeness

It is expected that 100% of the samples collected and intended for analysis will be analyzed. However, a sample loss of <5% for the entire project does not compromise the objectives of the project. Extra tissue left over from dissection will be archived at DLS until results are accepted by ENQUAD.

1.6 Special Training Requirements and Certification

Organic contaminant measurements and metals analysis for the HOM Fish and Shellfish study use routine laboratory analyses and data validation. Therefore, specialized training is not required. Once analysts have undergone the proper training in handling, storing, preparing, and analyzing tissue 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

Parameters	Lab Precision[2]	Accuracy[3]	Blank Cleanliness	MDL[1,5,6]	
				(dry weight)	
Trace metals				**MDL**	**RL[4]**
Silver (Ag)	≤ 25% RPD if value > 5*MDL	≤ 20% PD vs. SRM certified values	≤ 10% of the lowest sample concentration	0.009 ug/g	0.009 µg/g
Cadmium (Cd)				0.005 ug/g	0.003 µg/g
Chromium (Cr)				0.05 ug/g	0.07 µg/g
Copper (Cu)				0.1 ug/g	0.1 µg/g
Mercury (Hg)				0.0025 ug/g	0.0025 µg/g
Nickel (Ni)				0.12 ug/g	0.07 µg/g
Lead (Pb)				0.02 ug/g	0.024 µg/g
Zinc (Zn)				0.09 ug/g	0.15 µg/g
Polychlorinated biphenyls (PCBs)				**(wet weight)**	
2,4,-C12(8)	≤ 30% RPD	≤ 35% vs. SRM range	≤ RL[4] (2.0 ng/g)	0.299 ng/g	
2,2',5-C13(18)				0.280 ng/g	
2,4,4'-C13(28)				0.288 ng/g	
2,2',3,5'-C14(44)				0.233 ng/g	
2,2',5,5'-C14(52)				0.278 ng/g	
2,3',4,4'-C14(66)				0.301 ng/g	
3,3',4,4'-C14(77)				0.404 ng/g	
2,2',4,5,5'-C15(101)				0.189 ng/g	
2,3,3',4,4'-C15(105)				0.280 ng/g	
2,3',4,4',5-C15(118)				0.335 ng/g	
3,3',4,4',5-C15(126)				0.362 ng/g	
2,2',3,3',4,4'-C16(128)				0.303 ng/g	
2,2',3,4,4',5'-C16(138)				0.248 ng/g	
2,2',4,4',5,5'-C16(153)				0.269 ng/g	
2,2',3,3',4,4',5-C17(170)				0.253 ng/g	
2,2',3,4,4',5,5'-C17(180)				0.275 ng/g	
2,2',3,4,5,5',6-C17(187)				0.270 ng/g	
2,2',3,3',4,4',5,6-C18(195)				0.431 ng/g	
2,2',3,3',4, 4',5,5'6-C19(206)				0.394 ng/g	
Decachlorobiphenyl-C110(209)				0.347 ng/g	
2',3,5-trichlorobiphenyl (surrogate)				NA	
2,2',4,6',6-pentachlorobiphenyl (surrogate)				NA	
2,2',4,5',6-pentachlorobiphenyl (surrogate)				NA	
2,3,3',5,6-pentachlorobiphenyl (surrogate)				NA	

Parameters	Lab Precision[2]	Accuracy[3]	Blank Cleanliness	MDL[1,5,6]

