14.2 Preparation Batch QC Samples.

A summary of the minimum required QC samples for each preparation batch are as follows. All calibrations and QC samples analyzed shall be uniquely identified and traceable to that unique sample preparation batch.

14.2.1 Method Blank.

Method blanks are analyzed to assess background interference or contamination that exists in the analytical system that might lead to the reporting of elevated concentration levels or false positive data. The method blank is defined as an interference-free blank matrix similar to the sample matrix to which all reagents are added in the same volumes or proportions as used in sample preparation and carried through the complete sample preparation, cleanup, and determinative procedures. For aqueous analyses, analyte-free reagent water would typically be used. For soil analyses, a purified solid matrix (e.g., sand) would typically be used except for metals analyses. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. Sample results shall not be corrected for blank contamination.

14.2.2 Ongoing Precision and Recovery.

The Ongoing Precision and Recovery (OPR) is analyzed to assess general method performance by the ability of the laboratory to successfully recover the target analytes from a control matrix. The OCR is similar in composition to the method blank. For aqueous analyses use analyte-free reagent water. For soil analyses, a purified solid matrix (e.g., Ottawa sand, sodium sulfate, or other purified solid) would typically be used. However, due to the difficulty in obtaining a solid matrix which is metals-free, analyte-free reagent water is taken through the appropriate digestion procedures for metals analyses. The OPR is spiked with all single-component target analytes before it is carried through the preparation, cleanup, and determinative procedures. The use of solid standard reference materials (SRMs) as the OPR is discouraged for they do not typically include all target analytes, and the acceptance limits associate with them are wide – due to the heterogeneity of the spiked matrix. Instead, the use of an interference-free matrix (e.g., purified solid, or sodium sulfate) is suggested. When samples are not subjected to a separate preparatory procedure (i.e., aqueous Hg analysis), the Continuing Calibration Verification (CCV) may be used as the OPR, provided the CCV acceptance limits are used for evaluation. The spiking levels for the OPR would normally be set at the project-specific action limits assuming that the low standard used for the initial calibration was below this limit. If the low standard used was at this limit or if the site action levels were unknown, then the spiking levels would normally be set between the low and mid-level standards. The results of the OCR are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples. The laboratory shall also maintain control charts, or tables, for these samples to monitor the precision and bias of the method. The precision may be evaluated by comparing the results of the OCR from batch to batch, or by duplicate OCRs. Duplicate OCRs within the same batch are not required, but recommended.

14.2.3 Matrix Spikes.

The matrix spike (MS) is used to assess the performance of the method as applied to a particular matrix. A MS is an environmental sample to which known concentrations of all target analytes have been added before sample manipulation from the preparation, cleanup, and determinative procedures have been implemented. These spiking levels would normally be set at the same level as the OPR. For solid samples, care should be taken to ensure that the original field sample is properly divided into homogeneous fractions. Aqueous samples require the submittal of additional sample for several parameters. Therefore, the sample to be used for the MS would normally be specified in the field to ensure that sufficient sample was available to perform the test. From the laboratory perspective, preparation batches comprised of the same matrix require the frequency of MS analysis at one per preparation batch. The merging of the MS frequency requirements from the project(s) and laboratory perspectives may be difficult for the laboratory to implement. For instance, batches comprised of samples of the same matrix from multiple sites may require additional matrix spikes to meet project requirements to evaluate the samples included within the batch. An MS from one site cannot be used to evaluate the matrix effects on samples from other sites. The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. Sample results shall not be corrected for MS QC excursions.

14.2.4 Matrix Duplicates (Replicates) or Matrix Spike Duplicates.

The matrix duplicate (MD) and/or matrix spike duplicate (MSD) is used to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. MD or MSD sample(s) are analyzed to assess the precision of the method in an actual matrix. A MSD is a duplicate of the MS as previously described. A MD is an environmental sample that is either divided into two separate aliquots by the laboratory, or requires the submittal of an additional sample. When applicable, care should be taken to ensure that the sample is properly divided into homogeneous fractions. Both the MD and MSD are carried through the complete sample preparation, cleanup, and determinative procedures. The normal use of these QC samples would follow the same requirements as described for the MS. A MD would normally be included with each preparation batch of samples processed where target analytes were expected to be present. A MSD would normally be included with each preparation batch of samples processed where target analytes were not expected to be present. As a general rule of thumb, the MSD would normally be used for the organic methods and the MD would normally be used for the inorganic methods. The results of the MD or MSD are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the precision of the analysis.

