

Appendix C

Analytical Data

Data Quality Evaluation Report/Quality Assurance Project Plan

Summary

All (100 percent) of the data from Bolsa Chica Lowlands soil/sediment and water samples collected during the ERA Sampling (September 1998 through June 1999) and Focused Sampling (January 2000 through September 2000) were reviewed. Guidance for this data quality evaluation came from the Bolsa Chica Lowlands Site Specific Quality Assurance Project Plan (QAPP), USEPA method guidance documents, and the USEPA CLP National Functional Guidelines for Inorganic and Organic Data Review, February 1994. The calibration and quality control (QC) acceptance criteria are specified in the QAPP, which is included in this appendix. Chains-of-custody and summaries of calibration and QC results, including those for method blanks, laboratory control samples, duplicate matrix spikes, and laboratory duplicates, were reviewed. All data were received in hardcopy and electronic format. This report includes an evaluation of data quality for all samples collected from the site and analyzed as part of the ERA Sampling and Analyses, as well as those collected for characterization of materials that may be dredged.

The results of the data quality evaluation process can be summarized as follows (differences between sampling programs are discussed separately):

- Overall, the project data quality objectives for precision, accuracy, representativeness, completeness and comparability were met.
- There were holding time exceedances resulting in some analytes being qualified as estimated detects and non-detects. A few of the results were rejected as a result of holding time exceedances that were greater than method and QAPP specified holding times. Overall, the qualifications resulting from holding time exceedances involved approximately 0.7 percent of the results and less than 0.05 percent of the results were rejected.
- Matrix effects were evident for some analytes based on the matrix spike, surrogate and field duplicate results. Most of these were noted for sediments and tissues, and were expected because of the complexity of the sample matrices. Most of the matrix recovery failures were associated with the presence of high concentrations of chlorides in the samples. The matrix spike, surrogate and field duplicate deviations resulted in approximately 1.5 percent of the results being qualified as estimated detects and non-detects.
- Method blanks for method SW8270 routinely indicated phthalate contamination and may have affected the sensitivity required to meet the project objectives.
- **ERA Sampling.** There were calibration difficulties with some analytes resulting in a few results being rejected and some being qualified as estimated detects and non-detects.

The rejections were a result of failure to meet the minimum instrument response, and involved one analyte (2,4-dinitrophenol). Overall, the qualifications resulting from calibration difficulties involved approximately 3 percent of the results.

- **Focused Sampling.** There were calibration difficulties with some analytes resulting in results being qualified as estimated detects and non-detects. Overall, the qualifications resulting from calibration difficulties involved approximately 0.7 percent of the results.
- **Focused Sampling.** There were laboratory control sample (LCS) exceedances resulting in 443 results for some analytes being qualified as estimated detects and non-detects. A few of the non-detect results were rejected because of an LCS recovery of zero. Overall, the qualifications as a result of LCS exceedances involved approximately 1.3 percent of the results and less than 0.02 percent of the results were rejected.
- **ERA Sampling.** Approximately 1 percent of positive results for pesticides and PCB congeners were qualified as estimated given differences between the primary and confirmation results exceeding the acceptance criterion. The differences were mostly a result of interference from coeluting Aroclor peaks when at least one Aroclor was present.
Focused Sampling. Approximately 0.8 percent of positive results for were qualified as estimated because of differences between the primary and confirmation results exceeding the acceptance criterion. The majority of the data affected were for pesticides.
- In samples that contained Aroclors, some of the Aroclor peaks coeluted within the retention time windows for some of the pesticides on both the primary and confirmation columns. This made the identification of some of the pesticides that were reported questionable. The use of other confirmation techniques, such as GC/MS, should be considered in the future.

Validation Flags

Validation flags follow the common conventions of:

U Not Detected

The analyte was analyzed for, but not detected above the method detection limit.

J Estimated Value

The analyte was detected, but the reported value may not be accurate or precise. These data, however, should be usable in estimating the contamination at the site.

UJ Estimated Detection Limit

The analyte was analyzed for, but qualified as not detected. The result is estimated.

R Rejected

The analyte was analyzed for, but the result is rejected because of serious deficiencies. The presence or absence of the analyte cannot be verified.

Holding Times

ERA Sampling

There were 107 results for semivolatiles, toxaphene, diesel, or waste oil qualified as estimated detects and non-detects because of holding time violations. All other results met the holding time requirements.

Focused Sampling

There were 236 results for mercury, pesticides, diesel or waste oil qualified as estimated detects and non-detects because of holding time violations. The results for mercury and pesticides listed below were rejected because the holding time was greater than method and QAPP-specified holding time.

Affected Samples:

CAR_53_1A	Mercury	Holding time = 28 days	Analyzed = 50 days
CAR_53_1A	Mercury	Holding time = 28 days	Analyzed = 50 days
WG_50A	Pesticides	Holding time = 7 days	Extracted = 30 days

Calibration

ERA Sampling

The results for 2,4-dinitrophenol in the samples listed below were rejected because the initial and continuing calibration standards failed to meet the minimum relative response factor required. (These samples were from the dredge area and results were not used for the ERA.)

Affected Samples:

DC3-1	DCRD-60-02-1	DCRDA5102-1	DC16-1
DC3-2	DCRD-60-02-2	DCRDA5102-2	DC16-2
DCRD-58-01-4	DCRD-60-02-3	DCRDA6602-1	DC29-1
DCRD-58-02-1	DCRD-60-02-4	DCRDA6602-2	DCRD-58-01-1
DCRD-58-02-2	DC10-1	DC10-2	DCRD-58-01-2
DCRD-58-02-3	DCRD-60-04-1		DCRD-58-01-3
DCRD-58-02-4			

The rest of the calibration deviations consisted of the initial and continuing calibrations exceeding the acceptance criteria for response factor relative standard deviations and percent differences, respectively. These deviations accounted for "J" and "UJ" flags for approximately 3 percent of the results for semivolatiles, metals, PCB congeners, pesticides, volatiles, or general chemistry parameters.

Focused Sampling

There were 226 results qualified for calibration deviations. The deviations consisted of the initial and continuing calibrations exceeding the acceptance criteria for response factor relative standard deviations and percent differences, respectively. Interference check standards also exceeded criteria for the metals analysis only. These deviations accounted for “J” and “UJ” flags for approximately 0.7 percent of the results for semivolatiles, metals and pesticides.

Method Blanks

Method blanks were analyzed at the required frequency of at least 1 for every 20 environmental samples or one per analytical batch, whichever was more frequent. Phthalates were routinely detected in the method blanks. These compounds are ubiquitous and are considered common laboratory contaminants. Since the required detection limits for this project are very low, they were routinely exceeded by the levels found in the method blanks. Ecologically-based threshold (ET) benchmark levels were established as the acceptance criteria for method blanks for this project. The levels found in the method blanks were not above the ET levels at any time and were considered acceptable. Detected concentrations of phthalates in environmental samples less than ten times the method blank concentrations were flagged as not detected.

Surrogates

Surrogate recoveries are an indication of matrix effects and of the state of control in the laboratory at the time of sample analysis.

ERA Sampling

Approximately one-half of one percent of the results were qualified as estimated detects and non-detects because of surrogate recoveries outside the acceptance criteria. In addition, the results for all PCB congeners in samples DCRDA0308-1 and DCMN3-1, as well as all semi-volatile compounds in sample DC99-2 were rejected because of surrogate recoveries below 10 percent.

Focused Sampling

Less than 0.5 percent percent of the results were qualified as estimated detects and non-detects because of surrogate recoveries outside the acceptance criteria. These surrogate exceedances were limited to either TPH diesel or waste oil.

Quantitation and Sensitivity

A number of samples were diluted to minimize matrix interferences during organic analyses. This resulted in elevated detection limits above those required by the project. Quantitation was not part of the data evaluation, since the deliverables did not include raw data.

Matrix Spike Samples

The results of matrix spike analyses provide information about the possible presence of matrix effects. Where matrix spike recoveries and/or matrix spike relative percent differences (RPDs) exceeded acceptance criteria, detects were flagged “J” and non-detects “UJ”.

ERA Sampling

Most of the data qualifications for matrix spike deviations involved barium, silver, and zinc. Data qualification for matrix spike deviations involved less than 1 percent of the results.

Focused Sampling

Data qualification for matrix spike deviations involved less than 1 percent of the results.

Field Blanks

No field blanks affected sample results.

Field Duplicates

ERA Sampling

Where the RPD between the field duplicate results exceeded the acceptance criteria, the results were flagged as estimated. These flags accounted for only approximately 0.2 percent of the results.

Focused Sampling

There were 276 results qualified where the RPD between the field duplicate results exceeded the acceptance criteria. The results were flagged as estimated detects or non-detects.

Laboratory Control Samples

ERA Sampling

Laboratory control samples (LCSs) were analyzed at the required frequency. LCS recoveries outside the acceptance criteria were flagged “J” for detects and “UJ” for non-detects. These flags accounted for less than 1 percent of the results, and mostly involved the metals and total organic carbon (TOC).

Focused Sampling

There were 443 results for metals and organo phosphorous pesticides qualified as estimated detects and non-detects because of laboratory control sample violations. The results for the organophosphorous pesticide Naled listed below were rejected because there was no recovery of the LCS.

Affected Samples:

SW_01

SW_02

SW_03

SW_04

SW_05

Internal Standards

There were 37 results qualified for semi-volatiles because of internal standard exceedances of the acceptance criteria. The results were flagged as estimated detects or non-detects.

Chain-of-Custody

The chain-of-custody (CoC) procedures specified in the work plan were generally followed, except the sampled by block of the COC was not routinely signed, though the relinquished by block was signed.

Completeness

The completeness goal of 90 percent was met for all matrix and method combinations.

Other Information

No matrix spike analyses were performed for the general chemistry parameters with the exception of sulfate and chloride.

1.0 Introduction

This site-specific Quality Assurance Project Plan (QAPP) presents the policies, organization, functions, and specific quality assurance (QA) and quality control (QC) activities associated with analytical data generation and assessment for the Bolsa Chica Lowlands project, Orange County, California. The project involves developing and implementing a Work Plan that includes an Ecological Risk Assessment and Confirmation Sampling (ERA/CS) to properly characterize contaminated sites and establish adequate cleanup criteria within the Bolsa Chica Lowlands. Analytical data generation and assessment are designed to achieve the data quality objectives for this project.

This QAPP, along with sections of the Bay Protection and Toxic Cleanup Program QAPP (Stephenson et al., 1994) and the QAPP presented as Section 5 of the Phase II Environmental Assessment for Bolsa Chica Lowland and Pocket Area (Tetra Tech, Inc., 1996) reference documents, comprise the quality assurance plan for this effort (Reference QAPPs). Portions of the reference documents are considered part of this QAPP by reference herein, but any sections of this document that differ or enhance either of the reference documents shall supercede them.

Sampling protocols for the project and associated field activities are presented in the project Work Plan, which includes a Field Sampling Plan (FSP). Combined, the FSP and this QAPP represent the Sampling and Analysis Plan (SAP) for the Bolsa Chica Lowlands project.

2.0 Project Description

2.1 Project Description

The U.S. Fish and Wildlife Service (USFWS) and the Bolsa Chica Technical Committee are leading an evaluation of contamination in the Bolsa Chica Lowlands in Orange County, California. Preliminary investigations of about 950 acres indicated that contamination exists on portions of the property, including petroleum contamination in roads and sumps. The present project involves additional sampling to fully characterize and evaluate chemical contamination in the Bolsa Chica Lowlands.

2.2 Project Purpose and Scope

The project scope involves developing and implementing a Work Plan that includes an Ecological Risk Assessment and Confirmation Sampling (ERA/CS) to properly characterize contaminated sites and establish adequate cleanup criteria within the Lowlands (CH2M HILL, 2000).

The purpose of the ERA/CS is to establish cleanup criteria that will protect fish, wildlife, and human health by specifying appropriate media sampling and assays. The Lowlands provide nesting and feeding habitat for threatened, endangered, and sensitive resident and migratory animal species and habitat for sensitive plant species. The ERA will be developed using established, scientifically sound protocols and methodologies, such as guidance documents produced by the U.S. Environmental Protection Agency (USEPA, 1997a, 1997b) and California EPA's Department of Toxic Substances Control (California EPA, 1996a, 1996b). The ERA scope includes evaluating the risk associated with current and future oil production and urban runoff inputs.

Existing data will be incorporated into documentation of the ERA/CS, including Bennett et al. (1996), Steffek et al. (1996), Tetra Tech (1996), the Koll Real Estate Group Environmental Impact Report (EIR), documentation of cleanup efforts by AERA Energy LLC, and various reports on the Bolsa Chica Ecological Reserve.

2.3 Project Background

A preacquisition contaminants survey of the Lowlands was completed by the U.S. Department of the Interior (Bennett et al., 1996). This survey consisted of record searches and field reconnaissance of about 950 acres, including the Edwards Thumb area. The resulting report recommended sampling for chemical contamination and developing a comprehensive site assessment plan involving an ecological risk assessment.

Tetra Tech, Inc. (1996) conducted the contaminant sampling recommended in the Department of the Interior report, resulting in the December 1996 Report of the Preliminary Level II Preacquisition Environmental Contaminants Survey for the Bolsa Chica Lowlands,

Orange County, California (Steffeck et al, 1996). This report identified contaminant issues, including petroleum contamination in roads and sumps, and recommended additional sampling to fully characterize contaminated sites within the Lowlands and establish adequate cleanup criteria through an ERA. The present project is intended to implement these recommendations.

2.4 Project Objectives

The overall objectives of the project include:

- Fully characterizing contamination within the Lowlands, incorporating both existing and newly generated record search, field reconnaissance, and sampling and analysis data
- Conducting an ERA and identifying risk to ecological receptors
- Establishing adequate contamination cleanup criteria for site restoration that consider the ecological and human health risks associated with the contamination and cleanup operations
- Considering the risk associated with current and future oil production and urban runoff inputs

2.5 QAPP Format and Guidance

This QAPP was produced following the format provided in the Bay Protection and Toxic Cleanup Program QAPP (Stephenson et al., 1994) for sediments, pore water, and biota. Soil and surface water sampling elements of this project were designed following the format provided in the QAPP presented as Section 5 of the Phase II Environmental Assessment for Bolsa Chica Lowland and Pocket Area (Tetra Tech, Inc., 1996).

The QA/QC procedures described herein are consistent with standard EPA guidance documents, including those provided by California EPA (1996a, 1996b). Data Quality Objectives and Quality Assurance/Quality Control (DQO/QA) for sampling pathogens in both the Lowlands and Garden Grove Wintersburg Flood Control Channel are subject to review by the California State Water Resources Control Board (State Board). The State Board's review will be guided by its Water Quality Control Plan for Ocean Waters of California and the California Regional Water Quality Control Board's (1995) Water Quality Control Plan for the Santa Ana River Basin.

3.0 Project Organization and Responsibilities

The Work Plan describes the project organization and responsibilities. It includes a project organization chart identifying the CH2M HILL project manager, task managers, and other individuals responsible for performing the work. It includes descriptions of the qualifications of key participants and their roles and responsibilities. Contractors and subcontractors are identified along with the general scope of their anticipated project activities.