Polynuclear aromatic hydrocarbons (PAHs)[7]				**(wet weight)**
Naphthalene				1.90 ng/g
C1-naphthalenes				1.90 ng/g
C2-naphthalenes				1.90 ng/g
C3-naphthalenes				1.90 ng/g
C4-naphthalenes				1.90 ng/g
1-methylnaphthalene	≤ 30% RPD	≤ 35% vs. SRM range	≤ RL[4] (5.0 ng/g)	0.610 ng/g
2-methylnaphthalene				1.16 ng/g
2,6-dimethylnaphthalene				1.11 ng/g
2,3,5-trimethylnaphthalene				0.970 ng/g
1-methylphenanthrene				1.24 ng/g
Acenaphthylene				0.670 ng/g
Acenaphthene				0.460 ng/g
Fluorene				0.730 ng/g
C1-fluorenes				0.730 ng/g
C2-fluorenes				0.730 ng/g
C3-fluorenes				0.730 ng/g
Phenanthrene				0.790 ng/g
Anthracene				0.600 /g
C1-phenanthrenes/anthracene				0.600 ng/g
C2-phenanthrenes/anthracene				0.600 ng/g
C3-phenanthrenes/anthracene				0.600 ng/g
C4-phenanthrenes/anthracene				0.600 ng/g
Dibenzothiophene				0.970 ng/g
C1-dibenzothiophenes				0.970 ng/g
C2-dibenzothiophenes				0.970 ng/g
C3-dibenzothiophenes				0.970 ng/g
Fluoranthene				0.550 ng/g
Pyrene				0.440 ng/g
C1-fluoranthenes/pyrene				0.440 ng/g
C2-fluoranthenes/pyrene				0.440 ng/g
C3-fluoranthenes/pyrene				0.440 ng/g
Benzo(a)anthracene				0.620 ng/g
Chrysene				0.550 ng/g
C1-chrysene				0.550 ng/g
C2-chrysene				0.550 ng/g
C3-chrysene				0.550 ng/g
C4-chrysene				0.550 ng/g
Benzo[b]fluoranthene				0.290 ng/g
Benzo[k]flouranthene				0.830 ng/g
Benzo[a]pyrene				0.330 ng/g
Benzo[e]pyrene				0.720 ng/g

Parameters	Lab Precision[2]	Accuracy[3]	Blank Cleanliness	MDL[1,5,6]
Polynuclear aromatic hydrocarbons (PAHs)[7]				**(wet weight)**
Dibenzo[a,h]anthracene				0.740 ng/g
Benzo[g,h,i]perylene				0.610 ng/g
Indeno[1 ,2,3-c,d]pyrene				0.440 ng/g
Perylene	≤ 30% RPD	≤ 35% vs. SRM	≤ RL[4] (5.0 ng/g)	0.370 ng/g
Biphenyl		range		0.500 ng/g
Dibenzofuran				0.360 ng/g
Benzothiazole				1.29 ng/g
Napthalene-D8 (surrogate)				NA
Chrysene-D12 (surrogate)				NA
Phenanthrene-D10 (surrogate)				NA
Pesticides				**(wet weight)**
Hexachlorobenzene				0.920 ng/g
Lindane				0.839 ng/g
Heptachlor	≤ 30% RPD	≤ 35% vs. SRM	≤ RL[4] (2.0 ng/g)	1.63 ng/g
Aldrin				0.803 ng/g
Heptachlorepoxide				0.366 ng/g
Alpha-Chlordane				0.158 ng/g
Trans-Nonachlor				0.213 ng/g
Dieldrin				1.85 ng/g
Endrin				0.612 ng/g
Mirex				0.226 ng/g
2,4'-DDD				0.322 ng/g
4,4'-DDD				0.266 ng/g
2,4'-DDE				0.253 ng/g
4,4'-DDE				0.294 ng/g
2,4'-DDT				0.303 ng/g
4,4'-DDT				0.277 ng/g
DDMU				0.250 ng/g
Gamma-Chlordane				0.325 ng/g
Cis-Nonachlor				0.131 ng/g
Oxychlordane				0.790 ng/g

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

[2] Relative Percent Difference (RPD)% = | (replicate 1 - replicate 2) | / (replicate 1 + replicate 2)/2 x 100.

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

[4] RL= reporting limit. The RL is the typical reporting limit, which is based on the low point of the calibration curve. Concentrations below the RL are reported, as 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 results are reported on a dry weight basis because analyses are performed on the freeze-dried tissue. Metals MDLs are based on 0.5 gram initial dry weight and 50 mL final volume (except mercury, which uses 0.2 g and has a final volume of 50 mL). MDL values are from ADOC #2008-59 (non-potable GFA), #2008-55 (Axial ICP for Zn), and #9829 (Cetac for Hg). Organics MDLs are based on a 2-gram initial weight of tissue, 100% solids but will be adjusted based on actual moisture content. MDL values are from ADOC #2004-29.