14.2.5 Surrogates.

Surrogates are analyzed to assess the ability of the method to successfully recover these specific analytes from an actual matrix. Surrogates are organic compounds that are similar to the analytes of interest in chemical behavior, but are not normally found in environmental samples. Surrogates

to use are identified within the determinative methods. Other compounds may be chosen and used as surrogates, depending on the analysis requirements, whenever they are representative of the compounds being analyzed, and whenever they cover the chromatographic range of interest. These compounds should be spiked into all samples and accompanying QC samples requiring GC, LC, or GC/MS analysis prior to any sample manipulation. Surrogates are used in much the same way that MSs are used, but cannot replace the function of the MS. The results of the surrogates are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the individual sample determinations. Control charts, or tables, shall be maintained for surrogates contained within the OPR and MB to monitor the accuracy of the method for each particular matrix. These charts shall be utilized in the same manner as the OPR and MS control charts. Sample results shall not be corrected for surrogate excursions.

14.2.6 Standard Reference Materials.

Laboratories are encouraged to analyze additional natural matrix standard reference materials (SRMs) and participate in external performance evaluation (PE) programs.

15. Element B6 — Instrument/Equipment Testing, Inspection, and Maintenance Requirements.

See guidance in USEPA 1998b, B6.

16. Element B7 — Instrument Calibration and Frequency.

See guidance in USEPA 1998b, B7.

17. Element B8 — Inspection/Acceptance Requirements for Supplies and Consumables.

See guidance in USEPA 1998b, B8.

18. Element B9 — Data Acquisition Requirements (Non-direct Measurements).

See guidance in USEPA 1998b, B9.

19. Element B10 — Data Management.

See guidance in USEPA 1998b, B10.

20. Element C1 — Assessments and Response Actions.

See guidance in USEPA 1998b, C1.

21. Element C2 — Reports to Management.

See guidance in USEPA 1998b, C2.

22. Element D1 — Data Review, Validation, and Verification Requirements.

See guidance in USEPA 1998b, D1.

23. Element D2 — Validation and Verification Methods.

See guidance in USEPA 1998b, D2.

24. Element D3 — Reconciliation with Data Quality Objectives.

See guidance in USEPA 1998b, D3.

III. References

ASTM D 4840 –95, *Standard Practice for Sampling Chain of Custody Procedures*

Plumb 1997. Plumb, R. H., Jr. (1997) "Standard guidance for the preparation quality assurance project plans," Technical Report D-97-1, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

USACE/USEPA 1991. *Evaluation of Dredged Material Proposed for Ocean Disposal, Testing Manual*, ("Green Book"). EPA-503/8-91/001. US Army Corps of Engineers and US Environmental Protection Agency (OTM).

USEPA 1996. *Guide to Method Flexibility and Approval of EPA Water Methods,* Draft December 1996.

USEPA 1998a. U.S. Environmental Protection Agency, *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5, External Review Draft Final October 1998. http://es.epa.gov/ncerqa/qa/qa_docs.html#top

USEPA 1998b. U.S. Environmental Protection Agency, *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5, February 1998. http://es.epa.gov/ncerqa/qa/qa_docs.html#top

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US Environmental Protection Agency, *QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations,* Chemical Evaluations, Office of Water, EPA 823-B-95-001, April 1995

Table 1. SAP or QAPP Elements.

Group A: Project management Elements	
A1	Title and Approval Sheet
A2	Table of Contents
A3	Distribution List
A4	Project/Task Organization
A5	Problem Definition/Background
A6	Project/Task Description
A7	Quality Objectives and criteria for Measurement Data
A8	Special Training Requirements/Certification
A9	Documentation and Records
Group B: Measurement/Data Acquisition Elements	
B1	Sampling Process Design (Experimental Design)
B2	Sampling Methods Requirements
B3	Sample Handling and Custody Requirements
B4	Analytical Methods Requirements
B5	Quality Control Requirements
B6	Instrument/Equipment testing, Inspection, and Maintenance Requirements
B7	Instrument Calibration and Frequency
B8	Inspection/Acceptance Requirements for Supplies and Consumables
B9	Data Acquisition Requirements (Non-direct Measurements)
B10	Data Management
Group C: Assessment/Oversight Elements	
C1	Assessments and Response Action
C2	Reports to Management
Group D: Data Validation and Usability	
D1	Data Review, Validation, and Verification Requirements
D2	Validation and Verification Methods
D3	Reconciliation with User Requirements