3.1 Laboratory Services

Several laboratories that have a record of successfully meeting DQOs for projects that have similar requirements to this project have been retained by CH2M HILL. Combined, they will provide the full range of required analytical and bioassay services needed for the project. Having multiple laboratories on the team ensures both that the entire spectrum of required services are available and that there is sufficient overall laboratory capacity to keep the work on schedule. All laboratories will maintain certification under the California Department of Health Services Environmental Laboratory Accreditation Program.

Analytical services for this project will be provided by Columbia Analytical Services (CAS) located in Redding, California and by Kinnetic and its associated laboratory, ToxScan, located in Watsonville, CA.

CAS has more than 20 years of experience providing analytical services for projects subject to RCRA, CERCLA, and other regulatory programs. Kinnetic and ToxScan specialize in sediment toxicity testing and analysis of the exposure media (i.e., sediment or pore water). These laboratories have more than 20 years of experience in these areas of expertise.

Rapid turn around samples for screening purposes will be performed by local laboratories (primarily those of AERA Energy) as needed throughout the program.

4.0 Quality Program and Data Quality Objectives

4.1 Data Categories

Two categories of data will be obtained. One category includes data that will characterize the types and concentrations of contaminants in the Lowlands and will be obtained by sampling the appropriate soil, surface water, and groundwater media. The other category of data includes those that will indicate the existing or potential effects of these contaminants on ecological receptors. These data will be obtained from biological tissue samples that will be analyzed for contaminant concentrations and from bioassays that assess toxicity.

The type of data to be obtained from the sampling media and ecological receptors will include:

- Soils and sediments (including pore water)
 - Analytical chemistry and physical characterization
 - Toxicity and bioaccumulation
- Water column/surface water
 - Analytical chemistry
 - Toxicity and bioaccumulation
 - Microbial/pathogenic
- Groundwater
 - Analytical chemistry
- Biota
 - Analytical chemistry
 - In-situ biomarker/bioaccumulation

4.2 Precision, Accuracy, Representativeness, Completeness, and Comparability

Data collection and analyses for this project will be consistent with assessment and measurement endpoints of the ERA. Data quality objectives are designed to ensure consistency in data reporting and comparability among sampling sites, so that any contamination determined can be accurately described and its origins identified (e.g., oil

production operations, agricultural runoff, urban runoff). Detection limits will be established that are low enough to evaluate effects on the biota considered in the ERA, particularly with regard to toxicity benchmarks.

This QAPP has been designed to maximize the probability that environmental data collected during this program will meet or exceed the data quality objectives. It provides a systematic approach to data acquisition and management to accomplish the following purposes:

- Ensure that data collection and measurement procedures are standardized among all participants
- Monitor the performance of the various measurement systems being used in the program to maintain statistical control and provide rapid feedback, so that corrective measures, if needed, can be taken before data quality is compromised
- Periodically assess the performance of these measurement systems and their components
- Verify that reported data are sufficiently complete, comparable, representative, unbiased, and precise, so that they are suitable for their intended use

The data quality criteria for this project consist of qualitative and quantitative indicators, including precision, accuracy, representativeness, completeness, and comparability. Accuracy, precision, and completeness requirements for various indicators are shown in Table 4-2 of Stephenson et al. (1994).

4.2.1 Precision

Precision is a measure of reproducibility of analyses under similar conditions. Precision can be defined as the degree of mutual agreement among individual measurements and represents an estimate of random error. Precision will be evaluated based on laboratory or field duplicates or duplicate matrix spikes. When using matrix spikes, precision will be calculated as the relative percent difference (RPD) between the matrix spike (MS) and the matrix spike duplicate (MSD) recoveries. When using laboratory or field duplicates, it will be calculated as the RPD between the duplicate results when the sample concentration is at least five times the reporting limit, or as the difference between the duplicate results when the sample concentration is less than five times the reporting limit. Field replicates will comprise 5 percent of the sampling effort. MS/MSDs will be field-designated at a 5 percent frequency.

4.2.2 Accuracy

Accuracy is the degree of agreement between a measured value and the "true" or expected value. As such, it represents an estimate of total error from a single measurement, including both systematic error, or "bias," and random error that may reflect variability due to imprecision. Accuracy is expressed in terms of percent recoveries determined from results of MS/MSD and Laboratory Control Sample (LCS) analyses.

Accuracy requirements might not be definable for all parameters. For example, accuracy measurements for toxicity testing are not possible, because there are no "true" values for these measurement parameters (Stephenson et al., 1994).

4.2.3 Representativeness

Representativeness is the degree to which sample data accurately expresses the characteristics of a population of samples, parameter variations at a sampling point, or an environmental condition. It is a qualitative parameter that is achieved through proper sampling program design using appropriate sampling strategies and techniques. Factors that can affect representativeness include site homogeneity, sample homogeneity at a single point, and available information around which the sampling program is designed (Tetra Tech, 1996, CH2M HILL, 2000). The sampling program has been designed to maximize representativeness through the location selection process.

4.2.4 Completeness

Completeness can be defined both qualitatively and quantitatively. Qualitative completeness is determined as a function of all factors that contribute to sampling. Quantitative completeness is calculated as the percentage of measurements that are judged to be valid compared to the total number of measurements planned. Effectively, it measures the amount of data available for valid measurement compared to the amount that is lost or destroyed. For this investigation, a completeness factor of 90 percent for all matrices is established, and is strictly defined as the ratio of the number of usable data points (not flagged "R" - see Section 8) over the total possible number of data points, by method/matrix.

4.2.5 Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another. Confidence is achieved by maintaining standard techniques and procedures for collecting and analyzing representative samples and reporting the analytical results in standard units. Standard EPA methods are used for the analytical chemistry and accepted protocols for bioassay and toxicity testing are used throughout this program.

4.3 Method Detection Limits, Reporting Limits, and Instrument Calibration Requirements

4.3.1 Method Detection Limits

The Code of Federal Regulations (40 CFR 136) defines Method Detection Limits (MDLs) as follows: "The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte."

Each participating analytical laboratory will calculate and report an MDL for each analyte of interest in each matrix (i.e., surface water, sediment, tissue, etc.) prior to analyzing field samples. Each laboratory will calculate MDLs statistically, based on instrument performance, at least once annually for each analytical method employed, as required under 40 CFR 136.

4.3.2 Reporting Limits

Reporting limits are driven by the data quality objectives as defined in the Work Plan and must be greater than 2X the calculated MDL. Reporting limits used by the laboratory cannot be greater than the required detection limits (RDL) listed in Table 4-6 of the Work Plan (CH2M HILL, 2000) for organochlorine herbicides and organophosphorus pesticides and in Table 4-1 in the Work Plan (CH2M HILL, 2000) for the rest of the analytes for this project.

Reporting limits, as well as sample results must be reported on a dry-weight basis for sediment samples, and on a wet-weight basis for tissue samples.

4.3.3 Instrument Calibration

Laboratory instruments will be appropriately calibrated by qualified personnel prior to sample analysis. Calibration will be verified at specified intervals throughout the analysis sequence. The frequency and acceptance criteria for calibration are specified for each analytical method. When multi-point calibration is specified, the concentrations of the calibration standards should bracket those expected in the samples. Samples must be diluted, if necessary, to bring analyte responses within the calibration range. Tables 7-3 through 7-11 list the specific requirements for each method. Only those data that result from quantitation within the demonstrated working calibration range (see Section 4.3.2 above) may be reported by the laboratory. Quantitation based on extrapolation is not acceptable.

4.4 Elements of Quality Control

Internal QC checks are used to provide indications of the state of control that prevailed at the time of sample analysis. QC checks that involve field samples, such as matrix and surrogate spikes and duplicates, provide an indication of the presence of matrix effects. QC samples include method blanks, laboratory control samples, surrogate spikes, and matrix spikes and matrix spike duplicates.

4.4.1 Method Blank

Laboratory pure water (also called laboratory reagent blank) serves as a method blank to monitor each analytical batch for interference and for contamination from glassware, reagents, and other potential contaminants generated within the laboratory. The method blank is processed through the entire sample preparation and analytical procedures along with each sample batch. One method blank per sample batch is analyzed. If a target analyte is found at a concentration that exceeds the acceptance limit, corrective action is triggered to identify and eliminate contamination sources. See Tables 7-3 through 7-11 for details by method.

4.4.2 Laboratory Control Sample

Laboratory control samples are used as a reference to assess accuracy of an analysis. The laboratory control samples (LCS) for this project will consist of reagent water or cleaned sand spiked with known amounts of analytes that come from a source different than that used for calibration standards. All target analytes are spiked into the LCS for inorganic analyses. In the case of organic analyses, selected target analytes are spiked into the LCS. If

LCS results exceed the specified control limits, corrective procedures must be implemented. Quality control limits for LCSs are listed in Table 7-2 of this document.

4.4.3 Surrogates

Surrogates are analytes that behave similarly as the analytes of interest, but are not expected to occur naturally in the samples. They are spiked into the samples prior to sample preparation. Recoveries of surrogates can be used as an indicator of the accuracy of the measurement of target analytes. Surrogate recoveries must be reported for each sample preparation/analytical method combination. The acceptance limits for surrogate recoveries are listed in Table 7-2.

4.4.4 Matrix Spike/Matrix Spike Duplicate

A sample matrix fortified with known quantities of specific compounds is called a matrix spike (MS). It is subjected to the same preparation and analytical procedures as the native sample. All target analytes are spiked into the sample for inorganic analyses that are amenable to spiking. When analyzing for Total Organic Carbon in sediment/soil, laboratory duplicate analyses will be performed instead of matrix spikes. In the case of organic analyses, selected target analytes are spiked into the sample. Matrix spike recoveries are used to evaluate the effect of the sample matrix on the recovery of the analytes of interest. A matrix spike duplicate (MSD) is a second laboratory fortified sample matrix. The relative percent difference (RPD) between the recoveries from the duplicate matrix spikes is used as a measure of the precision of sample results. Table 7-2 lists the acceptance limits for MS/MSDs for this project.

4.4.5 Other Method - Specific Requirements

Other quality control parameters are described in each method. The frequency and acceptance criteria are listed in Tables 7-2 through 7-11.

4.4.8 Equipment Blank

Rinsate blanks are obtained by rinsing decontaminated sampling equipment with ASTM Type II water. The rinse water is collected in sample bottles, preserved, and handled the same as the samples. The frequency of sample collection is described by matrix in the Work Plan.

4.4.9 Trip Blank

Trip blanks are analyzed for VOCs only and consist of sample bottles filled in the laboratory with ASTM Type II water. The sample bottles are then sent to the sampling location(s) with sampling kits. The specified number of trip blanks are returned from the sampling location with every shipment of groundwater samples and analyzed for VOCs.

4.4.10 Field Duplicates/Replicates

Field duplicates provide yet another means of maintaining quality control by measuring the precision of the sampling process. The laboratory will not be given the identity of the duplicates, but the QA reviewer will receive source information to aid in data review and validation. Acceptance criteria are in Section 8 and frequencies are discussed by matrix in

the Work Plan. At a minimum, water and soil/sediment duplicate samples are collected at a 5 percent frequency.

4.4.11 Tissue Standard Reference Materials

All biota tests will include control tests using reference materials to ascertain the performance of the total analytical system. Appropriate reference materials are addressed for each method in Section 7.

4.5 Quality Control Procedures

4.5.1 Sample Custody

Table 5-2 of the Tetra Tech QAPP specifies the holding times and preservation conditions that will apply to this project.

Each laboratory will designate a sample custodian who will log in samples using a standardized Sample Receipt Form. The custody seal will be inspected to verify that it is intact, and the sample custodian will then check the condition of the samples and verify custody records. The presence or absence of ice in the sample cooler will be noted, and the cooler temperature will be recorded. Any breakage, leakage, or other damage will be noted and recorded. The sample custodian will record all tracking information and pass it on to the data librarian and the laboratory project manager. All of this information will appear on the Sample Receipt Form. If discrepancies are noted between the chain-of-custody report and actual contents of the container, these discrepancies will immediately be reported to the CH2M HILL project manager. Along with sample receipt documentation, the following information will be documented on Sample Receipt Forms by the sample custodian:

- Date samples received
- CH2M HILL sample identification number
- Laboratory sample identification number
- Analytical tests requested for the sample batch
- Sample matrix
- Number of samples in the batch
- Container description and location in the laboratory

After being logged in, the samples will be refrigerated as appropriate. The laboratory must have formally documented procedures for sample holding and storage, and laboratory personnel will know the required sample holding times and preservation conditions. If samples are not extracted or analyzed within the required holding time for the appropriate method, the CH2M HILL project manager will be notified immediately for guidance on corrective action. All corrective actions must be fully documented. After confirmation by the CH2M HILL project manager, samples with expired holding times will be discarded.

4.5.2 Deliverables

Laboratories that will perform analyses for the Lowlands must have established procedures to conduct data reduction, review, and reporting. The specific procedures and assigned personnel vary among laboratories; however, equivalent data reduction and review

protocols are required to ensure the overall objectives of analysis and reporting according to method and project specifications are achieved. Laboratory-specific procedures are evaluated during technical systems audits to ensure that the process steps discussed in this section are properly performed.

The primary analysts will be responsible for review of their work as their work is being performed. During this process, a case narrative or quality control exception report will be generated documenting nonconformance issues and resolution. A designated peer reviewer, a qualified staff member who is not the primary analyst(s), will perform an independent review to determine that the project specifications have been met. The Laboratory Manager or designee will be responsible for final approval of the laboratory analytical report prior to sending the report to the project staff. All raw data will be archived in confidential laboratory files.

Most laboratories use a Laboratory Information Management System (LIMS) to store, transfer, and report analytical data. The LIMS files must also undergo a QC check to verify that the results are complete and correct. The laboratory is responsible for generating hard copies (i.e., final analytical report) and electronic files of the analytical results in standard formats needed by the project staff. The specific information and electronic file formats are established and tested prior to analysis of any samples to ensure that the formats will be compatible with the project database, and that all required information is reported.

The hard-copy and electronic laboratory reports for all samples and analyses will contain the information necessary to perform data evaluation (described in Section 8). The following information is typically included for each preparation batch (when applicable) and each analytical batch:

- Field identification number
- Date received
- Date prepared
- Date analyzed
- Method
- Result for each analyte (including surrogates)
- Sample specific detection limit
- Surrogate spike recoveries
- Units
- Dilution factor
- Laboratory qualifier flags
- Narrative
- Matrix spike and laboratory control spike concentrations

- Matrix spike and laboratory control spike results
- Matrix spike and laboratory control spike recoveries and relative percent differences (RPDs)
- Method blank results
- Any other QC sample results
- Initial and continuing calibration verification results (required only for hard copy)
- Initial and continuing calibration verification recoveries (required only for hard copy)
- Analytical batch number
- Preparation batch number

Complete documentation of sample preparation and analysis and associated QC information will be maintained by the laboratory for all project samples in a manner that allows easy retrieval in the event that additional validation or information is required.