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

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.

1.7.2 Analysis Records

All data are recorded initially into bound laboratory logbooks, onto established data forms or into an 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. Currently the DLS is in the process of implementing a new LIMS system which is expected to be operational by July 1, 2009. This will be referred to as "LabWare LIMS" in this document.

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, weekly, and monthly by MWRA's MIS department. Once a month, the records are backed up onto tape and sent to an off-site location where they are kept for a period of ten 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 Methods Requirements

2.1.1 Sample Collection, Preparation, Preservation Procedures

Samples for each suite of analytes are collected and composited as described in Section 1.4.3. 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.

Table 7. Sample Collection and Storage

Parameter	Sample Mass (Target) (g)[a]	Sample Containers[b]	Shipboard Processing/ Preservation	Maximum Holding Time to Analysis
Metals	100	Clean, tared and labeled I-CHEM container	freeze (-20° C)	6 months after thawing to preparation and analysis; Hg holding time is 28 days after thawing to preparation and analysis
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

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

2.1.2 Sampling/Measurement System Failure Response and Corrective Action Process

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 2.7.6 for the DAIR process. Again, a corrective action may be initiated.

2.2 Sample Handling and Custody Requirements

2.2.1 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. Holding time for Hg and other metals begins when the samples are thawed after storage.

2.2.2 Sample Custody Procedure

The QAPP for fish and shellfish studies (Maciolek et al., 2008) describes sample tracking in the field. Field samples will be assigned IDs by AECOM and/or its subconsultant Battelle. Composited samples for analysis will be assigned LIMS IDs by DLS after sample reception in DLS's Central Lab. All composited samples are stored in a freezer in Battelle and then shipped to the Central Lab on Deer Island after the compositing of samples for each survey (Flounder, Lobster and Mussel, respectively) has been completed by Battelle. Upon receipt, the composited samples will be logged in by the Sample Management Team (Violet Team).

2.2.3 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,
  (3) all shipped samples have been received, and
  (4) holding temperatures were maintained.

- Completes the AECOM 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 AECOM Field Manager within 24 hours of receipt. **Note:** The original COC forms are sent to ENQUAD to be forwarded to AECOM 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. The LIMS 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 8. The archived samples (extra tissues) are also stored in the freezer with a copy of the original COC provided by AECOM/Battelle.

- Samples that are stored in the secure Sample Bank or freezer are in the custody of the Violet Team member who checked-in the samples until they are transferred from the Sample Bank to a member of laboratory staff for analysis. The receipt of samples by the analyst 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. Examples of the DLS internal LIMS Chain-of-Custody from current LIMS and LabWare LIMS are shown in Figure 2 and 3 respectively. (See Section 1.7.2).

- Sample archival and disposal are documented in LIMS.

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

Figure 2: DLS LIMS Internal Chain-of-Custody

3/01/2004 MWRA -LIMS 11:03:41
Internal Chain of Custody

ENTRY	Container #	Type	Current Storage Loc.	Responsible Person	Date and Time of Tran
4	04006748-01	FGF-CH	147-SAMPLE BANK	BERGER K	11:00:07 2/23/2004
3	04006748-01	FGF-CH	437-BIOLOGY LAB	BERGER-K	9:20:03 2/17/2004
2	04006748-01	FGF-CH	147-SAMPLE BANK	SEAMAN-C	13:46:2 12/09/2004
1	04006748-01	FGF-CH	141-SAMPLE RECVG	SEAMAN-C	13:44:33 2/09/2004

List of Revisions, Highlighted Fields have Changed
(RETURN) (RETURN) (RETURN) (RETURN)
Next Page Previous Page (RETURN) RETURN

Figure 3: LabWare LIMS Internal Chain-of-Custody

Internal Chain of Custody Record

Data Date and Time: 15-Apr-2009 10:03

Sample: M2009-0024467

Field Name:	LOCATION	New Field Value:	147-SAMPLE BANK
Audit Timestamp (GMT):	15-Apr-2009 14:03:20	Old Field Value:	421-ORGANIC PREP
Reason:	Internal Chain Of Custody	Changed By:	John Sculley