APPENDIX A
REPORTING REQUIREMENTS

When sampling, testing, and analyses are complete, applicants should provide the DMMO with a report containing adequate information to make a decision regarding suitability of the material tested. DMMO will use the report to determine if the material proposed for dredging is suitable for unconfined aquatic disposal (SUAD).[3] The report should include a cover sheet, table of contents, case narrative, the analytical results, and internal laboratory QA/QC information. The laboratory report should be organized such that the analytical results are reported on a per batch basis. Cross referencing the SAP or QAPP for unchanged previously submitted information is encouraged.

Cover Sheet. The cover sheet should specify the following information:

- Title of Report
- Name and location of laboratory(to include a point of contact, phone and facsimile numbers
- Name and location of any subcontractor laboratories, and appropriate test method performed
- Client name and address
- Project name and site location
- Statement of data authenticity and official signature and title of person authorizing report release
- Amendments to previously released reports shall clearly identify the identification for the previous report ands state the reason(s) for reissuance of the report

Table of contents. Reports should be organized in a format that allows for easy identification and retrieval of information.

Case narrative. A case narrative should be included in each report. The case narrative should contain a table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed and by which laboratories. Samples that were received but not analyzed should also be identified. Extractions or analyses that are performed out of holding times should be appropriately noted. The case narrative should define all data qualifiers. Deviations of any calibration standards or QC sample results from appropriate acceptance limits should be noted and associated corrective actions taken by the laboratory should be discussed. Any other factors that could affect the sample results should be noted.

Body of Report. The body of the report should contain the following elements:

[3] The Marine Protection, Research, and Sanctuaries Act (MPRSA) applies if the proposed disposal site is the San Francisco Deep Ocean Disposal Site (SF-DODS), and the Clean Water Act (CWA) applies if disposal is proposed in the Bay.

- A plan view of the dredging project area showing the actual sampling locations (highlighting and explaining any deviation from the locations proposed in the approved SAP or QAPP). Each sample location should be recorded using a uniform identification scheme and latitude and longitude reported for each sample. The benchmark(s) used should be specified.
- Sampling equipment used.
- Positioning method(s) and equipment.
- Field logs for each sample/station, including dates and time of sample recovery, weather conditions and logistical problems, core length, sampling depth and any other pertinent information.
- Core logs/descriptions: list sediment descriptive parameters and distinguishing characteristics (e.g., odors, visual stratification, debris, biological activity, oil sheen).
- All Chain of Custody forms.
- Subsampling protocols, including a discussion of method(s) and equipment. Split samples, or field duplicate samples, including rationale and use, should be clearly described.
- Compositing procedures, methodology and justification, including method to determine homogeneity of composites.
- Testing Results of physical, chemical, and bioassay testing should include the following information at a minimum. (Information need not be repeated if noted elsewerhe in the report):
  - Laboratory name and location (city and state)
  - Project name and unique ID
  - Field sample ID as written on custody form
  - Laboratory sample ID
  - Matrix (sediment, water, etc.)
  - Date sample collected
  - Date sample extracted or prepared
  - Date sample analyzed
  - Method numbers for all preparation, cleanup, and analysis procedures employed
  - Batch identification
  - Analyte or characteristic
  - Minimum levels
  - Method detection limits
  - Test results with correct number of significant figures
  - Any data qualifiers assigned
  - Concentration units
  - Dilution factors
  - Percent moisture or solids (all sediment are to be reported on a dry weight basis)
- Theoretical Bioaccumulation Potential (TBP) calculations, if appropriate.
- Statistical Analyses.
- Overlying and interstitial water quality monitoring data for bioassay testing.
- Sediment storage and archiving procedures.
- QC results including a discussion of any qualified data and any corrective actions taken.

- Any deviations from the approved SAP or QAPP, including a discussion of the impact of the deviations on data quality.