The electronic analytical data (if needed) from the laboratory are submitted with hard-copy reports and uploaded to the project database by using a set of programs to read, check, and match the analytical results to the field data in the database. The electronic results are reviewed by project staff to ensure accurate reporting and adherence to project specifications. Ten percent of all electronic results will be reviewed for correct sample identification, dates, sample specific detection limits, flags, and agreement between the hard copy and electronic data. If systematic errors or frequent occurrence of random errors are observed, a successively higher percentage of reports will be reviewed. After the analytical reports are used to verify the electronic transfer process, they are permanently stored in project files. See Section 9.

Data flow from the laboratory and field to the project staff and data users follows established procedures to ensure that data are properly tracked, reviewed, and validated for use.

4.5.3 Medium Level Extractions/Waste Dilutions

In the case where target concentrations and/or the nature of the sample matrix preclude low level analyses, a medium level protocol or waste dilution should be used in order to preserve the ability to monitor analytical efficiency by evaluation of measured surrogate recoveries. Medium level protocol is described in SW-846 method SW3550B. Waste dilution procedures are described in SW3580A and SW3585.

4.5.4 Additional Cleanup Procedures to Minimize the Effect of Petroleum Hydrocarbons on Recoveries and Reporting Limits

Hydrocarbons are expected to be present and will interfere with analyte integration/chromatography, resulting in dilutions that raise the reporting limits if cleanups are not performed. In order to maintain the lowest possible reporting limits, appropriate cleanup procedures must be employed. Methods for sample cleanup include but are not limited to gel permeation chromatography (GPC), silica gel, alumina, florisil, mercury (sulfur

removal), sulfuric acid and acid/base partitioning. GPC will be performed when necessary to eliminate or minimize matrix interference. When analyzing for Pesticides and PCBs, half of the sample extract must be set aside for PCB analysis. This half of the extract must be subjected to sulfuric acid cleanup prior to analysis for PCBs. Method blanks, MS/MSDs, and laboratory control samples must be subjected to the same cleanup procedures performed on the samples to monitor the efficiencies of these procedures.

4.5.5 Sample Dilutions

Dilution of the samples results in elevated reporting limits and ultimately affect the usability of the data as it pertains to decision making processes related to potential actions at the sampling site. It is important to minimize dilutions and maintain the lowest possible reporting limits. When dilutions are necessary due to high concentrations of certain target analytes, lesser dilutions should also be reported in order to fully characterize the sample for each of the low-concentration analytes. The level of the lesser dilution is directly related to the analytical system specified by the method and is defined as the dilution that provides the lowest possible reporting limits without having a lasting deleterious effect on the analytical instrumentation.

4.5.6 Brine Sample Chelation

Samples that are known to be of high salt content will undergo a chelation process to remove the salts prior to the method-specified sample preparation.

5.0 Sampling Procedures

5.1 Field Sampling

5.1.1 Sample Container

USEPA-recommended containers will be used for field sampling, and sampling procedures will adhere to USEPA-recommended preservation requirements for each parameter of concern. Use of proper containers and preservation methods will retain sample integrity. Containers and preservatives will be provided by laboratory personnel. The USEPA guidelines for sample containers and preservatives are summarized in Table 5-1 of Tetra Tech, Inc. (1996).

5.1.2 Sample Volumes, Container Types, and Preservation Requirements

Holding time compliance and proper sample preservation begin during field sampling. Temperature control and pH adjustment are the most common preservation techniques. Field personnel who will perform on this project will be thoroughly trained in proper use of sample collection gear and acceptable sampling procedures. Required holding times for various parameters are summarized in Table 5-2 of Tetra Tech (1996). Special sample volumes and container requirements for biota are discussed in the Work Plan (CH2M HILL, 2000).

5.2 Sample Handling and Custody

Field sampling personnel will maintain a waterproof field logbook that will be completed with each sampling event. The field logbook will contain the following information:

- Date and time of commencement of sampling
- Name of sampling personnel
- Location of sampling station (location coordinates)
- Station description, including designation number
- Type of grab sampling and equipment used
- Field observations (weather, soil, water conditions; texture; odors; benthos; sheens)
- Station depth
- Number of grabs made and amount of sample taken
- Type(s) of analyses to be performed

- Salinity and temperature of water samples

As required by the project manager, additional information will be recorded in the field logbook. This information might include dissolved oxygen, stratification profiles of salinity and temperature, pH, secchi depth, redox potential discontinuity depth, etc.

Samples will be transported to the appropriate laboratory daily with proper chain-of-custody (COC) records for each sample. Each person who releases a sample will sign and date the COC form and require the receiver to sign and date the form. Each will keep a copy of the signed form. Each form will consist of a record of all samples taken from each station. Each form will include the sample identification number, FWS station number and name, leg number, and date collected.

Standardized "Authorization/Instructions to Process Samples" forms completed by the FWS or authorized designee will accompany all samples transported to analytical laboratories. These forms are signed by the laboratory-designated sample custodian who logs in the samples. The forms will contain all information required by the laboratory to process the samples, including:

- Type and number of tests to run
- Number of laboratory replicates
- Dilutions
- Exact eligible cost
- Deliverable products
- Expected date of deliverables submittal by the laboratory

Field sampling personnel will attach labels to the outside and/or inside of the sample container. Jars will not contain hand-written labels. All jars will be pre-labeled by sampling personnel before samples are aliquoted. Labels will include the following information:

- Sample number
- Collection station number
- Station name
- Leg sampled
- Date samples collected

Replicate quality control samples for sediment chemistry will be taken at 5 percent of the sites sampled, as noted above.

Six-liter sample containers will be packed with sufficient ice to keep them cool for at least 48 hours. Each container will be double-bagged in pre-cleaned plastic bags closed with cable ties to keep all samples within the container isolated from one another. Ice chests must be driven or flown to the laboratory within 24 hours of collection.

Sampling procedures for collecting underwater sediment samples using a grab sampler, sample acceptability criteria, cleaning procedures, homogenization and aliquoting of samples, extracting pore water, collecting benthic and fish samples (if appropriate), and collecting and storing samples for acid volatile sulfide (AVS) analysis are presented in Section 3 of Stephenson et al. (1994).

6.0 Screening Analytical Methods

6.1 Field Instrument Calibration Procedures

Several types of real-time instruments can be used to monitor and evaluate the physical parameters of water and soil. This screening level data can be used to monitor worker health and safety and to assist sample collection. Field instruments that may be used for investigations include:

- pH meter
- Conductivity meter
- Thermometer
- Turbidity meter
- Photoionization detectors (PIDs), such as HNU®, organic vapor monitor (OVM), and Micro TIP®
- Flame ionization detectors (FIDs) or organic vapor analyzer (OVA)
- Radioactivity meter
- Canister sample flow controllers
- Aquifer and air permeability testing flowmeters and vacuum gauges

If different or additional field instruments are needed for a specific effort, these will be specified in the Work Plan.

To ensure that the instruments are operating properly and are producing accurate and reliable data, routine calibration will be performed prior to and during use. Factory calibrations will be performed at a frequency recommended by the manufacturer. Field calibrations will be performed at least once per day, prior to instrument use. If field calibration reveals that the instrument is outside established accuracy limits, the instrument will be serviced in the field. If necessary, the instrument will be returned to the manufacturer for immediate repair and servicing. A backup instrument will be available for each of the critical real-time instruments used in the field.

6.1.1 Water Sampling Instrument Calibration

Field pH meters, conductivity meters, turbidity meters, and thermometers may be used to measure water parameters when collecting groundwater and surface water samples. The meters will be calibrated prior to purging well water or collecting surface water. It is suggested that the pH and conductivity meters be calibrated with at least two standard

calibration solutions that bracket the expected range of measurements. The turbidity meters should be calibrated with a known standard solution. Standard solutions may be supplied by the instrument manufacturer or obtained commercially. Thermometers should be calibrated at the beginning and end of each field event using a National Institute of Standards and Technology (NIST) reference thermometer.

6.1.2 Real-Time Organic Vapor Monitoring Instrument Calibration

Real-time OVMs are used to monitor total airborne organic vapors during field operations; measurements are used to evaluate worker health and safety. Personal protective equipment (PPE) requirements and site control decisions will be determined using the results of real-time measurements. Real-time instruments also provide screening level data for volatile organic compound (VOC) concentrations in drill cuttings, soil boring samples, and groundwater wells.

Several types of OVMs are available. Generally, these instruments utilize one of two primary detection methods for quantifying total airborne VOCs: a FID or a PID. Suggested calibration frequencies for each commonly used instrument are presented in the following subsections. Due to the rigors of field use, backup instruments should always be available.

Flame Ionization Detector (FID)

FIDs measure total concentrations of hydrocarbon vapors. The instrument response for each specific compound is proportional to its response factor relative to methane. The instruments should be calibrated using methane in air.

The suggested calibration frequencies for field OVAs are:

- Factory calibration and service once per year
- Five-point calibration using four methane-in-air standards and ultra-high purity (UHP) air performed once each quarter
- Three-point calibration using two methane-in-air standards and UHP air prior to daily use
- Single-point calibration check using a representative methane-in-air standard after each 4 hours of operation and at the end of each working day

Photoionization Detector (PID)

PIDs measure total organic vapors and are highly sensitive to aromatic compounds, moderately sensitive to unsaturated chlorinated compounds, and less sensitive to aliphatic hydrocarbons. The instrument responds to organic compounds with ionization potentials less than the rated electron voltage (eV) of the ultraviolet (UV) bulb in the unit. Due to its longevity and range of detectable contaminants, the most frequently used UV bulb is a 10.2 eV. Other bulbs are available from the manufacturer (e.g., 9.6 eV, 11.7 eV, etc.). Field personnel will know which bulb is installed in the unit ensuring that the instrument is capable of detecting the particular contaminant of interest.

Several manufacturers produce instruments with PIDs for field monitoring of airborne VOCs. The manufacturer's calibration requirements should be followed. Suggested guidance for PID calibration includes:

- Factory service and calibration once per year
- The HNU Systems PI-101 requires a three-point calibration on a quarterly basis using UHP air and two representative concentrations of isobutylene-in-air standards
- For any PID instrument, a two-point calibration prior to daily use (UHP air and a representative concentration of isobutylene in air standard)
- Single-point calibrations at the end of each day of use

6.3 Flowmeter Calibration

Flowmeters that are typically used during air permeability testing include pitot tubes with inclined mercury manometers or Magnehelics®, rotameters, and vortex meters. Standard water meters may be used during aquifer testing; graduated buckets may also be used.

Although calibration is not required for the pitot tubes, they should be inspected to ensure the holes are not plugged and cleaned, if required. Similarly, the mercury manometers and Magnehelics® do not require adjustment; however, both require zeroing versus atmospheric air pressure before readings are taken. The accuracy of the Magnehelic® will be verified in the field by attaching it to a mercury manometer and to a common vacuum/pressure source (such as a canister) and comparing the reading. If required, Magnehelics® will be returned to the factory for calibration.

The flow rate of the water meters used for aquifer testing will be checked against a graduated bucket to determine if the meter is functioning correctly.

7.0 Analytical Requirements

7.1 Bioassays

7.1.1 Bioassay Test Procedures

Toxicity will be assessed by bioassays that will help establish toxicity/concentration relationships for the main classes of COPECs at the Lowlands. The Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual) developed by the USEPA and the US Army Corps of Engineers (1991) provides specific guidelines for the various bioassay and bioaccumulation tests required. All species used for this project comply with the Testing Manual recommendations. For suspended particulate phase bioassays the mysid *Mysidopsis bahia*, a marine teleost fish (the inland silversides, *Menidia beryllina*), and the larvae of the mussel *Mytilus edulis* are used. For the solid phase bioassays the polychaete *Nephtys caecoides*, the mysid *Mysidopsis bahia*, and the amphipod *Rhepoxynius abronius* are used. Methods and procedures are summarized below.

7.1.1.1 Suspended Particulate Phase Bioassays

Suspended particulate phase elutriates are prepared by using laboratory seawater and test sediments. Three concentrations (100%, 50%, and 10%) of suspended particulate phase are tested. Bioassays using control seawater (from the Santa Cruz facility) are used to provide comparative data for statistical analyses and to provide quality assurance data for assessing test validity. The lower concentrations are evaluated only if the 100% concentrations produced >50% mortality or inhibition of development. If exposure to 100% elutriate produces <50% effect, calculation of a meaningful LC or EC value is not possible. The data from the 50% and 10% concentrations are only used to calculate LC50 or EC50 values for use in initial mixing calculations.

Test containers are randomly positioned within the temperature-controlled water baths, and other conditions in the laboratories are designed for uniform exposure to the controlled laboratory environment. The control exposure, performed for quality assurance purposes, used seawater from the laboratory system. Five replicate containers are used for each test exposure. Temperature, dissolved oxygen, and pH are monitored daily in each test concentration and control. Salinity is monitored in each test concentration and in controls at the beginning and end of the test.

The sediment samples are placed in cleaned containers with laboratory seawater for elutriate preparation. The sediment to water ratio is 1:4 as specified in the Implementation Manual. The sediment and seawater are mixed for 30 minutes. After settling, the elutriates are siphoned off and used as suspended particulate phase media. The elutriates are aerated to near saturation before test organisms are added.

7.1.1.2 Bivalve Larvae (*Mytilus edulis*)

Bivalve larvae bioassays are carried out according to protocols given in ASTM (1989). Adult *Mytilus edulis* are purchased from Carlsbad Aquafarm in Carlsbad. Adult mussels are induced to spawn by high-temperature stimulation. Eggs and sperm are collected in separate basins filled with aerated seawater at spawning temperature. Egg density is determined by microscopically counting at least six, 1-ml aliquots taken from the well-mixed egg basin. Fertilization is accomplished by addition of an appropriate amount of sperm suspension, and confirmed by microscopic examination.

Larvae are tested in 250-ml polyethylene beakers containing approximately 150 ml of appropriate test solution. After fertilization is confirmed, an aliquot containing approximately 6,000 fertilized eggs is pipetted into each test beaker. Five extra beakers are prepared in addition to those required for test and control replicates. These “extra” test containers are not incubated for 48 hours, but rather they are evaluated immediately after inoculation to provide the “initial recovery” data used to establish the mean number of embryos added to each experimental beaker.

At the end of the 48-hour exposure period the contents of each dish are poured through a 45mm nytex screen. Surviving larvae are retained on the screen. The test beaker is rinsed several times with seawater and each successive rinse is poured through the screen to ensure complete transfer of larvae. Larvae are quantitatively transferred from the screen into a graduated cylinder. Contents of the cylinder are mixed by inversion to ensure uniform distribution of larvae, and a 1-ml aliquot is transferred to a Sedgwick-Rafter counting slide containing a few drops of formalin for microscopic evaluation. Larvae are scored for evidence of internal tissue inside a complete larval shell. Larvae that had a complete larval shell containing tissue are counted as normal, whereas empty shells and larvae with incomplete shells are scored as abnormal. Data are reported as percent of initial embryos that survived and as percent of survivors that showed normal development, as calculated below.

The raw data generated from these bioassays include the following:

- Counts of embryos added to five replicate test containers that had not been incubated for 48 hours (=initial recovery)
- Counts of normal and abnormal embryos from each test container that was incubated for 48 hours

$$\% \text{ Survival} = 100 - \left(\frac{\text{mean \% control survival} - \% \text{ sample survival}}{\text{mean \% control survival} * 100} \right)$$

where N = the mean initial number of embryos added (from the initial recovery data).