Field Name:	LOCATION	New Field Value:	421-ORGANIC PREP
Audit Timestamp (GMT):	15-Apr-2009 14:03:03	Old Field Value:	147-SAMPLE BANK
Reason:	Internal Chain Of Custody	Changed By:	John Sculley

Field Name:	LOCATION	New Field Value:	147-SAMPLE BANK
Audit Timestamp (GMT):	15-Apr-2009 14:02:28	Old Field Value:	141-SAMPLE RECVG
Reason:	Internal Chain Of Custody	Changed By:	John Sculley

Field Name:	LOCATION	New Field Value:	322-GLASS WASH 3
Audit Timestamp (GMT):	14-Apr-2009 14:09:43	Old Field Value:	CENTRAL
Reason:	Internal Chain Of Custody	Changed By:	Chris Newman

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2.3 Analytical Requirements

2.3.1 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.

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).

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 Tissue Sample Analyses to be Conducted by DLS

Parameter	LIMS test code	LabWare LIMS test code	Units	Instrument [1]	DLS SOP DCN[2]
Metals					
Silver	AG—TSICP, AG—TSGFA	ICP-TSRAD, GFA-TSABS	μg/g	ICP/GFA	#1195/ #1008/ #1150
Cadmium	CD—TSICP, CD—TSGFA	ICP-TSRAD, GFA-TSABS		ICP/GFA	#1195/ #1008/ #1150
Chromium	CR—TSICP, CR—TSGFA	ICP-TSRAD, GFA-TSABS		ICP/GFA	#1195/ #1008/ #1150
Copper	CU—TSICP, CU—TSGFA	ICP-TSRAD, GFA-TSABS		ICP//GFA	#1195/ #1008/ #1150
Mercury	HG—TSCVA	HG—TSCVA		CVA	#1236/ #1049
Nickel	NI—TSICP, NI—TSGFA	ICP-TSRAD, GFA-TSABS		ICP//GFA	#1195/ #1008/ #1150
Lead	PB—TSICP, PB—TSGFA	ICP-TSRAD, GFA-TSABS		ICP/GFA	#1195/ #1008/ #1150
Zinc	ZN—TSICP, ZN—TSGFA	ICP-TSRAD, GFA-TSABS		ICP/GFA	#1195/ #1008
PCBs	PCB-TSNOA	PES-TSSIM	μg/kg	GC/MS	#1188/ #1173
PAH	PAH-TSGMS	PAH-TSGMS	μg/kg	GC/MS	#1188/ #1030
Pesticides	PES-TSNOA	PES-TSSIM	μg/kg	GC/MS	#1188/ #1173
% Lipids	LIP-TSGRV	Pending	%	NA	Info. contained in SOP #1189
Dry weight [3]	NA	DRYWTSGRV	%	NA	Info. contained in SOP #1195

[1] When more than one instrument is listed, this is the order that would be applied. (i.e. First they are run on ICP, then GFA if necessary).
[2] 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.
[3] The sample dry weight is referred to as freeze dry weight (as stated in SOP #1195).

2.3.1.1 Organic Chemical Analysis

The MWRA Central Laboratory performs all organic fish and shellfish tissue chemistry analyses. Tissue samples are extracted for PAH, chlorinated pesticides, and PCB congeners by following MWRA SOP #1189, *Combined Tissue Sample Extraction by Sonication for PAH, Pesticides, and PCB Congener Analyses*. This extraction method utilizes sonication, and is based on EPA Method 3550B. Between 2 and 5 g of homogenized tissue is mixed with sodium sulfate and is serially extracted with methylene chloride (DCM) using sonication techniques. The sample is weighed in 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 in an Erlenmeyer flask through a powder funnel containing glass wool and sodium sulfate to remove any water and solid particles. After each extraction (total of three solvent additions) the filtered solvent is combined in the flask. If a percent lipids determination is to be performed, 10 mL of the total extract is removed and transferred to an aluminum weighing dish. The solvent is allowed to evaporate overnight and the pan is weighed for the percent lipids determination. The remaining extract is measured in a graduated cylinder and then concentrated to 1 mL using the TurboVap automatic concentrator technique. This concentrated extract is then processed through a silica gel cartridge and concentrated to 2 mL using the TurboVap automatic concentrator technique. The post-cleanup extracts are then 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, which is determined during metals analysis.