Table 2. Recommended Sediment Sample Sizes, Containers, Storage Criteria, and Holding Times, for Physical, Chemical, and Sediment Biological Testing

Parameter	Sample Size[A]	Container Type[B]	Storage	Holding Time[C]
Total Solids[TS]	50g	G,PE,PP	4°±2°C	7 days
Total Organic Carbon [TOC]	50g	G,PE,PP	4°±2°C	14 days
Grain Size	100g (60mL)	G,PE,PP	4°±2°C	6 months
Total Volatile Solids [TVS]	50g	G,PE,PP	4°±2°C	7 days
Soluble Sulfides	50g	G,PE,PP	4°±2°C	7 days
Metals	200g (600 mL)	G,PE,PP, PTFE	4°±2°C	6 months 28 days for Hg
Butyltins	200g (250ml)	G[D]	4°±2°C	14 days (until extraction) 40 days (after extraction)
Pesticides	200g (250ml)	G[D]	4°±2°C	14 days (until extraction) 40 days (after extraction)
PCBs	200g (250ml)	G[D]	4°±2°C	14 days (until extraction) 40 days (after extraction)
PAHs	200g (250ml)	G[D]	4°±2°C	14 days (until extraction) 40 days (after extraction)
Water Column Toxicity	3 L[E]	G, PP, LP	4°±2°C	14 days[F]
Benthic Toxicity	1 L[E]	G, PP, LP	4°±2°C	14 days[F]
Bioaccumulation	3.5 L [EG]	G, PP, LP	4°±2°C	14 days[F]

A. Recommended sediment or tissue sample size in wet weight (volumetric equivalents are given for field collection of sediments). The amounts shown are not intended as firm values. The sample volumes indicated should be sufficient to allow for an analysis, a replicate or matrix spike replicate and enough to repeat the analysis if necessary. If additional laboratory analyses are required (e.g., laboratory replicates, allowance for retesting), the field sample size should be increased accordingly. To reduce detection limits, it may be necessary to use a larger sample size (in conjunction with a sample cleanup step and smaller extract volume for GC/MS analysis). For some chemical analysis, smaller samples sizes may be used if comparable sensitivity can be obtained by adjusting instrumentation extract volume, or other factors of the analysis.

B. G = borosilicate glass; PE = linear polyethylene; PP = polypropylene; PTFE = Teflon; LP = Epoxy- or Phenoxy Acetate-lined pails.

C. Holding times indicate maximum elapsed time between sample collection and sample analysis. These holding times are for sediment and tissue based on administrative guidance. There are no promulgated, scientifically based holding time criteria available.

D. Sample containers should be 250-mL widemouth glass with Teflon lined lid.

E. Recommended minimum field collection for a sample that is expected to be composited with three other samples for analysis. This includes sufficient material for archiving in case the test has to be repeated. Actual volumes to be collected may have to be increased (e.g., to accommodate the number of species or to provide a margin of error).

F. The Inland Testing Manual gives a maximum holding time of 8 weeks for samples used for biological testing. However, DMMO recommends that testing commence as soon as possible after test collection to minimize confounding factors that may be associated with holding sediments.

G. *Macoma* and *Nephtys* test species can be run in the same container, thus reducing the overall quantity of sediment required for bioaccumulation testing when these two species are used.

Table 3. Recommended Methods for Sample Preparation, Cleanup, Analysis, and Detection Limits for Sediments and Tissues.

Characteristic	Preparation Method	Cleanup Methods[a]	Analysis Method	Sediment Minimum Level[b]	Tissue Minimum Level[b]
Total Solids [TS](%)	-	-	2540G[c]	0.1	-
Total Organic Carbon [TOC](%)	Acidify to release carbonates	-	5310B[c]	0.1	-
Total Ammonia (mg/kg)	-	-	4500-NH_3D[c]	0.2	-
Soluble Sulfides (mg/kg)	Zinc acetate preserve	-	4500-S^{2-}G[c];	0.1	-
Grain Size (%)	-	-	ASTM[d]; Plumb[e]	1	-
Total Lipid (%)	-	-	Bligh and Dyer[f]	-	0.1
Total Volatile Solids [TVS] (%)	-		2540G[c];	0.1	
Arsenic (mg/kg)	33051[g]	-	6020[g];	5.0	1
Cadmium (mg/kg)	3051[g]	-	6020[g];	0.3	0.1
Copper (mg/kg)	3051[g]	-	6020[g]	5.0	1.0
Chromium (mg/kg)	3051[g]	-	6020[g]	5.0	1.0
Lead (mg/kg)	3051[g]	-	6020[g]	5.0	1.0
Mercury (mg/kg)		-	245.6[i];7471A[g]	0.02	0.02
Nickel (mg/kg)	3051[g]	-	6020[g]	5.0	1.0
Silver (mg/kg)	33051[g]	-	6020[g]	0.2	1.0
Selenium (mg/kg)	3051[g]	-	6020[g]	1.0	0.5
Zinc (mg/kg)	3051[g]	-	6020[g]	1.0	1.0
PAHs[h] (µg/kg)	3545[g]	3640A[g]/3660B[g]	8270C[g]	20	20

Table 3. Recommended Methods for Sample Preparation, Cleanup, Analysis, and Detection Limits for Sediments and Tissues.

