STATE OF CALIFORNIA
DEPARTMENT OF FISH AND GAME
OFFICE OF SPILL PREVENTION AND RESPONSE

LABORATORY QUALITY ASSURANCE PROGRAM PLAN

California Department of Fish and Game
Fish and Wildlife Water Pollution Control Laboratory
2005 Nimbus Road
Rancho Cordova, CA 95670

SAMPLING AND ANALYTICAL ACTIVITIES

State of California
Department of Fish and Game
Office of Spill Prevention and Response
Scientific Program
Fish and Wildlife Water Pollution Control Laboratory

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DEPARTMENTAL QUALITY ASSURANCE PROGRAM POLICY

The Fish and Game Departmental (DFG) quality assurance program describes the requirements, controls and responsibilities for implementation of quality assurance principles specified in applicable regulations, codes, and standards applied to the environmental laboratory activities. The program begins with quality assurance training for all new employees, and an orientation to the Departmental quality assurance/quality control practices. The importance of quality assurance is recognized by Department management and is documented within the Office of Spill Prevention and Response for all laboratory operations.

The primary commitment of the Departmental Quality Assurance/Quality Control Program is to implement the program activities and requirements committing time and resources ensuring that data are as precise, accurate and complete as required by the data quality objectives of the projects involved.

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3.0 QUALITY ASSURANCE DESCRIPTION

3.1 Overview

The purpose of this document is to describe the State of California Department of Fish and Game's Quality Assurance Program as implemented within the Office of Spill Prevention and Response (OSPR) Laboratories. This program plan summarizes those quality assurance and quality control (QA/QC) elements which ensure the accurate and precise development of Department sampling and analytical results, as is consistent with project objectives. The program plan has been designed to meet requirements of many projects and specifically addresses all elements of the Environmental Protection Agency Office of Environment Information "Guidance for Quality Assurance Project Plans" EPA QA/G-5, EPA/240/R-02/009 December 2002 and "Specifications for Preparing Quality Assurance Project Plans" QAMS-005/80. This plan establishes the quality assurance and quality control procedures common to most of the Laboratory services. When necessary, particular project protocols or Standard Operating Procedures (SOPs) will be used to define any project-specific requirements.

3.1.1 Department Quality Assurance System

Quality assurance is a system for integrating the quality planning, quality assessment and quality improvement efforts of various sections to enable operations to meet specified project needs. Quality assurance of field and laboratory systems is concerned with all activities that have an important effect on the quality of measurements as well as the establishment of methods and techniques to monitor the performance of these systems. In addition, quality assurance is composed of those activities performed on a routine basis to gain an independent assessment of the operation and validity of the product. In summary, quality assurance is an essential system of activities to provide the confidence that quality control methods are performing adequately.

3.1.2 Department Quality Control System

In contrast, quality control is the system of activities which provide a quality product for a data user, consisting of internal laboratory operations which document product quality.

3.2 Summary

In summary, this Quality Assurance and Quality Control Program Plan is designed to satisfy the requirements and concerns of the analyst, management, and regulatory agencies concerned with the project.

4.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

4.1 OSPR Scientific Branch Chief

The Scientific Unit Chief is responsible for administrative and financial oversight of all activities within the OSPR Scientific Unit. Organizational chart can be found in Appendix A.

4.2 OSPR Scientific Branch Environmental Program Manager (Laboratories)

The EPM is responsible for administrative and financial oversight of all activities within the OSPR Scientific Unit's laboratory system.

4.3 Laboratory Directors

Laboratory directors are designated for each of the laboratories within OSPR. The laboratory directors are accountable for all operational activities, including examination of all analytical data, quality assurance parameters, and report preparation and review.

4.4 Quality Assurance Officer (Acting)

The Quality Assurance Officer is responsible for laboratory certification, performance evaluation studies, and document control**.**

4.5 Contract Program Quality Assurance Officer

The Project Quality Assurance Officer is responsible for the evaluation of all sample logging/numbering procedures, final evaluation of quality control data for all contract projects, and preparation of QA summary reports.

4.6 Project/Section Leaders

Project Leaders are responsible for daily laboratory activities relating to their individual project assignments. Responsibilities include: making daily work assignments for laboratory staff, generation and review of data and preparation and initial review of all laboratory data reports.

4.7 Laboratory Staff

The responsibilities of the laboratory staff include sample container and glassware preparation, calibration standard and reagent preparation, sample preparation, analysis, and preparation of analytical reports with quality control data for the

project/section leaders and laboratory supervisor. Staff members will be familiar with all general laboratory procedures and quality assurance objectives.