2.3.1.2 Metal Analysis

The MWRA Central Laboratory performs metals digestions and analyses for Ag, Cd, Cr, Cu, Ni, Pb, and Zn. Tissue samples are prepared by weighing, freeze drying, and then weighing again to determine the dry weight. Then tissue samples are digested using a nitric acid digestion according to DLS SOP #1195, *Preparation for Analysis of Total Elements in Tissue Samples by Microwave Digestion*. A 500 to 1000 mg aliquot of each homogeneous lyophilized sample is combined with 5 mL HNO_3 and 5 mL water 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 water (final volume is 50 mL). Digestates are analyzed by ICP according to DLS SOP #1008, *Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy*. Elements that are undetected by ICP may be analyzed by GFA (DLS SOP #1150, *Graphite Furnace Atomic Absorption Spectroscopy*) for lower reporting limits. 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 #1236, *Digestion of Tissue Samples for Mercury Analysis* and DLS SOP #1049, *Mercury Analysis by Cold Vapor Atomic Absorption Spectroscopy (CETAC M6000A).* A 200 mg lyophilized aliquot is cold-digested with 15 mL dilute HNO_3 and H_2SO_4 overnight. Samples are then heated in a 58^{o}C waterbath for 1 hour, then heated again at 80^{o}C for an additional 30 minutes. Cooled samples are further oxidized with $KMnO_4$ and $K_2S_2O_8$ overnight. Deionized water is added to bring the final sample volume to 50 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.

2.3.2 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.

2.4 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

All analytical equipment associated with tissue analyses (GC/MS, ICP, GFA, mercury analyzer, analytical balances, thermometers, and waterbaths) are calibrated and maintained according to manufacturer's specifications. Calibration is performed or checked as described in Section 10.0 of DLS' QAMP (DCN #5000) or the pertinent SOP. Equipment logbooks are maintained to document periodic maintenance of major equipment.

2.5 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.

2.6 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 Tissue Chemical Analyses

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 sample notepad and/or test_comments. If appropriate, flag with test_comment 'B' (Not blank corrected, blank >5x MDL)
Metals	1 per 20 samples	≤ 10% of the lowest sample concentration	
Accuracy			
Matrix Spike			
Organics	1 per 20 samples	≤35% vs. SRM range[2]	Document, justify deviations. Corrective action (*e.g.*, re-extraction, reanalysis, data qualifier) is documented in LIMS sample notepad and/or test_comments. Flag with test_comment 'Q' (accuracy does not meet DQO).
Metals	1 per 20 samples	PD ≤ 30%	
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 sample notepad and/or test_comments. Flag with test_comment '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 sample notepad and/or test_comments. Flag with test_comment 'Q' (accuracy does not meet DQO).
Metals	1 per 20 samples	PD ≤ 20% vs. SRM certified values[5]	
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 sample notepad and/or test_comments. Flag with test_comment 'R' (precision does not meet DQO).
Metals	1 per 20 samples	≤ 25% RPD if value is >5 X MDL	

[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 2.0 ng/g and for PAHs this is 5.0 ng/g based on 2 g initial weight, 100% solids.) 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) = | (replicate 1 - replicate 2) | / /(replicate 1 + replicate 2)/2 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.
	ICP [2]	1	See footnote 3	Prior to analytical run	± 10 % Rec.	Every 10 samples	
	GFAA [2] (as required)	3	R ≥ 0.995 [1]	Prior to analytical run	± 10 % Rec.	Every 10 samples	

[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] IPC: ± 5%, ICV: ±5%, ICB: <MDL, ICS: ±10%.

2.7 Data Management

2.7.1 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, test results are either entered manually into LIMS from laboratory logbooks, spreadsheets, or instrument data system printouts or are electronically transferred. In the current LIMS system, the LIMS worklist module (WKLIST) is used to create sample/test fields for routine internal laboratory QC parameters (method blanks, laboratory control samples, and laboratory duplicates). In the new LabWare LIMS system, the LIMS batch module (Batch Manager) is used for this. 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 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. Example data entry screens from both the current LIMS system and the new LabWare LIMS system for this project are shown in Figure 4 and 5 respectively. 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 2.7.4.