For each test chamber other than controls, percent of survival data are adjusted to correct for mortality observed in the control exposures by use of Abbott’s correction, which serves to normalize mortality and normality to 100% in controls (see Finney, 1971):

$$\text{Corrected Sample \% Survival} = 100 - \left(\frac{\text{mean \% control survival} - \% \text{ sample survival}}{\text{mean \% control survival} * 100} \right)$$

Percent normal development data are similarly adjusted.

For the bioassay to be considered a valid test, an average of at least 70% of the exposed embryos must survive in the controls; abnormal embryos are counted as mortalities. Also, an average of at least 90% of the exposed embryos must develop normally in the controls.

The 100% elutriate concentrations are evaluated initially. If mean percent of survival and/or % normal values are $\geq 50\%$, no further evaluations are performed. If survival and/or normal development values are $< 50\%$, the 10% and 50% elutriate exposures are evaluated and EC50 and/or LC50 values are calculated using the Trimmed Spearman-Kärber method. For LC50 calculations, abnormal larvae and calculated mortalities are combined; whereas for EC50 calculations, separate abnormality counts are used.

A reference toxicant bioassay is performed for quality assurance purposes, to verify the health and sensitivity of the test organism population. The reference toxicant used is cupric sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) dissolved in laboratory seawater.

Testing Manual guidelines for interpretation of suspended particulate phase bioassays require that, for any sample producing toxicity sufficient to generate an LC50, initial mixing calculations be performed to determine the concentration of suspended particulate material remaining at the disposal site within four hours after dumping (Csp). If the Csp does not exceed 1% of the LC50, the sediment is judged to comply with water column toxicity criteria.

7.1.1.3 Teleost Fish (*Menidia beryllina*)

Inland silversides (*Menidia beryllina*), 9-14 days old are cultured by and purchased from Aquatic Indicators, St. Augustine, FL. The fish are allowed to acclimate to laboratory conditions prior to testing. They are fed *Artemia* nauplii prior to test initiation and at the midpoint (48 hours) of the test.

The fish are tested in 1-liter glass jars containing 750 ml of test solution. To initiate the testing, the *Menidia* are sorted into groups of 10 in beakers containing 15 ml of seawater. The *Menidia* are transferred to the test containers by submerging the containers and slowly tipping the fish into the test medium. The 15 ml of seawater added with the fish is equivalent to 2% of the volume of test solution. This small dilution, although undesirable, is necessary to minimize handling stress to the test organisms. During the bioassays, the number of survivors of the original 10 fish per container is recorded as experimental data at 24, 48, 72, and 96 hours after test initiation. At each of these checkpoints, dead fish (i.e., those nonresponsive to mechanical stimulus) are removed from the test containers.

Monthly reference toxicant testing is performed by the organism supplier on the *Menidia* cultures, using copper sulfate. Reference toxicant data are provided by the supplier for quality assurance purposes to verify the health and sensitivity of the test organism population.

7.1.1.4 Mysid (*Mysidopsis bahia*)

Juvenile mysids (*Mysidopsis bahia*), < 5 days old, are cultured and purchased from Aquatic indicators, St. Augustine, Florida. Throughout testing, the mysids are fed about 100 brine shrimp (*Artemia salina*) nauplii per mysid twice per day to prevent mortality from starvation and cannibalism.

Mysids are tested in one-liter glass jars containing 500 ml of test solution. To initiate testing, mysids are sorted into groups of 10 in beakers containing 10 ml of seawater. The mysids are transferred to the test containers by submerging the containers and slowly tipping the animals into the test medium. The 10 ml of seawater added with the mysids is equivalent to 2% of the volume of test solution. This small dilution, although undesirable, is necessary to minimize handling stress to the test organisms. During the bioassays, the number of survivors of the original 10 animals per container are recorded as experimental data at 24, 48, 72, and 96 hours after test initiation. At each of these checkpoints, dead animals (i.e., those nonresponsive to mechanical stimulus) are removed from the test containers.

Monthly reference toxicant testing is performed by the organism supplier on the Mysid cultures, using sodium dodecyl sulfate (SDS). Reference toxicant data is provided by the supplier for quality assurance purposes to verify the health and sensitivity of the test organism population.

7.1.1.5 Initial Mixing Calculations

In cases where an EC50 or LC50 is obtained, calculations of initial mixing are made using standardized formulae developed by the USACE and USEPA (EPA/USACE 1991).

7.1.1.6 Solid Phase Static Bioassays

Solid Phase materials from the sites are bioassayed simultaneously with control and reference sediments. Control sediments, which provide data for quality assurance assessment purposes, are collected from the same area in Whidbey Island, WA where amphipods are collected. Reference sediments, which provide data for statistical comparisons, are collected from the EPA-designated reference site near the LA2 and LA3 disposal areas. All sediments are dry-sieved through a 1.0 mm screen prior to testing to remove organisms which might prey on or be confused with the test species.

7.1.1.7 Amphipod (*Rhepoxynius abronius*)

Adult *Rhepoxynius abronius* are collected from Whidbey Island, Washington by John Brezina & Associates, and bioassay-tested following procedures outlined by ASTM (1990) for amphipods. Seven replicates of each station and reference treatment are randomly assigned to test jars. A 2-cm deep layer of appropriate sediment is added to each jar on the day prior to test initiation. On the following day, each test jar is provided with aeration via Pasteur pipette and the test is started by randomly assigning 20 amphipods to each of six of the seven replicate jars. The seventh replicate, without amphipods, is used to determine initial sediment interstitial water pH, salinity, and total ammonia at test initiation. The test is continued for 10 days under static conditions, with constant illumination and aeration. Daily observations are made of each container, and the number of animals that appeared on the sediment surface is noted. At this time, environmental test conditions (temperature, pH

and dissolved oxygen) are measured in each test container. Salinity is measured at test initiation and termination.

At the end of the ten day exposure period, the contents of each jar are poured through a 0.5 mm sieve and the number of surviving amphipods counted. Survivors from each replicate are transferred into bowls containing control sediment and monitored for their ability to rebury within one hour. Test data for each replicate therefore include number of survivors and number of survivors able to rebury.

Salinity, pH, and total ammonia measurements are made on sediment interstitial water (pore water) as received, and as necessary to ensure ammonia concentrations are held below threshold levels for *Rhepoxynius* (EPA /USACE, 1993). Initial and final pore-water ammonia measurements are taken from one replicate of each test sediment at test initiation and at test termination. Pore waters are extracted by centrifugation. Interstitial water salinity is measured using a salinometer-calibrated refractometer. Interstitial water ammonia concentrations are measured with an ammonia probe calibrated with three concentration standards.

A reference toxicant bioassay is performed for quality assurance purposes, to verify the health and sensitivity of the test organism population. The test is a 96-hour, sediment-free static exposure; the reference toxicant used is cadmium chloride (CdCl_2) dissolved in laboratory seawater.

7.1.1.8 Solid Phase Static Renewal Bioassays

Solid Phase materials from the site are bioassayed simultaneously with reference sediments as identified in Section 7.1.1.6 above. Control sediments are also tested. For these static-renewal bioassays, control sediments are collected from an area in Tomales Bay that has proven to be non-toxic in previous sediment projects. Test procedures in the Testing Manual are used.

7.1.1.9 Mysid (*Mysidopsis bahia*)

Juvenile *Mysidopsis bahia* are cultured by and purchased from Aquatic Indicators, St. Augustine, Florida. During holding and testing, the mysids are fed about 75 brine shrimp nauplii (*Artemia salina*) per mysid three times per day to prevent mortality from starvation and cannibalism. The tests are initiated with < 5 day-old mysids.

Five replicates of each station, reference and control treatment are randomly assigned to test jars. A 2-cm deep layer of appropriate sediment is added to each jar on the day prior to test initiation. On the following day, each test jar is provided with aeration via Pasteur pipette and the test is started by randomly assigning 10 mysids to each jar. The test is continued for 10 days with water renewals every 48 hours and with constant aeration. Daily observations are made of each container. At this time, environmental test conditions (temperature, pH, dissolved oxygen and salinity) are measured in each test container.

At the end of the ten day exposure period, the contents of each jar are poured through a 0.5 mm sieve and the number of surviving mysids counted. Test data for each replicate include number of survivors.

Monthly reference toxicant testing is performed by the organism supplier on the Mysid cultures, using sodium dodecyl sulfate (SDS). Reference toxicant data are provided by the supplier for quality assurance purposes to verify the health and sensitivity of the test organism population.

7.1.1.10 Solid Phase Flow-through Bioassays with Polychaete Worm (*Nephtys caecoides*)

Solid phase materials from the site are bioassayed simultaneously with reference sediments. Control sediments are also tested. For these flow-through bioassays, control sediments are collected from an area in Tomales Bay that has proven to be non-toxic in previous sediment projects. Testing is performed using testing procedures in EPA/USACE (1991). *Nephtys* are collected from Tomales Bay by Brezina and Associates and purchased by ToxScan.

Worms are tested in large glass aquaria (31 liter). Five replicates of each test, reference, and control sediment are randomly assigned to the test tanks. A 3.0 cm layer of appropriate sediment is added to each test container. Tanks are then filled with laboratory seawater and allowed to settle overnight. The following morning, the flow-through seawater system is activated and adjusted to a flow rate equivalent to a 90% tank volume change every 4 hours (7 liters/hour). Twenty polychaete worms (*Nephtys caecoides*) are added to each container.

Solid phase flow-through bioassays are continued for 10 days. At least twice each day, environmental systems are checked for proper functioning. Once each day, the salinity, dissolved oxygen, pH, and temperature of the system are measured.

After the 10-day bioassay period, the contents of each tank are gently washed with seawater through a 0.5-mm nylon screen. The animals are retrieved from the screen and counted. Test data are the number of survivors.

Reference toxicant bioassays are performed on the worms for quality assurance purposes, to verify the health and sensitivity of the test organism populations. The reference toxicant used is cupric sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) dissolved in laboratory seawater for each species.

7.1.1.11 Bioaccumulation Assessment: Clam and Polychaete Worm

Bioaccumulation assessments are performed using the clam, *Macoma nasuta* and the polychaete worm, *Nereis viriens*. Clams are purchased from Brezina and Associates, and worms are supplied by Aquatic Research Organisms, Hampton, NH. The test specimens are exposed to test and control sediments in an array of 31-liter flow-through glass aquaria. Five replicates of each sample, reference, and control sediments are randomly assigned to the test tanks. The control sediment is collected from Tomales Bay, CA. A 3.0-cm layer of appropriate sediment is added to each test container. Tanks are then filled with laboratory seawater and allowed to settle overnight. The following morning the flow-through seawater system is activated and adjusted to a flow rate equivalent to a 90% tank volume change every 4 hours (7 liters/hour). Twelve clams and 10 worms are added to each test container. Five percent of the test containers receive 25 clams and 5 percent receive 20 worms for quality assurance purposes during tissue chemical analyses.

Bioaccumulation assessment exposure is continued for 28 days. At least twice each day, environmental systems are checked for possible malfunction. Daily monitoring of each tank for temperature, dissolved oxygen, salinity, and pH is performed.

After exposure, the contents of each tank are gently washed with seawater through a 0.5-mm nylon screen from which the animals are retrieved. Surviving clams and worms are transferred to filtered flowing seawater for 24-hours to evacuate their guts. Directly following these treatments, the soft tissues of clams and worms are thoroughly homogenized for chemical analyses. For this purpose, a stainless steel Tekmar Tissuemizer is used. Before use, the entire blade and barrel assembly is cleaned in hot DI water with Micro detergent, then rinsed thoroughly in DI water after washing and again just prior to use. Between samples a three-stage rinse is done. This has proven to minimize the chance of sample cross-contamination. Samples are triple-wrapped and frozen when not being used. All tissue handling and processing is conducted at a laminar flow bench in a trace metal clean lab. Tissue samples are analyzed according to the list of constituents and analytical detection limits in Table 4-1 of the Work Plan (CH2M HILL, 2000). The analytical methods are listed in Table 7-1. Tissues are stored at -20+5 degrees Celsius until analysis.

7.1.1.12 Data Reduction Analysis and Interpretation

Statistical analysis of experimental data follows each of the bioassay and bioaccumulation experiments. Tests of fundamental assumptions (e.g., variance homogeneity) are followed by the appropriate parametric or non-parametric analyses.

In cases where a contaminant is detected in tissues of organisms exposed to test sediment but is not detected (ND) in reference tissues, a value is assigned to the ND sample which equals 50% of the analytical detection limit (DL) for that contaminant. This is consistent with interim recommendations by the EPA/COE in the Inland Testing Manual (EPA/COE, 1994).

Variance homogeneity is one of the underlying assumptions of most parametric statistics. Cochran's test is therefore applied to the data from the bioassays and the tissue chemistry of the bioaccumulation experiments. Significant results for this and all subsequent parametric tests are determined by the critical value ($\alpha = 0.05$) of the appropriate distributions.

Once homogeneity has been established, the analysis of variance (ANOVA) and Dunnett's test are employed to analyze differences between treatment responses (e.g., test sediment tanks).

When sample variances do not exhibit homogeneity, as determined by Cochran's test, the Testing Manual recommends data transformation. Arcsine/square-root data transformation is applied to proportional data of bioassays and logarithmic data transformation is applied to bioaccumulation data. When the data transformation is unable to compensate for the non-homogeneity of the variance, non-parametric tests are employed.

Non-parametric procedures use ranked values for calculating test statistics and the corresponding hypotheses use rank sums for comparison. Kruskal-Wallis and Wilcoxon-Wilcox tests are used to identify differences between treatment responses.

The Testing Manual guidelines for interpretation of suspended particulate phase bioassays require that, for any sample producing toxicity sufficient to generate an LC50, initial mixing calculations be performed to determine the concentration of suspended particulate material remaining at the disposal site within four hours after dumping (Csp). If the Csp does not

exceed 1% of the LC50, the sediment is judged to comply with water column toxicity criteria.

Guidelines for interpretation of Solid Phase bioassay results are provided by the Testing Manual. If survival responses in test sediment are statistically significantly lower than those in reference sediment and if the difference in mean survival between groups is greater than 10% (20% for amphipods), then the test sediment is considered to have the potential to significantly degrade the marine environment.

Guidelines for evaluation of bioaccumulation are described in the Testing Manual and final interpretation is left to the District Engineer and the Regional Administrator. Therefore, statistical testing of bioaccumulation test phase results is complete when appropriate comparison (Dunnett's or Wilcoxon-Wilcox) describes significant or non-significant tissue burden from exposure to dredged material.

7.1.2 Bioassay Quality Assurance

Quality assurance measures applied to aquatic toxicity testing are explicitly stated in all standard protocols. Such measures include test temperatures and acceptable limits for variation, minimum acceptable dissolved oxygen levels with aeration procedures to be used as required by each method, and acceptable pH range to be employed. Salinity ranges are specified for marine tests. A schedule for monitoring these environmental parameters is usually provided, and bioassay results must include these monitoring data. Organism assignment to test tanks and test tank positioning in the laboratory are randomized.