Characteristic	Preparation Method	Cleanup Methods[a]	Analysis Method	Sediment Minimum Level[b]	Tissue Minimum Level[b]
Pesticides[i] (µg/kg)	3545[g]	3620B[g]/3640A[g] /3660B[g]	8081A[g]	2	2
PCBs[j] (µg/kg)	3545[g]	3620B[g]/3640A[g]/ 3660B[g]/3665A[g]	8082[g]	20	20
Butyltins (µg/kg)			Rice[k]	10	10
Interstitial **Water Measurements**					
Salinity			2520[c]	0.5 ppt	-
pH			4500-H^{+c}	0.1 pH units	

a. Alternative cleanup procedures are described in U.S. EPA SW846 (1986). Additional cleanup procedures may be necessary on a sample-by-sample basis.

b. Sediment minimum levels are on a dry-weight basis. Tissue minimum levels are on a wet-weight basis. To achieve the recommended minimum levels for some compounds in sediment, it may be necessary to use a larger sample size than the method describes, a smaller extract volume for gas chromatography/mass spectrometry analyses, and one of the recommended sample cleanup methods, as necessary, to reduce interference.

c. Standard Methods for the Examination of Water and Wastewater, 19th Edition 1995.

d ASTM D1234

e. Procedures for Handling and Chemical Analysis of Sediment and Water Samples, Russell H. Plumb, Jr., EPA/CE-81-1, May, 1981, Particle Size, Method 2, apparent particle-size distribution.

f. Bligh and Dyer, 1959. A rapid method of total lipid extraction and purification. Can. J. Biochem. Physiol. 37:911-917. Dichloromethane must be used as the extraction solvent [EPA 823-R-95-007, Guidance for Assessing Chemical Contaminant Data for use in Fish Advisories, Volume 1: Fish Sampling and Analysis, 2nd Edition, 8.2.1, pg.8-1]

g. SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Revision 3 (Nov 1986), as amended by

Table 3. Recommended Methods for Sample Preparation, Cleanup, Analysis, and Detection Limits for Sediments and Tissues.

Updates I (Jul 1992), II (Sep 1994), IIA (Aug 1993), IIB (Jan 95), and III (Dec 96).

h. Includes fourteen PAH compounds (LPAHs: naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene; HPAHs: fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b&k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-c,d)pyrene, dibenzo(a,h,)anthracene, benzo(g,h,i)perylene

i. Includes: Aldrin, a-BHC, b-BHC, g-BHC (Lindane), d-BHC, Chlordane, 4,4=-DDD, 4,4=-DDE, 4,4=-DDT, Dieldrin, Endosulfan I and II, Endosulfan sulfate, Endrin, Endrin aldehyde, Heptachlor, Heptachlor epoxide, and Toxaphene.

j. Includes Aroclors 1242, 1248, 1254, and 1260. **OR** Includes individual PCB congeners.

k. Rice C.D., F. A. Espourteille, and R. J. Huggett. 1987 Analysis of tributyltin in estuarine sediments and oyster tissue, *Crassostrea virginica*. Applied Organometallic Chemistry, 1:541-544.

Table 4. Minimum Laboratory Quality Control[1]

Analysis Type	Method Blanks[2]	Surrogates[3]	Duplicate[2,4]	Ongoing Precision And Recovery[2,5,6]	Matrix Spike[2,5,7]	Matrix Spike Duplicate[2,4,5,7]
Total Solids	X		X	X		
Total Organic Carbon	X		X	X	X	
Ammonia	X		X	X	X	
Sulfides	X		X	X	X	
Particle (Grain) Size			X			
Total Lipid	X		X	X		
Total Volatile Solids	X		X			
Metals	X		X	X	X	
PAH	X	X		X	X	X
Pesticides	X	X		X	X	X
PCB	X	X		X	X	X
Butyltins	X	X		X	X	X