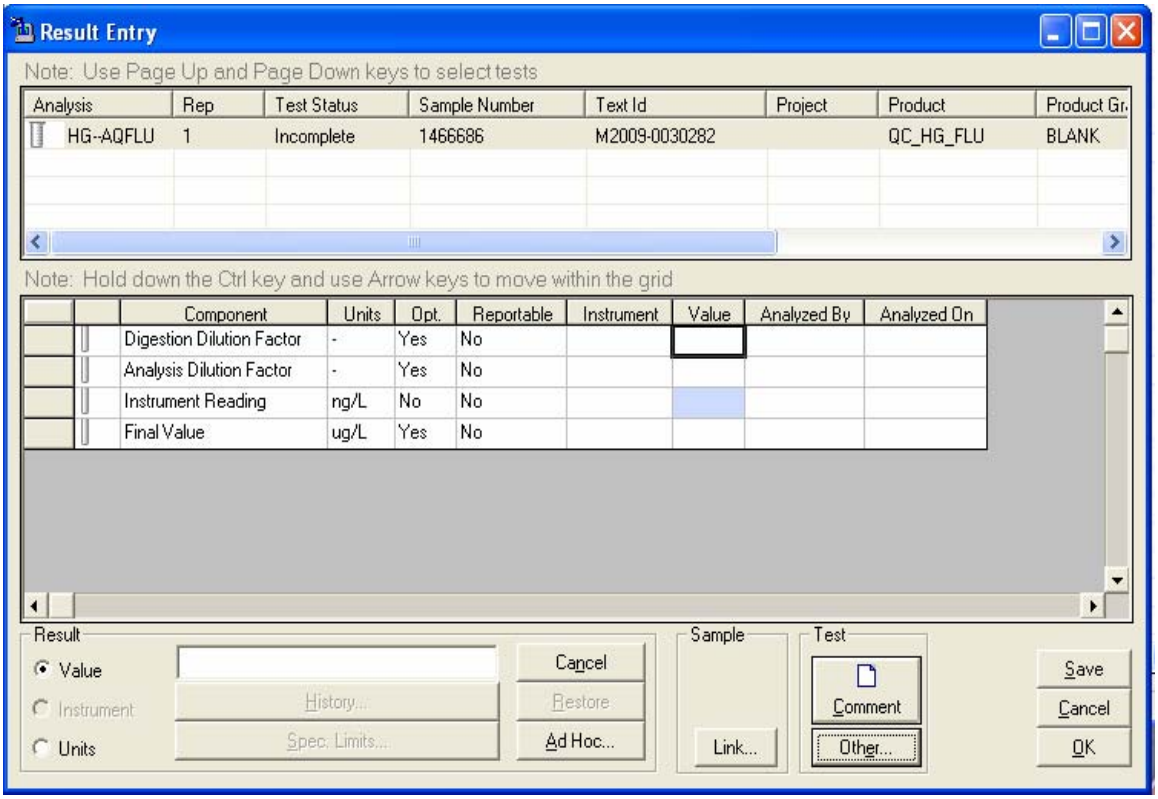
Figure 4: LIMS Data Entry Screen

```
SCNTE:TEST DATA ENTRY BY SAMPLE ID MWRA - LIMS DATE: 8/26/2004 TIME: 14:01:49
Sample ID: 04047809 Sample Due Date:  9/16/2004 Type: G  Sample Note Pad: (*)
SAMPLE STATUS: awaiting TESTING
TOC-SOCIR   Instrument :        Status : Pend Units of Measure : %
Sample ID : 04047809    Client: NPDES    Project: HOM-BN     Location: NF17
Container : 04047809-01   Lab: CENTRAL    Worklist Position:    Y/C/D:
Collected : 14:17:00   8/02/2004  Analysis Due Date:  8/30/2004  Notepad : ()
                   Analyst :____________________Analyzed :_________________
 Comment:_____________________________________________________________________
 TOTAL ORGANIC CARBON-SOLID-CO____________________   ____________________________
 TOTAL ORGANIC CARBON  RES_____________




Ready, Waiting for input!                              Page ( 1) of (25)
  Search Sample      Qualify All Data      Save Data       Control Chart
    Next Page         Previous Page       (Help/ More)          Exit
```

Figure 5: LabWare Data Entry Screen



2.7.2 Analysis Comments

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 are part of the LIMS database record for the analysis of the respective sample.