The single most important quality assurance measure in bioassay tests is the inclusion of an experimental control, wherein organisms are simultaneously exposed to laboratory test conditions in the absence of any toxicant stress. For suspended particulate phase test media, control organisms are generally exposed to dilution water only. For sediment testing, the control exposure consists of a known non-toxic or artificial sediment. All protocols require that an identified minimum level of normal organism end point behavior (e.g. survival, normal development, fertilization) be achieved in order for the test to be considered valid. If, for example, less than 90% control survival in a whole sediment bioassay is observed, then the test must be repeated.

Another important QA measure that is routinely implemented in bioassay testing is the reference toxicant bioassay. Documented biological variations in test organisms themselves can affect toxicity test results. Routine parallel reference toxicant bioassays provide a way to normalize this type of variability. In addition, the routine use of reference toxicants provides useful data towards calibrating individual laboratory performance in programs where different laboratories are providing test data from the same protocol. In this situation, comparable reference toxicant results would support the assumption of comparable test performance quality and therefore would increase confidence in overall program data comparability.

In addition to the QA guidelines in the Testing Manual, the laboratory may provide additional bioassay quality assurance measures. Organism culture and collection, transportation, feeding, and acclimation procedures are designed to minimize stress and to maintain organisms in optimal condition. Laboratory water supply and environmental

control systems should be redundant whenever possible to avoid undue variation during holding acclimation.

7.2 Definitive Chemical Data

Table 4-1 of the Work Plan lists the analytes for each analytical suite. The methods to be used are listed in Table 7-1. These methods supersede those specified in the Work Plan. The accuracy and precision limits are listed in Table 7-2. Calibration and QC requirements are specified in Tables 7-3 through 7-12.

TABLE 7-1 Analytical Methods for Sediment, Water, and Tissue Samples		
Analyte	Method	
	Water	Sediment/Tissue
Arsenic	SW7061	SW7060 (Suite A/B) SW7061 (Suite C/E)
Lead	SW6020	SW7421(Suite A/B) SW6020 (Suite C/E)
Mercury	SW7470	SW7471
Selenium	SW7741	SW7740 (Suite A/B) SW7741 (Suite C/E)
Thallium	SW6020	SW7841 (Suite A/B) SW6020 (Suite C/E)
Chromium VI	SW7196	SW7196
All other metals	SW6020	SW6010A (Suite A/B) SW6020 (Suite C/E)
TEPH (Total Extractable Petroleum Hydrocarbons)	SW8015 Modified	SW8015 Modified
Organochlorine Pesticides and PCBs	SW8081 (see section 4.5.4 for PCB analysis)	SW8081 (see section 4.5.4 for PCB analysis)
VOC	-	SW8260A
PAH/Phthalates	SW8270B	SW8270B
Organophosphorus Pesticides	SW8141A	SW8141A
Chlorinated Herbicides	SW8150B	SW8150B
Total Organic Carbon	SW9060	Gaudette (Walkley-Black method)
Oil and Grease/Percent Lipids	EPA 1664	EPA 1664
Sulfate	EPA 300.0	EPA 300.0
Sulfide	EPA 376.1	SW9030
Ammonia	EPA 350.2	EPA 350.2
pH	EPA 150.1	SW9045B
Conductivity	EPA 120.1	EPA 120.1
Total Volatile Solids	-	EPA 160.4
Particle Size	-	Plumb/D422
Moisture Content	-	D2216

TABLE 7-2 Accuracy and Precision Limits					
Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Sediment and Tissue (%R)	Precision Sediment and Tissue (%RPD)
All methods	All metals and general inorganic chemistry parameters	75-125	≤20	75-125	≤20
SW8270C	Acenaphthene	46-118	≤31	31-137	≤19
	Benzo(b)fluoranthene	26-127	≤31	35-142	≤36
	Pyrene	26-127	≤31	35-142	≤36
	2-Fluorobiphenyl (Surr.)	43-116	-	30-115	-
	Terphenyl-d14 (Surr.)	33-141	-	18-137	-
SW8260A	Benzene	-	-	66-142	21
	Chlorobenzene	-	-	60-133	21
	Trichloroethene	-	-	62-137	24
	Toluene	-	-	59-139	21
	Toluene-d8 (Surr.)	-	-	84-138	-
	Bromofluorobenzene (Surr.)	-	-	59-113	-
	1,2-Dichloroethane-d4 (Surr.)	-	-	70-121	-
	Dibromofluoromethane (Surr.)	-	-	80-117	-
SW8081	Aldrin	40-120	≤22	34-132	≤43
	Dieldrin	52-126	≤18	31-134	≤38
	4,4'-DDT	38-127	≤27	23-134	≤50
	Endrin	56-121	≤21	42-139	≤45
	Gamma-BHC	56-123	≤15	46-127	≤50
	Aroclor 1254/PCB 101 ^a	50-150	≤50	50-150	≤50
	Tetrachloro-m-xylene (Surr.)	30-150	-	30-150	-
	Decachlorobiphenyl/ Dibutylchloroendate ^b	30-150	-	30-150	-
SW8015 ^c Modified	Diesel	50-150	≤50	50-150	≤50
	o-Terphenyl (Surr.)	65-125	-	65-125	-
	Octacosane (Surr.)	26-152	-	25-162	-
	Triacontane (Surr.)	40-140	-	30-150	-
SW8141A	Diazinon	50-150	≤30	50-150	≤50
	Disyston	50-150	≤30	50-150	≤50
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Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Sediment and Tissue (%R)	Precision Sediment and Tissue (%RPD)
	Tributyl Phosphate (Surr.) (An alternate surrogate may be used.)	50-150	-	50-150	-
SW8150B	2,4,5-TP	75-125	≤30	65-135	≤50
	2,4-DB	65-125	≤30	55-135	≤50
	Dalapon	70-125	≤30	60-135	≤50
	2,4-Dichlorophenylacetic acid (Surr.)	60-140	-	50-150	-

^a Use PCB 101 when analyzing for PCB congeners.
^b Use Dibutylchlorodate when analyzing for PCB congeners.
^c At least one surrogate must be spiked.

QC Check	Frequency	Criteria	Corrective Action
Initial calibration (a blank and at least one standard)	Before initial sample analysis, every 24 hours, whenever modifications are made to the analytical system, or when continuing calibration verification fails	N/A	N/A
Initial calibration verification (ICV); must be from second source	Immediately following each initial calibration	All analytes within ±10% of expected value	Correct problem and repeat initial calibration.
Calibration blank	After every calibration verification (ICV and CCV)	No analytes detected at or above the RDL	Correct the problem, then reanalyze previous 10 samples.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ±10% of expected value	Recalibrate and reanalyze all samples since the last acceptable CCV
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prepare and reanalyze all associated samples

TABLE 7-3 Calibration and QC Requirements for SW6010A			
QC Check	Frequency	Criteria	Corrective Action
Interference check standard (ICS)	At the start and end of each analytical sequence of twice during an 8-hour period, whichever is more frequent	All analytes within $\pm 20\%$ of expected value	Correct the problem, recalibrate, reanalyze ICS and all affected samples.
MS/MSD	One set per 20 Bolsa Chica samples	All analytes within limits specified in Table 7-1	None
LCS	At least one per analytical batch	All analytes within limits specified in Table 7-1	Correct the problem, and re-prepare and reanalyze the LCS and all samples in the analytical batch.
Dilution test	Each new sample matrix	Result from 1:5 dilution must be within $\pm 10\%$ of the undiluted sample result (applies only if undiluted sample result is at least 25 times the MDL)	Perform post-digestion spike addition.
Post-digestion spike addition	When dilution test fails	Recovery within 75-125% of expected value	None

TABLE 7-4 Calibration and QC Requirements for SW6020			
QC Check	Frequency	Criteria	Corrective Action
MS tuning	Prior to initial calibration	Per SW6020, section 5.8	Retune instrument and reanalyze tuning solution.
Initial calibration (a blank and at least one standard)	Before initial sample analysis, every 24 hours, whenever modifications are made to the analytical system, or when continuing calibration verification fails	N/A	N/A
Initial calibration verification (ICV); must be from second source	Immediately following each initial calibration	All analytes within $\pm 20\%$ of expected value	Correct problem and repeat initial calibration.
Calibration blank	After every calibration verification (ICV and CCV)	No analytes detected at or above the RDL	Correct the problem, then reanalyze previous 10 samples.

TABLE 7-4 Calibration and QC Requirements for SW6020			
QC Check	Frequency	Criteria	Corrective Action
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 20\%$ of expected value	Recalibrate and reanalyze all samples since the last acceptable CCV
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prepare and reanalyze all associated samples
Interference check standard (ICS)	At the start and end of each analytical sequence of twice during an 8-hour period, whichever is more frequent	All analytes within $\pm 20\%$ of expected value	Correct the problem, recalibrate, reanalyze ICS and all affected samples.
MS/MSD	One set per 20 Bolsa Chica samples	All analytes within limits specified in Table 7-1	None
LCS	At least one per analytical batch	All analytes within limits specified in Table 7-1	Correct the problem, and re-prepare and reanalyze the LCS and all samples in the analytical batch.
Dilution test	Each new sample matrix	Result from 1:5 dilution must be within $\pm 10\%$ of the undiluted sample result (applies only if undiluted sample result is at least 25 times the MDL)	Perform post-digestion spike addition.
Post-digestion spike addition	When dilution test fails	Recovery within 75-125% of expected value	None
Internal standards	Every sample	IS intensity within 30-120% of the IS intensity in the initial calibration	Perform corrective action as described in SW6020, section 8.3.

TABLE 7-5 Calibration and QC Requirements for SW7470/SW7471			
QC Check	Frequency	Criteria	Corrective Action
Multi-point initial calibration (a blank and at least five standards)	Before initial sample analysis, every 24 hours, whenever modifications are made to the analytical system, or when continuing calibration verification fails	Correlation coefficient of linear regression is ≥ 0.995	Correct the problem and repeat the initial calibration.
Initial calibration verification (ICV); must be from second source	Immediately following each initial calibration	All analytes within $\pm 20\%$ of expected value	Correct the problem and repeat initial calibration.

QC Check	Frequency	Criteria	Corrective Action
Calibration blank	After every calibration verification (ICV and CCV)	No analytes detected at or above the RDL	Correct the problem, then reanalyze previous 10 samples.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 20\%$ of expected value	Recalibrate and reanalyze all samples since the last acceptable CCV
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prep and reanalyze all associated samples
MS/MSD	One set per 20 Bolsa Chica samples	All analytes within limits specified in Table 7-1	None
LCS	At least one per analytical batch	All analytes within limits specified in Table 7-1	Correct the problem, and re-prep and reanalyze the LCS and all samples in the analytical batch.
Dilution test	Each new sample matrix	Result from 1:5 dilution must be within $\pm 10\%$ of the undiluted sample result (applies only if undiluted sample result is at least 25 times the MDL)	Perform post-digestion spike addition.
Recovery test	When dilution test fails	Recovery within 85-115% of expected value	Dilute sample to reduce background, if necessary, and repeat recovery test; otherwise, analyze all samples by MSA.

QC Check	Frequency	Criteria	Corrective Action
Multi-point initial calibration (a blank and at least three standards)	Before initial sample analysis, every 24 hours, whenever modifications are made to the analytical system, or when continuing calibration verification fails	Correlation coefficient of linear regression is ≥ 0.995	Correct the problem and repeat the initial calibration.
Initial calibration verification (ICV); must be from second source	Immediately following each initial calibration	All analytes within $\pm 10\%$ of expected value	Correct the problem and repeat initial calibration.

QC Check	Frequency	Criteria	Corrective Action
Calibration blank	After every calibration verification (ICV and CCV)	No analytes detected at or above the RDL	Correct the problem, then reanalyze previous 10 samples.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 10\%$ of expected value	Recalibrate and reanalyze all samples since the last acceptable CCV
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prepare and reanalyze all associated samples
MS/MSD	One set per 20 Bolsa Chica samples	All analytes within limits specified in Table 7-1	None
LCS	At least one per analytical batch	All analytes within limits specified in Table 7-1	Correct the problem, and re-prepare and reanalyze the LCS and all samples in the analytical batch.
Dilution test	Each new sample matrix	Result from 1:5 dilution must be within $\pm 10\%$ of the undiluted sample result (applies only if undiluted sample result is at least 25 times the MDL)	Perform post-digestion spike addition.
Recovery test	When dilution test fails	Recovery within 85-115% of expected value	Dilute sample to reduce background, if necessary, and repeat recovery test; otherwise, analyze all samples by MSA.

QC Check	Frequency	Criteria	Corrective Action
BFB tuning	Prior to initial calibration and prior to each calibration verification (every 12 hours)	Must meet BFB ion abundance criteria	Re-tune the instrument and reanalyze the tuning standard.
Multi-point initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification fails	Average RRF of SPCCs must meet minimum values (0.10 for chloromethane, 1,1-dichloroethane, and bromoform; 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane). %RSD for each CCC must be $\leq 30\%$. To use average RRF for quantitation of any analyte, % RSD must be $\leq 15\%$; otherwise use calibration curve with coefficient of correlation or determination ≥ 0.99 .	If 1) or 2) is not met, correct the problem and repeat the initial calibration.
Continuing calibration verification (CCV)	At the start of each analytical sequence and after every 12 hours	Average RRF of SPCCs must meet minimum values (0.10 for chloromethane, 1,1-dichloroethane, and bromoform; 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane). All CCCs within $\pm 20\%$ of expected value if using calibration curves; if using average RRFs for quantitation, %D between the RRF of CCCs and the initial calibration average RRF must be $\leq 20\%$.	If 1) or 2) is not met, correct the problem, then recalibrate and reanalyze all affected samples.
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and reanalyze all associated samples
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS	All surrogates in samples, method blank, MS/MSD, and LCS within limits specified in Table 7-1	Correct the problem and reanalyze.
MS/MSD	One set per 20 Bolsa Chica samples	All analytes within limits specified in Table 7-1	None

TABLE 7-7 Calibration and QC Requirements for SW8260A			
QC Check	Frequency	Criteria	Corrective Action
LCS	At least one per analytical batch	All analytes within limits specified in Table 7-1	Correct the problem, and reanalyze the LCS and all samples in the analytical batch.
Internal standards	Each CCV standard	<p>RT of each internal standard in the CCV standard must be within 30 seconds of the RT of that IS in the last 12-hour CCV standard.</p> <p>The area of each internal standard in the CCV standard must be within – 50% to +100% of the area of that IS in the last 12-hour CCV standard.</p> <p>If the CCV standard is the first in the sequence, its internal standard RTs and areas must be compared with those of the initial calibration standard of the same concentration as the CCV standard.</p>	If 1) or 2) is not met, inspect the GC/MS for malfunctions, correct the problem, and recalibrate and reanalyze all affected samples.

TABLE 7-8 Calibration and QC Requirements for SW8270B			
QC Check	Frequency	Criteria	Corrective Action
DFTPP tuning	Prior to initial calibration and prior to each calibration verification (every 12 hours)	Must meet DFTPP ion abundance criteria	Re-tune the instrument and reanalyze the tuning standard.
Multi-point initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification fails	<p>Average RRF of SPCCs must be at least 0.050.</p> <p>%RSD for each CCC must be $\leq 30\%$.</p> <p>To use average RRF for quantitation of any analyte, % RSD must be $\leq 15\%$; otherwise use calibration curve with coefficient of correlation or determination ≥ 0.99.</p>	If 1) or 2) is not met, correct the problem and repeat the initial calibration.