1. The laboratory may (and is encouraged to) perform and report more than the minimum required quality control. All quality control cited in the method specified in the sampling and analysis plan (SAP) must also be performed.
2. Prepare, extract, clean up, and concentrate the QC sample with each sample batch; batches are normally limited to 20 samples of the same matrix started through the extraction or preparation process on the same 12-hour shift.
3. Surrogate spikes are required for every sample, including method blanks, ongoing precision and recovery (OPR), reference materials, matrix spikes, and all actual samples including duplicates.
4. Duplicates are to be run when a hit is expected (e.g., most metals) and a matrix spike is to be run when a hit is not expected. The purpose of these duplicates is to access precision. Replicating a sample with a non-detect value does not provide a measure of precision. Likewise, spiking a sample with a native background level is often ineffective because of the ratio of the spike level to the native background level.
5. All analytes will be included in the OPR and matrix spikes.
6. The OPR test, sometimes termed a "laboratory control sample," "quality control check sample," "blank spike," or "laboratory-fortified blank," is used to ensure that the laboratory remains in control during the period that samples are analyzed, and it separates laboratory performance from method performance in the sample matrix. The test consists of a single aliquot of reference matrix spiked with the analyte(s) of interest and carried through the entire analytical process with each batch of samples. The concentration of the target analyte(s) in the spike solution may vary between one and five times the concentration used to establish the lowest calibration point (i.e., one to five times the minimum level [ML]).
7. Matrix spikes and Matrix spike duplicates must be performed on project samples. The recoveries of the analytes, relative to the spike, are determined in each sample. The precision of the determinations also is assessed by measuring the relative standard deviation of the analyte concentrations measured in the MS and MSD. The MS and MSD samples should each be spiked at a level that results in the concentration of the target analytes being one to five times the background concentration of unspiked field samples or at the level specified in the method, whichever is greater.

Table 5. Acceptance Limits and Corrective Actions For Minimum Quality Control

QC Category	Acceptance Limits	Corrective Action
Method Blank (MB)	< 2.2MDL or ≥0.10 analyte level	Correct source of contamination, obtain acceptable blank values, and prepare fresh aliquots of the samples and reanalyze.
Surrogate[1]		[2]
Duplicate RSD	25	
OPR	18	The source of the problem must be identified and resolved before continuing analyses. Prepare fresh aliquots of the samples and reanalyze[2].
MS & MSD[1]	35	If MB and OPR are in control the problem is judged to be matrix related, not system related. Evaluate need to perform method of standard addition (MSA) or the data need to be qualified as suspected matrix effect[2].
MS & MSD RSD	25	

1. It is essential that laboratories calculate in-house performance criteria for matrix spike recoveries and surrogate recoveries. The development of in-house performance criteria and the use of control charts or similar procedures to track laboratory performance cannot be over-emphasized. Many data systems and commercially available software packages support the use of control charts. Once established, control limits and warning limits for surrogates should be reviewed after every 20-30 field samples of the same matrix, and should be updated at least semi-annually. The matrix spike and surrogate results should all be generated using the same set of extraction, cleanup, and analysis techniques. For example, do not mix results from sediment samples extracted by ultrasonic extraction with those extracted by Soxhlet. In-house QC limits must be examined for reasonableness. It is not the intent to legitimize poor recoveries that are due to the incorrect choice of methods or spiking levels.

2. Any matrix spike, surrogate, or OPR results outside of the control limits require evaluation by the laboratory. Such actions should begin with a comparison of the results from the samples or matrix spike samples with the OPR results. If the recoveries of the analytes in the OPR are outside of the control limits, then the problem may lie with the application of the extraction or cleanup procedures applied to the sample matrix or with the analytical procedures. Once the problem has been identified and addressed, corrective action may include the reanalysis of samples, or the extraction and analysis of new sample aliquots, including new matrix spike samples and OPR.

Table 6. Minimum Laboratory Quality Control[1] for Bioassay Testing.

Analysis Type	Reference Toxicity Test	Dissolved Oxygen Concentration	Test Chamber Temperature	Control Organism Health	Age of Organism	Organism Maintenance	Salinity
ASTM E 724	X	X	X	X	X		
ASTM E 729	X	X	X	X		X	
ASTM E 1022	X	X	X	X		X	
ASTM E 1367	X	X	X	X		X	
ASTM E 1463	X	X	X	X			X
ASTM E 1562	X	X	X	X			X
ASTM E 1611	X	X	X	X	X	X	
ASTM E 1688							