2.7.2.1 Comment Types

Comments are entered as either as free-flowing text (SAMPLE NOTEPAD COMMENTS) or as predefined text (Flags or TEST COMMENTS) in current LIMS, or as comments on individual results in LabWare LIMS.

2.7.2.1.1 Sample Notepad Comments

From time to time, the Analyst, Validator, and/or the Approver need to comment on the analyses. In such circumstances, the Validator/Approver uses the SAMPLE NOTEPAD COMMENT to enter a free-flowing text descriptive.

2.7.2.1.2 Test Comments

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 pre-defined text into the appropriate test comment field. The pre-defined qualifiers are listed in Table 11, below. If more than one test comment (qualifier) needs to be annotated, the pre-defined qualifier = X (See Sample Notepad) is used. The entry into the Sample Notepad contains the multiple qualifier codes and any free text deemed necessary. Note: When using the sample notepad in this manner, the comment must be prefaced with the test_code identifier. For example:

AG—TSICP: R; Precision does not meet data quality objectives. (Current LIMS)
ICP-TSRAD: R; Precision does not meet data quality objectives. (LabWare LIMS)

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

Table 11. Test Comments Qualifiers for Qualifying/Annotating Sample Test Results

LIMS Test Comment	Description
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.

2.7.3 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.

2.7.4 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 2.7.6, 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**.**

2.7.4.1 Validation of Analytical Results

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:** 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:** 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:** 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 invalid in LIMS unless the data validator has provided an explanation with a Validation Comment and a Sample Notepad Comment. Data that do not meet the Data Quality Objective of this plan are annotated (See Section 2.7.2 above). When all samples from a survey are approved in LIMS, the DLS HOM Project Manager notifies the ENQUAD Fish and Shellfish Project Manager and Data Management group.

2.7.5 Reporting of Results

All data are reported electronically to the ENQUAD Fish and Shellfish Project Manager as approved results in LIMS. Also, a QA Package (see 2.7.5.4, below) is to be forwarded to the ENQUAD Fish and Shellfish Project Manager immediately subsequent to the completion of the analyses of all survey samples.

2.7.5.1 Turnaround Times

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

2.7.5.2 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 TEST_COMMENTS in LIMS and the 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: Results for metals 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. Results are expressed in the units listed in Table 8.

2.7.5.3 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.

2.7.5.4 QA Package

Upon completing the chemical analyses, DLS forwards to the Fish and Shellfish Project Manager a QA Package (Figure 6) consisting of:

- The Metals QC results including SRM results and reference ranges
- Organics QC results including SRM results and reference ranges, MS/MSD results, and surrogate recoveries for all samples
- Any descriptive QA trail relevant to the delivered data (sample notepad comments)
- Any relevant audit reports
- A missing samples report
- Any relevant corrective actions
- Any relevant DAIRs
- The signed original Chains of Custody
- A QA statement from the DLS Project Manager and Section Manager

A separate package is needed for flounder, mussels and lobster. All information, including the signed QA statement is forwarded by inter-office mail to the Fish and Shellfish Project Manager.

Figure 6: Quality Assurance Statement

MWRA DEPARTMENT OF LABORATORY SERVICES

MWRA Harbor and Outfall Monitoring Project

Quality Assurance Statement

Description of Data Set or Deliverable: BF081 Survey (07/28/08 - 08/01/08)

1.0 Sample Analyses

All samples were handled, analyzed and reported according to the procedures and requirements specified in the CW/QAPP (*Prasse et al*., 2007), 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: 5000.0, 2003) and the CW/QAPP (*Prasse et al.*, 2007) 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	☐ Control Charts
☐ Corrective Actions	☒ Battelle COC Forms (Originals)
☐ DAIRs	☒ LIMS Notepad Comments

Comments:

All samples expected from this survey were received, and have been analyzed.