TABLE 7-8 Calibration and QC Requirements for SW8270B											
QC Check	Frequency	Criteria	Corrective Action								
Continuing calibration verification (CCV)	At the start of each analytical sequence and after every 12 hours	Average RRF of SPCCs must be at least 0.050. All CCCs within $\pm 20\%$ of expected value if using calibration curves; if using average RRFs for quantitation, %D between the RRF of CCCs and the initial calibration average RRF must be $\leq 20\%$.	If 1) or 2) is not met, correct the problem, then recalibrate and reanalyze all affected samples.								
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL*	Correct the problem and reanalyze all associated samples								
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS	All surrogates in samples, method blank, MS/MSD, and LCS within limits specified in Table 7-1	Correct the problem and reanalyze (re-prep if necessary).								
MS/MSD	One set per 20 Bolsa Chica samples	All analytes within limits specified in Table 7-1	None								
LCS	At least one per analytical batch	All analytes within limits specified in Table 7-1	Correct the problem, and re-prep and reanalyze the LCS and all samples in the analytical batch.								
Internal standards	Each CCV standard	RT of each internal standard in the CCV standard must be within 30 seconds of the RT of that IS in the last 12-hour CCV standard. The area of each internal standard in the CCV standard must be within -50% to $+100\%$ of the area of that IS in the last 12-hour CCV standard. If the CCV standard is the first in the sequence, its internal standard RTs and areas must be compared with those of the initial calibration standard of the same concentration as the CCV standard.	If 1) or 2) is not met, inspect the GC/MS for malfunctions, correct the problem, and recalibrate and reanalyze all affected samples.								
<p>*Maximum limits for phthalates in soil/sediment method blank:</p> <table border="0"> <tr> <td>Butylbenzyl phthalate</td> <td>11 mg/kg</td> </tr> <tr> <td>Diethyl phthalate</td> <td>0.63 mg/kg</td> </tr> <tr> <td>Di-n-butyl phthalate</td> <td>11 mg/kg</td> </tr> <tr> <td>Bis(2-ethylhexyl)phthalate</td> <td>No limit</td> </tr> </table>				Butylbenzyl phthalate	11 mg/kg	Diethyl phthalate	0.63 mg/kg	Di-n-butyl phthalate	11 mg/kg	Bis(2-ethylhexyl)phthalate	No limit
Butylbenzyl phthalate	11 mg/kg										
Diethyl phthalate	0.63 mg/kg										
Di-n-butyl phthalate	11 mg/kg										
Bis(2-ethylhexyl)phthalate	No limit										

QC Check	Frequency	Criteria	Corrective Action
Multi-point initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification fails	If the average %RSD is \leq 20%, the average RRF may be used for quantitation; otherwise use calibration curve with coefficient of correlation or determination \geq 0.99.	Correct the problem and repeat the initial calibration.
Continuing calibration verification (CCV)	At the start of each analytical sequence and after every 10 samples, and at the end of the sequence	All analytes within \pm 15% of expected value	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prepare and reanalyze all associated samples
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS	All surrogates in samples, method blank, MS/MSD, and LCS within limits specified in Table 7-1	Correct the problem and reanalyze (re-prepare if necessary).
MS/MSD (Diesel)	One set per 20 Bolsa Chica samples	Within limits specified in Table 7-1	None
LCS (Diesel)	At least one per analytical batch	Within limits specified in Table 7-1	Correct the problem, and re-prepare and reanalyze the LCS and all samples in the analytical batch.

QC Check	Frequency	Criteria	Corrective Action
Multi-point initial calibration (minimum five points) for single-response pesticides and individual PCB congeners (single-point calibration for Toxaphene and Chlordane); multi-point calibration for Aroclors 1016 and 1260 only, but include mid-point standard for all other Aroclors for pattern recognition; if a specific Aroclor is found in any sample, quantitation for that Aroclor must be done using 5-point calibration.	Prior to sample analysis, or when calibration verification fails	If the average %RSD is \leq 20%, the average RRF may be used for quantitation; otherwise use calibration curve with coefficient of correlation or determination \geq 0.99.	Correct the problem and repeat the initial calibration.

TABLE 7-10 Calibration and QC Requirements for SW8081			
QC Check	Frequency	Criteria	Corrective Action
Continuing calibration verification (CCV) – pesticides, individual PCB congeners, and Aroclors 1016 and 1260 (or Aroclors identified in samples)	At the start of each analytical sequence, after every 12 hours or 10 samples, whichever is more frequent, and at the end of the sequence	All analytes within $\pm 15\%$ of expected value	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Endrin/DDT breakdown check	At start of each 12 hour period	Breakdown of either Endrin or DDT $\leq 15\%$	Evaluate injector port and take corrective action; re-calibrate and reanalyze affected samples if necessary
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prep and reanalyze all associated samples
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS	At least one of the surrogates in samples, method blank, MS/MSD, and LCS within limits specified in Table 7-1	Correct the problem and reanalyze (re-prep if necessary).
MS/MSD	One set per 20 Bolsa Chica samples	Within limits specified in Table 7-1	None
LCS	At least one per analytical batch	Within limits specified in Table 7-1	Correct the problem, and re-prep and reanalyze the LCS and all samples in the analytical batch.
Second column confirmation (not required for Aroclors)	All samples with detections at or above the RDL must be confirmed within the holding time.	Confirmation to be done using second column of dissimilar phase and retention characteristics (or GC/MS if sample concentration is sufficiently high)	Failure to perform confirmation will result in potential resampling and analysis at no cost to the project.

TABLE 7-11			
Calibration and QC Requirements for SW8141A and SW8150B			
QC Check	Frequency	Criteria	Corrective Action
Multi-point initial calibration (minimum five points).	Prior to sample analysis, or when calibration verification fails	If the average %RSD is \leq 20%, the average RRF may be used for quantitation; otherwise use calibration curve with coefficient of correlation or determination \geq 0.99.	Correct the problem and repeat the initial calibration.
Continuing calibration verification (CCV)	At the start of each analytical sequence, after every 12 hours or 10 samples, whichever is more frequent, and at the end of the sequence	All analytes within \pm 15% of expected value	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prepare and reanalyze all associated samples
Surrogate spike	Every standard, sample, method blank, MS/MSD, and LCS	At least one surrogate in samples, method blank, MS/MSD, and LCS within limits specified in Table 7-1	Correct the problem and reanalyze (re-prepare if necessary).
MS/MSD	One set per 20 Bolsa Chica samples	Within limits specified in Table 7-1	None
LCS	At least one per analytical batch	Within limits specified in Table 7-1	Correct the problem, and re-prepare and reanalyze the LCS and all samples in the analytical batch.
Second column confirmation	All samples with detections at or above the RDL must be confirmed within the holding time.	Confirmation to be done using second column of dissimilar phase and retention characteristics (or GC/MS if sample concentration is sufficiently high)	Failure to perform confirmation will result in potential resampling and analysis at no cost to the project.

TABLE 7-12			
Calibration and QC Requirements for General Inorganic Chemistry			
QC Check	Frequency	Criteria	Corrective Action
Multi-point initial calibration (minimum three points); for titrimetric methods, titrant must be standardized in duplicate, and the average concentration used; for gravimetric methods, balance must be calibrated using standard weights that bracket sample weights.	Prior to sample analysis, or when calibration verification fails	Correlation coefficient for linear regression must be ≥ 0.995 (not applicable to titrimetric and gravimetric methods)	Correct the problem and repeat the initial calibration.
Continuing calibration verification (CCV) – does not apply to titrimetric and gravimetric methods.	At the start of each analytical sequence, after every 10 samples, and at the end of the sequence	All analytes within $\pm 10\%$ of expected value	Correct the problem, then recalibrate and reanalyze all samples since the last acceptable CCV.
Method Blank	At least one per analytical batch	No analytes detected at or above the RDL	Correct the problem and re-prepare and reanalyze all associated samples
MS/MSD (One MS and one set of laboratory duplicates may be substituted for MS/MSD)	One set per 20 Bolsa Chica samples	Within limits specified in Table 7-1	None
LCS	At least one per analytical batch	Within limits specified in Table 7-1	Correct the problem, and re-prepare and reanalyze the LCS and all samples in the analytical batch.

8.0 Data Validation

Measurement data should be consistently assessed and documented to determine whether program DQOs have been met, to assess data quality quantitatively, to identify potential limitations on data use, and to assess whether site-specific DQOs have been met. The data quality evaluations of the chemical data for this project are patterned after the U.S. EPA Contract Laboratory Program Functional Guidelines for Organic and Inorganic Data Review, February 1994. The data evaluation for bioassay/toxicity testing is included in the QAPP documents listed in the References section.

8.1 Chemical Data Evaluation

A batch QA review will be performed by the contractor for all data. A batch review is typically referred to as data evaluation.

The routine QC procedures conducted in the laboratory are established in the published methods, this QAPP, and the analytical SOPs prepared by each laboratory. The laboratory will be responsible for following the procedures as specified in this QAPP (and/or the site-specific FSP) and operating the analytical systems within statistical control limits. These procedures include proper instrument maintenance, calibration and calibration checks, and laboratory QC sample analyses at the required frequency. Associated QC sample analytical results are reported with the sample results so the project staff can evaluate the analytical process performance.

All project data will be reviewed as part of data evaluation. The review will be conducted on an analytical or preparation batch basis or by evaluating QC samples and all associated field sample results. Project data evaluation procedures established for the project generally include:

- Review of initial and continuing calibration verifications;
- Initial review of analytical and field data for complete and accurate documentation, chain-of-custody records, analytical holding time compliance, and required frequency of field and laboratory QC samples;
- Evaluation of method and field blank results to identify systematic contamination;
- Comparison of all types of spike and duplicate results with project objectives for precision and accuracy;
- Statistical calculations for overall method accuracy and precision using the appropriate QC sample results;
- Assigning data qualifier flags to the data as necessary to reflect limitations identified by the process; and

- Calculating completeness by method and matrix or by analyte, if designated.

Some of the statistical calculations commonly used for the data evaluation process and ways in which the calculations apply to environmental sample results are presented in Table 8-1. Additional statistical procedures may be applied to the data to assess reporting limits or other quality-related parameters. The calculations and procedures are documented in the QA/QC summary report.

Qualifier flags will be applied to sample results that fail to meet the DQOs according to the flagging conventions in Tables 8-2 and 8-3. The tables should be used as minimum data evaluation criteria. The data evaluator should use professional judgment and apply additional criteria when appropriate. The qualifier codes, or flags, will be stored with the data in the Bolsa Chica database. Circumstances may be encountered which warrant deviations from these flagging guidelines. The technical reasoning will be documented with the data package or the data quality assessment report in these instances. Reanalysis or resampling may be recommended as a corrective action if data are determined to be unacceptable for the intended use. Definitions of the qualifier flags are presented in Table 8-4. Table 8-5 shows the relationships between QC and field samples that may be similarly influenced by QC problems.

A distinction must be made between quality control and data review conducted as a part of laboratory operations (Section 8.0) and the project-related data evaluation conducted after data have been reported. Planning, use of standard field, analytical, and QC procedures, and auditing performed during field and laboratory activities are designed to control the sampling and analytical processes to produce data of sufficient quality for project needs. If a problem occurs in spite of these controls, the data evaluation must identify the problem, determine which data are affected, state how use of the data may be limited, and make recommendations for corrective actions as necessary.

The QA/QC staff conducting data evaluation are responsible for ensuring that data qualifier flags are assigned as needed based on the established QC criteria, and any limitations are communicated to the data users. These data qualifier flags are not related to any flags that may be assigned by the laboratory. Data qualifier flags explain the type and extent of the limitation placed on a result, while laboratory flags identify QC results that are outside laboratory tolerances and may or may not lead to subsequent data qualifiers assigned during data evaluation. The QA/QC staff are also responsible for initiating corrective actions for analytical or other problems identified during the data evaluation process. Corrective actions range from verifying that the method was in statistical control during the analytical runs to reanalysis of the sample or re-sampling, or re-issuing the laboratory report due to clerical errors in the report.

8.2 Chemical Blank Data Evaluation

Blank results indicate whether any reported analytes may be attributed to laboratory sources (reagents, glassware, instrumentation) or field sources or conditions (equipment, shipping and handling, ambient conditions) rather than the sample. Laboratory blanks include method or system blanks included in each preparation and analytical batch.

Equipment, trip, and ambient blanks are field blanks collected at specified frequencies or under selected conditions to monitor contamination from non-laboratory sources.

The most common laboratory contaminants, methylene chloride, phthalates, acetone, toluene, and 2-butanone are ubiquitous; controlling them within acceptable low levels is part of standard laboratory procedures. When these or other analytes are reported in field samples or field blanks at concentrations within ten times that found in an associated laboratory blank, the field sample results will be "U" flagged to indicate that the analytes should be considered not detected. Common contaminants in field samples that were not reported in the associated laboratory blank may be qualified if the contamination appears systematic (i.e., if the contaminant is detected in a majority of the other laboratory blanks).

Field blank results are evaluated individually, and related to the field samples as shown in Table 8-5. The probable contamination source is identified and associated sample results are qualified as necessary based on the relative concentrations between the blank and the sample. For example, if equipment blank results show contamination and the sample collected from the bailer shows the same analyte at concentrations attributable to blank concentrations, the sample results are "U" flagged to indicate that they should be considered not detected. Samples collected before and after the blank are also evaluated to determine the potential sources and impacts of carryover. Because equipment blanks are water samples, contaminant concentrations reported in blanks cannot be directly related to concentrations in soil samples. Judgment must be used to determine whether any analyte reported in the blank and associated soil samples should be qualified.

8.3 Chemical Accuracy

Accuracy is associated with correctness and is a comparison between a measured value and a known or expected value. Accuracy is assessed by comparing LCS, MS, surrogate spike and performance evaluation (PE) sample recoveries with the project objectives presented in Table 7-2, and also taking into account manufacturer's tolerances on commercially purchased PE samples.

Laboratory Control Samples

Laboratory control samples (LCS) are spikes of method analytes in reagent-grade water (or may be commercially purchased solid LCSs). The LCSs are taken through sample preparation and analysis to assess statistical control of the method. If the recovery is outside the established tolerances, samples from the same preparation and/or analytical batch should be suspected to have similar analyte recoveries and should be qualified. Any not-detected sample results associated with low LCS recoveries may indicate potential false negatives and the reporting limits for the analytes should be flagged as estimated. Positive sample results associated with low or high LCS recoveries should be flagged as estimated. The system must be assessed to determine the reason for the out-of-tolerance occurrence, and corrective action may be indicated, up to and including re-extraction and reanalysis (if still within holding time) or re-sampling of affected samples.

Matrix Spikes

Matrix spike results are assessed by comparison with the recovery ranges presented in this QAPP. If MS recoveries are outside this range, two conditions must be evaluated:

- The spike concentration relative to the parent sample concentration; and
- The associated LCS recovery.