5.0 DATA QUALITY OBJECTIVES AND ASSESSMENT METHODS

5.1 Overview

The primary data quality objective is to provide a product that fulfills all project and/or agency requirements. The requirements for projects are established prior to their commencement. In the absence of specific data requirements, standard methods or verified alternative protocols will be routinely applied.

5.2 Objectives

The data that is produced from the laboratory must be scientifically valid, defensible, comparable, and of known precision and accuracy. Objective measures of data quality such as method blanks, duplicates, spikes, standards, and recoveries will be employed. Acceptance limits will be established for data accuracy and precision. Whenever possible, statistical methods such as confidence limits, significance tests and/or variability measures will be used to evaluate precision and accuracy of data as well as conformance to acceptance limits. Corrective action will be initiated when the quality of the data does not meet established quality standards.

5.3 Standard Operating Procedures

5.3.1 Standard Quality Control Procedures

Where appropriate, standard quality control procedures, data reduction, and reporting will be in compliance with requirements in Standard Methods for the Examination of Water and Wastewater, 18th edition (1992), with requirements in USEPA Handbook for Analytical Quality Control in Water and Waste Water Laboratories: EPA-600/4-79-019, and with the requirements in USEPA Test Methods for Evaluation of Solid Waste: Physical/Chemical Methods, SW-846, 3rd edition, Update III, 1996.

5.3.2 Standard Operating Procedures

Written standard operating procedures (SOPs) for receipt of samples, tracking of custody, sample preparation and analysis, use of equipment and instrumentation shall be followed. These SOPs shall include use of standard data logging formats, logbook/worksheet entry procedures, and other written or printed documents relevant to the samples. These SOPs are available on request.

6.0 SAMPLING PROCEDURES

6.1 Objectives

The reason for sampling and the parameters of concern for each sampling event establish the requirements for sample container type and preparation, sample amount and preservation, and the sampling technique. Information on the sampling site is assembled so that a project work plan can be developed for the collection of representative samples.

6.2 Preparation for Sampling

Prior to conducting project field sampling operations, pre-cleaned sample containers and sampling devices are assembled along with the necessary equipment and portable instrumentation. A team of trained personnel with appropriate protective equipment will then conduct actual sampling using established procedures.

6.2.1 Field Quality Measures

Field quality measures such as trip blanks for water control samples, field blanks, duplicates, and background references are employed to assure data quality. Sample filtration, when required, can be performed in the field. Sample preservation is routinely provided by using sample containers with pre-added preservatives. Sampling record sheets and chain-of-custody or record-of-custody forms are completed at the time of sampling to document collection operations. Samples are carefully placed in suitable containers or coolers for prompt transportation to the laboratory. Appendix D summarizes the type of sample container and preservation methods used, as well as the maximum acceptable holding time between sampling and analysis for various types of analyses and matrices. When available and applicable, the holding times, sample container type and preservatives will follow regulatory guidance.

6.2.2 Sampling Site Identification

Sampling sites will be identified in a field logbook or project sampling form used for recording information during the conduct of sampling activities. Each sampling site will be identified by exact location, which may include address, GPS coordinates, well number, or site name. A unique sample site name and/or number is recorded in the field logbook and the sample collection form. The sample site name and/or number is also used to identify the sample on the project sampling form and chain-of-custody form.

6.2.3 Sample Container Inspection

Inspect sample containers for good closure, proper labeling, and correct number and type required for the site. Where split samples are being collected, additional containers will be needed.

6.3 Sample Collection

6.3.1 Sample Identification

When appropriate each sample will be uniquely identified by a number previously designated by the project/section officer. This number will also be used on the project sampling forms. The numbers assigned to splits, duplicate samples, and spiked samples will be coded in such a way to prevent easy identification as blind quality control samples when handled in the laboratory. Labels with adhesive backings and with the sample number on the face will be used. Extra labels will be available. Should more labels be required, they may be prepared with a permanent marking pen in the field, or a permanent marking pen may be used on the sample container. In the latter case, an adhesive label should be prepared and attached to the sample container as soon as the sample is returned to the laboratory. Ziplock bags used to carry samples will be labeled by writing appropriate identification directly on the bag using a permanent marking pen.

6.3.2 Collection of Field Replicate Quality Control Samples

Quality control criteria require that more than one set of samples be collected at a selected number of sampling events. These samples will be used to verify the consistency of results. Appropriate type and number of quality control samples will be specified with each project.