QC samples were analyzed in accordance with the DLS QAMP and the CW/QAPP, however some results were outside of the acceptance limits. QC results that fell outside of the acceptance criteria and their associated batch sample results are flagged with the appropriate qualifier(s) in the test comment field and/or in the sample notepad comment area.

SRM and QC recoveries for metals and organics are attached.

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)*

2.7.6 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. The client (ENQUAD) is notified of any corrections made as the result of a DAIR.

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.

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: 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 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 Fish and Shellfish 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, any outstanding issues that could affect data quality, and a QC narrative of activities, as described in Section 2.7.5.4, 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 and then approved by the appropriate Laboratory Manager and QA Coordinator. After required corrective action has been taken, the information is documented and verified to be effective and sufficient by the appropriate Laboratory Manager and QA Coordinator. All information is maintained in the Corrective Action QA files.

3.2 AECOM Environment

3.2.1 Performance and System Audits

The AECOM 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 Fish and Shellfish Monitoring QAPP (Maciolek et al., 2008). 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 AECOM's QAPP (Maciolek et al., 2008), "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 will be reported to Dr. James A. Blake, AECOM's Project Manager. He will be accountable to MWRA and to AECOM management for overall conduct of the Fish and Shellfish Monitoring Project, including the schedule, costs, and technical performance. Dr. Blake 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 AECOM management or with MWRA. He will be responsible for evaluating the overall impact of the problem on the project and for discussing corrective actions with the MWRA Fish and Shellfish Monitoring Project Manager and the MWRA/ENQUAD Program Manager,Water Quality.

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 tissue monitoring tasks, and any issues affecting data quality, are reported to the MWRA/ENQUAD Fish and Shellfish Project Manager, the MWRA/ENQUAD Program Manager,Water Quality, and to the AECOM Project Manager. The DLS HOM Project Manager and the ENQUAD Fish and Shellfish 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 AECOM Project Management.

3.3 Work Stoppage for Cause

The ENQUAD Fish and Shellfish Project Manager and the MWRA/ENQUAD Program Manager,Water Quality, in consultation and conjunction with the Director of DLS, have the authority to stop any and all work for cause.

3.4 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 Report and Rolling Audit Report. The QA Coordinator prepares the monthly QA Report and the Rolling Audit Report. Specific information resulting from any oversight activities is included in the QA Package (2.7.5.4) accompanying the survey results. Guidance for QA reporting can be found in Section 13.0 of DLS' QAMP (DCN #5000).

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 AECOM or ENQUAD 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 2.7 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. Following LIMS approval, data is transferred overnight from LIMS automatically to Plant Operations Management System (OMS) by tested automated routines. Transfer of data from OMS to EM&MS work tables is done by tested automated routines.

- Application of qualifiers in EM&MS is done by automated routines that parse test comments applied by the laboratory, or by the ENQUAD Fish and Shellfish 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 Fish and Shellfish 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 by the ENQUAD Fish and Shellfish 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 Fish and Shellfish Project Manager uses the data preview application HOM Review, written by ENQUAD using Oracle SQL*Forms, to review the analytical results, test comments, and LIMS notepad entries. Standard LIMS test comments are parsed into EM&MS

qualifiers. In order to review and assess the HOM results, the ENQUAD Fish and Shellfish 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 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 2.7.6.).

The ENQUAD Database Manager:

- Makes available for the ENQUAD Fish and Shellfish Project Manager's review: the Survey Samples Results Report, the Notepad comments Report, and the Test Comments Report.

- 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 OMS.

- 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 QA statement, test comments, sample notepad comments, and ENQUAD Fish and Shellfish Project Manager review along with the data.

4.1.4.2 Data Validation/Fitness-for-Use

The ENQUAD Fish and Shellfish 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 AECOM. This QAPP does not address sampling design.

4.1.4.4 Data Transmittal to AECOM

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 AECOM.

4.1.4.5 Data Analysis

Data are analyzed and reported by ENQUAD.

5.0 REFERENCES

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