If the parent sample concentration is greater than four times the spike concentration, the spike concentration is considered insignificant, relative to sample dilution and/or analytical variability. Since the recovery does not represent the ability to recover the analyte from the matrix, it is generally not calculated, or at least should not be used to qualify data.

If MS and/or MSD recovery is outside the specified range and the associated LCS is within specification, a matrix interference is demonstrated and sample results are qualified as estimated or are rejected if recoveries are extremely high or low. If systematic matrix interference is exhibited, similar sample results such as those from the same site or lithology must be evaluated. The reviewer's judgment is used to determine if the results should be qualified.

The qualified data are discussed in the sampling task QC report, and specific limitations such as poor or enhanced recovery for specific analytes is discussed. Further investigation or corrective action may be taken to find alternatives to reduce interferences.

Surrogate Spikes

Surrogate spike results, associated with organic analyses, are used to assess target analyte recovery for each sample and measure system performance and matrix interference.

Surrogate spike recoveries are compared to the recovery tolerances presented in this QAPP. Surrogates represent the different types or classes of analytes measured by a method, and the results are used to qualify similar analytes (e.g., acid extractable, phenolic, etc.). Field sample results that have surrogate recoveries outside the project specifications are qualified as estimates or are rejected if recoveries are very low or zero. Where surrogates coelute with non-target analyses or are low due to sample dilution, qualification of data will not be required.

8.4 Precision

Precision is a measure of variability between duplicate analyses and is calculated for field and laboratory duplicates. Precision is evaluated by comparing the relative percent difference (RPD) between MS and MSD results, and between laboratory duplicate results with the RPD criteria listed in Table 7-2. Precision criteria for field duplicate results are specified in Tables 8-2 and 8-3.

If RPDs exceed the criteria, the analytical results for the samples collected by the same sampling team, from the same equipment, from the same site, from similar matrices (soil samples), or on the same day, may be affected. Close evaluation of the results should indicate the most likely source of variability, and the corresponding samples are qualified as warranted.

If all analytical specifications are satisfied and sampling error is not suspected, the RPD results may indicate variability in the matrix. RPD results should be used to initiate further evaluation but are not necessarily considered to be indicators of the state of control during analysis or of field conditions. Estimated qualifier flags may be assigned for samples or matrices with high RPDs to indicate sample heterogeneity or high matrix variability rather than a data quality problem.

An average RPD may be calculated and reported as a measure of overall analytical precision or matrix variability for methods and analytes with many duplicate samples or analyses.

8.5 Completeness

Completeness is calculated for each method and matrix after the QC data have been evaluated and data qualifiers assigned. The calculation for completeness can be found in Section 4.2.

8.6 Interlaboratory Data Comparison

Multiple laboratories may perform the same analytical methods on project samples. An interlaboratory comparison may be conducted to identify laboratory contamination or conditions that may influence the comparability of the results. The complexity of the comparison will depend upon the number of samples and volume of QC results reported by each laboratory. At a minimum, a qualitative evaluation must be performed to evaluate:

- Blank contaminants and concentrations reported;
- LCS and MS/MSD recoveries and RPD ranges;
- Surrogate spike recovery ranges; and
- PQLs and dilution factors.

If the types or concentrations of blank contaminants differ, further data assessment and qualification may be warranted. The spike recovery ranges and RPD ranges should be evaluated for large differences that may indicate greater analytical variability in one laboratory than that in another. Recoveries and ranges for one laboratory that are consistently higher or lower than others could indicate a systematic bias that should be addressed with corrective action.

Influence on sample results should be addressed in the project report, and corrective actions should be initiated if systematic problems are indicated. Performance evaluation samples submitted to all participating laboratories may be considered as a follow-up check on the findings of the comparison.

TABLE 8-1
Statistical Calculations

Statistic	Symbol	Formula	Definition	Uses
Mean	\bar{X}	$\frac{n}{\sum_{i=1}^n \frac{X_i}{n}}$	Measure of central tendency	Needed for additional statistical calculations
Standard Deviation	S	$\left[\frac{n}{\sum_{i=1}^n \frac{(X_i - \bar{X})^2}{n-1}} \right]^{1/2}$	Measure of relative scatter of the data	Needed for additional statistical calculations
Coefficient of Variation	CV	$\left(\frac{S}{\bar{X}} \right) \times 100$	Also called the relative standard deviation (RSD); adjusts for the magnitude of observations	Used to assess precision for replicate results
Pooled CV	CV	$\left[\frac{n \sum_{i=1}^n (CV_i)^2 df_i}{\sum_{i=1}^n df_i} \right]^{1/2}$	Measure of overall variability of a series	Used to assess overall performance for compounds or methods with multiple measurements

TABLE 8-1
Statistical Calculations

Statistic	Symbol	Formula	Definition	Uses
Relative Percent Difference	RPD	$\left[\frac{(X_1 - X_2)}{\left(\frac{(X_1 + X_2)}{2} \right)} \right] \times 100$	Measure of variability that adjusts for the magnitude of observations	Used when there are only two observations; mathematically related to CV
Average Relative Percent Difference	\overline{RPD}	$\frac{\sum_{i=2}^n RPD}{n}$	Average relative percent difference -- analogous pooled CV for duplicate measurements	Used to assess overall performance for compounds with multiple measurements
Confidence Interval	CI	$\frac{X \pm t_{(\alpha, n-1)} S}{n^{1/2}}$	Interval about X that contains the true value, with probability α	Assign confidence intervals or error bars to measurement data
Percent Recovery	R	$\left(\frac{X_{meas}}{X_{true}} \right) \times 100$	Recovery of spiked compound in pure matrix	Recovery of LCS, surrogate spikes
Percent Recovery	R	$\frac{\text{value of spiked sample} - \text{value of unspiked sample}}{\text{value of added spike}} \times 100$	Recovery of spiked compound in sample matrix	MS recovery

X = Observation (concentration)
n = Number of observations
df = Degrees of freedom, usually (n-1)
t = Statistic from Students' "t" distribution

TABLE 8-2
Flagging Conventions for Bolsa Chica -Minimum Data Evaluation Criteria for Organic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for extraction or analysis	J positive results UJ non-detects	Sample
	Holding time exceeded by a factor of two	R non-detects	
Sample Preservation SW8260A, SW8015 Modified	Sample not preserved	J positive results UJ non-detects	Sample
Sample Integrity SW8260A, SW8015 Modified	Bubbles in VOA vial used for analysis	J positive hits UJ non-detects	Sample
Temperature blank	> 6°C	J positive results (except PCBs will not be flagged) UJ non-detects (except PCBs will not be flagged)	All samples in same cooler
Initial Calibration	RRF below 0.050 (SW8260A and SW8270B)	J positive results, R non-detects	All associated samples in analysis batch
	%RSD of CCCs above 30.0% (SW8260A and SW8270B)	J positive results, UJ non-detects	
	%RSD > 20% (SW8015; SW8141A; SW8150B; SW8081), or > 15% (SW8260A and SW8270B) <u>AND</u> calibration curve not used; <u>OR</u> calibration curve used, but with coefficient of correlation or determination ≤ 0.99	J positive results, UJ non-detects	
Calibration Verification (ICV, CCV)	RRF below 0.050 (SW8260A and SW8270B)	J positive results, R non-detects	All associated samples in analysis batch
	%Drift above 25.0% (SW8260A and SW8270B) or above 15% (SW8015; SW8141A; SW8150B; SW8081)	J positive results, UJ non-detects	
Laboratory Control Sample (LCS)	%R > UT	J positive results	All samples in extraction batch
	%R < LT	J positive results, UJ non-detects	

TABLE 8-2
Flagging Conventions for Bolsa Chica -Minimum Data Evaluation Criteria for Organic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Calibration Blank Method Blank Equipment Blank Trip Blank	Convert to soil units, if applicable, multiply the highest blank concentration by 5 (10 for common lab contaminants)	U reported results < calculated value	All samples in extraction batch and/or analytical batch, whichever is appropriate associated with method blank or calibration blank All samples, same site, matrix and date (water) or all samples, same site, matrix (soil) associated with equipment blank All samples shipped in the same cooler as the trip blank
Matrix Spikes			
% Recoveries	%R > UT	J positive results	Flag matrix spike analytes in parent sample only.
	%R < LT	J positive results UJ non-detects	
RPDs	RPD > UT	J positive results	Flag matrix spike analytes in parent sample only.
Unspiked reported analytes	RPD > UT	J positive results	Flag matrix spike analytes in parent sample only.
	Compound reported in only one sample	J positive results UJ non-detects	Flag matrix spike analytes in parent sample only.

TABLE 8-2
Flagging Conventions for Bolsa Chica -Minimum Data Evaluation Criteria for Organic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Surrogates			
SW8260A	If one or more surrogates: %R > UT	J positive results	Sample
	%R <LT and > 10%	J positive results UJ non-detects	
	%R < 10%	J positive results R non-detects	
SW8270B (evaluate acid and base/ neutral surrogates separately)	If 2 or more surrogates per fraction: %R > UT	J positive results	All associated analytes (acid or base/neutral) in sample
	%R < LT and > 10%	J positive results UJ non-detects	
	%R < 10%	J positive results R non-detects	
GC Methods	%R > UT	J positive results	All analytes in associated sample
	%R < LT and > 10%	J positive results UJ non-detects	
	%R < 10%	J positive results R non-detects	All analytes in associated sample
Field duplicates	Reported in both samples, RPD > UT (30% for water; 50% for soil/sediment/tissue)	J positive results	Field duplicate pair
	Reported in one sample	J positive results UJ non-detects	
Tentatively identified compounds (TICs) in samples for GC/MS only	Reported	J reported results	All samples with TICs

TABLE 8-2
Flagging Conventions for Bolsa Chica -Minimum Data Evaluation Criteria for Organic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Presence of PCB, chlordane, or toxaphene analytes	PCB, chlordane, or toxaphene peaks coelute with some single-analyte organochlorine pesticides on either column and PCB, chlordane, or toxaphene reported	J positive results of PCB, chlordane or toxaphene R affected single-analyte organochlorine pesticide results	Sample
Confirmation (Methods SW8081, Sw8141A, Sw8150B)	RPD between primary and confirmation results > 25%	J positive results	Sample

Organic Methods include: SW8015, SW8081, SW8141A, SW8150B, SW8260A, SW8270B.

Common lab contaminants: methylene chloride, acetone, 2-butanone, toluene, and phthalates.

Spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

Number of surrogates varies with GC Method; For Method SW8081, two or more surrogates must exceed the criteria for qualification of results.

For GC Methods SW8081, SW8141A, and SW8150B, the qualification of non-detects applies to primary column tolerances (either of the two GC columns may be designated as the primary column).

Where one MS recovery meets acceptance criteria and the other MS of the pair does not, professional judgment may be used to determine if the parent sample should be qualified for matrix effects by comparing the matrix spike recoveries to other quality control results within the batch or sample site.

Qualifier may not apply in cases where a surrogate coelutes with a non-target analyte.

Qualifier may not apply in cases where low surrogate recoveries are due to sample dilution.

Professional judgment must be used in determining the effect of the bubbles on data usability; use SW-846, Update II, 9/95 for guidance.

CCV = Continuing calibration verification.

LT = Lower tolerance.

MB = Method blank.

UT = Upper tolerance.

PQL = Practical quantitation limit.

ICV = Initial calibration verification.

RPD = Relative percent difference.

%R = Percent recovery.

MDL = Method detection limit.

TABLE 8-3
 Flagging Conventions for Bolsa Chica - Minimum Data Evaluation Criteria for Inorganic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Holding Time	Holding time exceeded for digestion or analysis	J positive results R flag mercury, cyanide, and hexavalent chromium non-detects UJ non-detects for all other methods	Sample only
	Holding time for digestion or analysis exceeded by a factor of 2	R non-detect results	
Sample Preservation	Sample preservation requirements not met (If sample preservation was not done in the field , but was performed at the laboratory upon sample receipt, no flagging is required)	J positive results UJ non-detects for all methods except mercury and cyanide R mercury and cyanide non-detects	Sample
Temperature Blank	> 6°C	J mercury, cyanide, and hexavalent chromium positive results UJ mercury, cyanide, and hexavalent chromium non-detects	Samples in same cooler
Initial Calibration (Multi-point only)	Correlation coefficient ≤ 0.995	J positive UJ non-detects	All associated samples in analytical batch
Calibration verification (ICV, CCV)	%R > UT	J positive results	All associated samples in analytical batch
	%R < LT	J positive results, UJ non-detects	
Interference check sample SW6010A/SW6020 only)	%R > UT	J positive results	All associated samples in analytical batch
	%R < LT	J positive results UJ non-detects	

TABLE 8-3
Flagging Conventions for Bolsa Chica - Minimum Data Evaluation Criteria for Inorganic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Laboratory Control Sample	%R > UT	J positive results	All samples in digestion batch
	%R < LT	J positive results UJ non-detects	
Blanks: MB, ICB, CCB, Leachate Blank, Equipment Blank	Multiply highest blank concentration by 5, convert to soil units if applicable	U reported results < calculated value	All samples in digestion batch (MB); All samples in analysis batch (ICB, CCB); All samples, same site, matrix and date (water) or all samples, same site, matrix (soil) associated with equipment blank or leachate blank
Matrix Spikes	%R > UT	J positive results	All samples from same site as parent sample
	%R < LT	J positive results UJ non-detects	
	RPD > UT	J positive results	
Laboratory Duplicates	One or both sample results < 5 times the RDL and a difference of \pm RDL for water (± 2 times RDL for soil) not met.	J positive results	All samples in digestion batch
	Concentration of reported analyte > 5 times RDL in either sample and RPD > UT.	J positive results	
Dilution Test (Metals only)	If concentration is >25 times MDL and % difference >UT	J positive results UJ non-detects	All samples from same site as parent sample if analytical spike not performed
Post-digestion Spikes (Metals only)	Spike results do not indicate performance of MSA		All samples in digestion batch if MSA not performed
	%R > UT	J positive	
	%R < LT	J positive results, UJ non-detects	
MSA (GFAA only) for samples where analytical spike fails (only perform analytical spike as a result of out-of-specification serial dilution)	MSA not done	J positive results	Sample
	MSA spike levels inappropriate	J positive results	
	r < 0.995	J positive results	

TABLE 8-3
 Flagging Conventions for Bolsa Chica - Minimum Data Evaluation Criteria for Inorganic Methods

Quality Control Check	Evaluation	Flag	Samples Affected
Field Duplicates	Concentration of reported analytes are > 5 times RDL in either sample and RPD > UT (20% for water; 35% for soil/sediment/tissue).	J positive results	Field duplicate pair
	One or both sample results < 5 times the RDL and a difference of ± 2 times RDL for water (± 4 times for soil).	J positive results UJ non-detects	

Spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

CCB = Continuing calibration blank.
 ICB = Initial calibration blank.
 LT = Lower tolerance.
 MB = Method blank.
 UT = Upper tolerance.
 PQL = Practical quantitation limit.

CCV = Continuing calibration verification.
 ICV = Initial calibration verification.
 MSA = Method of standard addition.
 RPD = Relative percent difference.
 %R = Percent recovery.
 MDL = Method detection limit.

TABLE 8-4
Qualifier Flag Definitions

J	Analyte was present but reported value may not be accurate or precise.
R	This result has been rejected.
U	This analyte was analyzed for but not detected at the specified detection limit.
UJ	The analyte was not detected above the reported PQL. However, the reported PQL is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample

TABLE 8-5
QC and Field Sample Relationships

QC Samples	Tracking Parameters	Associated Field Samples
Method Blank	Analytical batch, preparation date	Samples prepared and analyzed in the same analytical or preparation batch
LCS	Analytical batch, preparation date	Samples prepared and analyzed in the same analytical or preparation batch
MS/MSD	Analytical batch, preparation date, matrix	Samples prepared and analyzed in the same analytical or preparation batch; similar matrix conditions (same soil type, site, well, boring, etc.)
Surrogate Spikes	Sample ID, location, preparation date	Sample spiked
Trip Blanks	Cooler ID, sample date	VOC samples shipped in the same cooler, containers from the same lot
Equipment Blanks	Equipment ID, sample dates	Samples collected in the same time period at the same site, similar sampling conditions (used as indicator)
Ambient Blanks	Location ID, dates, observations	Samples collected in the same area, or under similar ambient conditions
Field Duplicates/ Replicates	Sample ID, location, sampling team, matrix	Samples collected from similar conditions/matrix using similar procedures
Laboratory Duplicates	Sample ID, analytical batch, preparation batch, matrix	Samples in the same analytical or preparation batch, similar matrix conditions

ID = Identification number
LCS/LCSD = Laboratory control sample/laboratory control sample duplicate
MS/MSD = Matrix spike/matrix spike duplicate
QC = Quality control
VOC = Volatile organic compound

9.0 Data Management

9.1 Purpose and Objectives

This section describes the processes used to collect, validate, disseminate, and archive new analytical data as they are generated during the field investigation. Refer to the Data management section of the Work Plan (Section 7) for the overall data management strategy for both historical and new data. To facilitate information utilization and decision making, CH2M HILL has developed an internal set of guidelines for delivering data management services on site characterization and remediation projects that will be followed for this project.

9.2 Manage Confirmatory Sampling Data

9.2.1 Data centralization

ArcView GIS will be used to facilitate development and implementation of the field sampling plan and to display site information from the planning stages through validation. The data types that will be incorporated into the GIS database are presented in Section 7 of the Work Plan.

9.2.2 Laboratory Data

For new data being generated as part of the field effort, the data management system evolves around six somewhat overlapping phases of activity:

1. Planning: The approved SAP is used as the basis for incorporating sampling and analysis information into a sampling and analysis program independent of GIS. This program is called the Sample Tracking Program (STP) and is part of CH2M HILL's Environmental Data Management System (EDMS). EDMS is used for detailed data quality evaluation, sample receipt tracking, invoice checking, and other detailed tasks involved with "cradle-to-grave" field sample management.
2. Field work: Field efforts are carried out according to the information in the STP and GIS.
3. Sample analysis: Analyses are performed in accordance with the QAPP. Hardcopy and/or electronic data are delivered to the data management team in the agreed upon format.
4. Data handling: Hard copy and electronic data are entered into their physical and electronic placeholders, and are tracked, imported, and catalogued as appropriate.
5. Database management and data validation: The electronic data are checked for completeness and consistency with hard copy data reports. Semi-automated data validation occurs using hardcopy and electronic data. All validation flags and findings are stored in EDMS, resulting in a relational database from sample tracking through validation.

6. Data reduction: Summary statistics and data reports are generated for the project team. In addition, files are generated for downloading into GIS.

9.3 Data Filing

The following procedures for filing are followed for analytical and field data:

1. All hard copies of data are date stamped upon arrival
2. Data are logged into an internal tracking system using unique identifiers
3. Data are filed both in electronic format and hard copy according to unique identifiers

These procedures are followed throughout the project. Once the project has been completed, the data are archived. Electronic data are stored on a compact disc and hard copies are boxed and moved to storage.

10.0 Corrective Action

During the activities at Bolsa Chica, the project manager, Quality Assurance Coordinator (QAC), field managers, and sampling team members must verify that all measurement and field procedures are followed as specified in this QAPP and the work plan and that measurement data meet the prescribed acceptance criteria. If a problem arises, prompt action to correct the problem is imperative.

Analytical Request Form

Problems or questions about analytical data quality that may require corrective action are documented by the use of an analytical request form (ARF) or similar document. The QC chemist, QAC, or a data management staff member initiates the request if QC results exceed method or project criteria and a QC exceptions report or narrative is not present, if reporting or flagging errors are identified, or if requested information has not been reported. Laboratory response usually involves a written explanation of the problem or reissuing laboratory reports and/or electronic data files. If significant data quality problems have occurred and the data are critical to decision making, samples may be reanalyzed or recollected and reanalyzed. That determination must be made by the project manager.

Recommendation for Corrective Action

Significant and/or systematic deficiencies identified during audits or other independent QA reviews of field and laboratory activities will be addressed as follows:

- A Recommendations for Corrective Action (RCA) report will be completed by the QAC or auditor. The RCA must specify the problems or deficiencies that were identified, and request a timeframe for response and corrective action implementation.
- The RCA is sent to the responsible party with a copy to the project files.
- The responsible party sends a written response to the QAC indicating corrective action to be taken and the timeframe for implementation.
- If satisfactory resolution is not obtained, the RCA is transmitted to the Project Manager until a corrective action is agreed upon, or until another response is deemed sufficient.

11.0 Preventive Maintenance

The primary objective of a preventive maintenance program is to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to expected or unexpected component failure. In implementing this program, efforts are focused in three primary areas:

- Establishment of maintenance responsibilities
- Establishment of maintenance schedules for major and/or critical instrumentation and apparatus
- Establishment of an adequate inventory of critical spare parts and equipment.

These are discussed in the following subsections.

Maintenance Responsibilities

Equipment and apparatus used in environmental measurement programs fall into two general categories:

- Equipment permanently assigned to a specific laboratory (e.g., Gas Chromatography [GC] Laboratory, Gas Chromatography/Mass Spectrometry [GC/MS] Laboratory, etc.); and
- Field sampling equipment available for use on an as-needed basis (e.g., field meters, pumps, vehicles, etc.).

Maintenance of laboratory instruments is the responsibility of the laboratory contracted to perform the analytical portion of this program. Generally, the laboratory manager or supervisor of a laboratory is responsible for the instruments and equipment in his or her work area. The laboratory manager will establish maintenance procedures and schedules for each major equipment item. Although this responsibility may be delegated to laboratory personnel, the manager retains responsibility for ensuring adherence to prescribed protocol. All laboratories are bound by analytical contractual agreements to maintain the ability to produce data that meet the project objectives and to follow method specifications. This ensures that adequate spare parts, maintenance schedules, and emergency repair services are available.

Maintenance responsibilities for field equipment are assigned to the field manager and task leaders for specific sampling tasks. However, the field team using the equipment is responsible for checking the status of the equipment prior to use and reporting any problems encountered. The field team is also responsible for ensuring that critical spare parts are included as part of the field equipment checklist. Non-operational field equipment is removed from service and a replacement obtained.

All field instruments will be properly protected against inclement weather conditions during the field investigation. Each instrument is specially designed to maintain its operating integrity during variable temperature ranges that are representative of ranges that will be encountered during hot or cold weather working conditions. It is recommended, but not required, that at the end of each working day, all field equipment be taken out of the field and placed in a cool, dry room for overnight storage.

Maintenance Schedules

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each piece of equipment. Other maintenance activities are conducted on an as-needed basis. Manufacturers' recommendations provide the primary basis for established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/MS instruments, atomic absorption spectrometers, analytical balances, etc.).

Each analytical instrument is assigned an instrument logbook. All maintenance activities are recorded in the instrument log. The information to be entered includes:

- Date of service;
- Person performing service;
- Type of service performed and reason for service;
- Replacement parts installed (if appropriate);
- Date of next scheduled service; and
- Miscellaneous information.

Spare Parts

In addition to a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment down time. The inventory includes those parts and supplies that:

- Are subject to frequent failure;
- Have limited useful lifetimes; or
- Cannot be obtained in a timely manner should failure occur.

Field managers and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, an in-house source of backup equipment and instrumentation should be available.

12.0 Audits

Technical systems and performance audits are independent assessments of sample collection and analysis procedures. Audit results are used to evaluate a system's ability to produce data that fulfill program objectives and identify any areas requiring corrective action. A technical systems audit is a qualitative review of the overall sampling or measurement system, while a performance audit is a quantitative assessment of a measurement system.

Audits are conducted by a person(s) familiar with the objectives, principles, and procedures being reviewed, but who has authority to act independently. A detailed checklist is prepared for each procedure and contains items that delineate the critical aspects of the procedure under review. All observations are documented, and the checklist is submitted with a written assessment and recommendations to the Quality Assurance Coordinator (QAC), project manager, representatives of the audited sampling or analytical task, and others as appropriate. This information and any corrective action documentation are also summarized and included in project reports. Additionally, the auditor may check to ensure that personnel training and laboratory certification files are up-to-date. The project report is submitted to the regulatory agencies.

Audit records for the laboratories are reviewed by the QAC or designated staff to determine whether laboratory data will fulfill the program objectives. A systems audit for designated methods may be conducted, or additional information may be requested if data quality problems are indicated.

The following audits may be performed by the contractor and/or the regulatory agencies during Bolsa Chica activities:

- Technical systems audits may be performed for each field activity and for each analytical laboratory analyzing samples.
- One set of performance evaluation (PE) samples, single or double blind, (i.e., one performance audit) may be submitted to each laboratory performing analyses on samples for the applicable method(s). PE samples will only be submitted for methods performed on at least 50 samples, if PE samples can be purchased or prepared (e.g., particle size PE samples are not available).

12.1 Technical Systems Audit

A technical systems audit is an on-site, qualitative review of the field sampling or laboratory system. Audits are conducted, preferably at the beginning of the field or laboratory activity, by the QAC or a qualified technical staff member who has the authority to act independently of the project staff.

The technical systems audit for the laboratory results are used to review operations and ensure the technical and documentation procedures provide valid data.

Critical items for a technical systems audit of the laboratory include:

- Calibration procedures and documentation;
- Treatment and handling of standards;
- Completeness of data forms, notebooks, and other reporting requirements;
- Data review and verification procedures;
- Data storage, filing, and recordkeeping procedures;
- Sample custody procedures;
- Quality control procedures, tolerances, and documentation;
- Operating conditions of facilities and equipment;
- Documentation of staff training and instrument maintenance activities; and
- Systems and operations overview.

Critical items for a technical systems audit of the field sampling include:

- Calibration procedures and documentation for field meters;
- Complete field activity documentation in logbooks and on sampling data sheets;
- Provisions for minimization of potential sample contamination in the field;
- Proper equipment decontamination procedures;
- Proper sample collection, storage, and transportation procedures; and
- Compliance with the established chain-of-custody procedures for sample documentation and for transfer to the laboratory.

The checklist for each audit contains detailed questions pertaining to each critical item, yes/no answer blocks, and comments. A de-briefing session is held for all participants to discuss the preliminary audit results. The auditor then completes the audit evaluation and submits a Technical Systems Audit (TSA) report, including observations of strengths and deficiencies and recommendations for improvement.

If the auditor identifies procedures that could result in unacceptable data quality, he or she is authorized to stop sample collection until corrective action is taken and sampling procedures are altered.

The TSA report will be reviewed by the QAC. Copies of the report will be distributed to the QAC and the project manager. The report will be summarized in the project report that is sent to the regulatory agencies. The original TSA report, associated checklist, and other documentation are retained in the project files.

12.2 Performance Audits

Performance audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting certified samples, either single or double blind, for each analytical method and/or analytical instrument. The matrix standards are selected to reflect the concentration ranges expected for the sampling program while taking into

account any limitations of the specific analytical methods. The performance audit evaluates whether the measurement system is operating within tolerance and the data produced meet the analytical quality assurance specifications.

The PE samples are procured from an independent source and are developed from standard reference materials, National Institute of Standards and Technology (NIST) traceable materials, U.S. EPA quality control materials, or neat compounds of the highest purity available. The samples are prepared in a clean matrix or medium that allows evaluation of the analytical success of the method assuming no matrix interferences. When possible, the samples are submitted as a field sample to realistically assess the accuracy of the field samples with which they were submitted. In some cases, a PE sample may be prepared from an actual sample matrix (e.g., soil). In those cases, the QC staff coordinate with the field coordinator and vendor to ensure that representative materials are collected and provided to the vendor. The PE samples can be analyzed by independent laboratories (at additional cost) to provide confirmation of the analytes and concentrations in the prepared samples. A discussion of PE samples, their frequency, acceptance criteria and corrective action for non-compliance will be detailed in the site-specific FSPs as necessary.

Critical items for performance evaluation audits are:

- Accurate identification of the analytes included in the PE samples;
- Quantitation within acceptable limits (i.e., the manufacturer's acceptance criteria);
- Accurate reporting of results and any problems identified; and
- Acceptable analytical batch QC sample results.

These items are used to determine whether a system is operating within acceptable tolerances. Appropriate corrective action indicated by the results of a performance audit must be identified by the QAC and addressed by the laboratory. Any unresolved problems identified with PE samples must be evaluated to determine the impact on sample analyses conducted during the same time period.

12.3 Data Quality Audits (DQAs)

DQAs may be performed (by an entity independent of the laboratory) to verify whether an analytical method has been performed according to method and program specifications, and the results have been correctly calculated and reported. DQAs are modeled after those presented in the U.S. EPA Data Validation Functional Guidelines for Evaluating Inorganic (February 1994) and Organic Analyses (February 1994). DQAs involve reviewing all documentation, instrument output, and analytical reports associated with selected samples or groups of samples. Checklists are developed for each class of analytical methods (inorganics, GC, GC/MS) and used to document the audit process.

The samples or groups of samples to be audited will be selected during the planning stages of the task. Selection may focus on critical methods or samples, or a random analytical batch may be selected. A request is made to the laboratory to provide a data package containing all required information to perform the audit. The laboratory will be notified at the beginning of the field activity that a data package(s) will be requested; there may be additional costs required to provide this information.

Specific items that are reviewed during the audit are:

- Chain-of-custody records;
- Documentation of laboratory procedures (e.g., run logs, data reduction and verification);
- Accuracy of data reduction transcription, and reporting; and
- Adherence to project measurement quality objectives.

The results of all DQA activities will be reported in narratives, which supplement the checklists and data packages. Requests for additional information and any other follow-up documentation and response are also added to the DQA package. If corrective action is required based on the audit findings, the Recommendations for Corrective Action (RCA) procedure described in Section 10.0 will be followed. The DQA results will be reviewed by the QAC, and will be summarized in the project report that is sent to the regulatory agencies. Copies of the RCA report will be distributed to the QAC and the project manager. The original DQA report, associated checklists, and other documentation are retained in project files.

12.4 Recommended Audit Frequency

In addition to audits conducted for Bolsa Chica tasks, most laboratories undergo systems and performance audits conducted internally or by various state agencies and private clients. All audit results should be available for review upon request.

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