6.3.3 Field Storage of Samples

All sample containers will be kept in chilled storage in the field unless specific sampling protocol stipulates otherwise. Insulated ice chests and frozen plastic-encased coolants (Blue Ice, for example) will be used. For long term field storage of biological samples, dry ice will be used. Ice may also be used in sealed ziplock bags. The sampling team will have sufficient number of ice chests and frozen coolants to assure that samples remain chilled throughout the day. The samples must always be kept in the possession of the sampling team until they are transferred to the custody of the laboratory. Since the ice chests will have to be kept in a locked car or truck, the vehicle should be parked in the shade to the extent possible. Sampling vehicles

should use unleaded fuels. Ice chests will be cleaned with water and stored uncovered after each day. Sealed refrigerants will be washed with water and put into a freezer for reuse. The vehicle will be refilled with fuel after samples are transferred when possible.

6.3.4 Storing and Shipping Samples

6.3.4.1 Storage at the Laboratory

The samples received at the laboratory will be kept in refrigerators or freezers. Temperature will be kept as close as possible to the storage temperature required for each sample matrix and type of analysis. Generally, refrigerators will be kept at 4 +/-2 degrees C, freezers will be kept at -15 +/-5 degrees C or colder. Storage shall be in an environment where the sample identification numbers will remain attached. Mechanical refrigeration units shall be used. The use of ice as a refrigerant for sample storage at the laboratory is not allowed.

6.3.4.2 Shipping

All samples will be refrigerated or frozen during shipment through the use of ice, cold packs, or dry ice. Samples will be shipped in insulated containers. All caps and lids will be checked for tightness prior to shipping. To the extent possible, transporting vehicles will use unleaded fuel.

7.0 SAMPLE CUSTODY

The Department of Fish and Game's chain-of-custody procedures for sample tracking are initiated during the time of actual sample collection by field personnel and maintained throughout the time the samples are in their possession. Chain of custody documents must be initiated and maintained for all samples received by the laboratory.

7.1 Chain of Custody

The person responsible for sample collection must originate the chain-of-custody record. The sampler will clearly label the sample with the project name, sample location, field identification number, the date and time of sampling, and his/her own name and initials. The same information will be entered on the chain-of-custody record along with information concerning the sample type, the analyses to be performed and the sample container. The individual collecting the samples will be responsible for the custody of samples until they are transferred or properly dispatched. If samples are hand-carried to the laboratory by Fish and Game personnel, custody of samples will be transferred to laboratory staff. Shipping containers (ice chests) transported by commercial carrier will be secured with strapping tape. Documentation of the shipment will be kept with a copy of the chain-of-custody record by the person shipping the samples. The original copy of the chain-of-custody record will accompany the sample(s) when transported to a departmental or commercial laboratory.

The laboratory staff person logging the sample(s) in will carefully inspect each sample for chain-of-custody documentation, sample labeling, packing lists, and for the condition of the custody seals, sample packing materials, and the sample containers. Any discrepancies or problems associated with sample shipment will be documented on the chain-of-custody form. In the case of a discrepancy between information on the container and the COC form, the information written on the container will be used and the sample collector or project manager will be notified of the discrepancy.

After inspection, the samples will be entered into the laboratory sample receiving logbook, and will be assigned a unique sample identification number. The following information shall be included when samples are logged-in:

- Laboratory number (assigned when samples are submitted)
- Laboratory storage location (refer or freezer no.)
- Spill Title (if applicable)
- Suspects name (if applicable)
- Index-PCA code (if applicable)
- Sampler’s name, address and phone number

- Date samples received by the laboratory
- Analysis requested
- Sample identification/location
- Sample type (matrix)
- Number of containers and container type
- Sample preservation
- Required report completion date
- Signatures of person submitting samples and person receiving samples for the laboratory
- Problem description (if applicable)
- Incident location (if applicable)
- Special instructions (if applicable)

The person logging the samples in will ensure that the samples are either retained in secure storage or are given directly to an authorized analyst. A copy of the chain-of-custody form will be used to provide analysis requirements to the analyst(s). This form will accompany the sample containers and/or prepared extracts as each authorized employee performs a required task on the samples.

After all analyses have been completed and disposal of the sample is authorized, a designated sample custodian will make proper disposition of the sample with appropriate documentation. Disposal method and approximate disposal date will be noted in the laboratory log-in records. The completed chain-of-custody form(s) will be retained as a permanent part of the project record.

7.2 Sample Handling, Storage, and Holding Times

All samples will be handled, prepared, transported, and stored in a manner designed to minimize bulk loss, analyte loss, contamination or biological degradation. The sample containers will be clearly labeled with permanent marker. Soil and tissue samples for organic constituents must be frozen to prevent degradation or volatilization.

Samples will be stored for the maximum sample holding time for the required analyses as specified for the analysis. Samples which do not have a maximum holding time specified, will be stored for the duration of the research or study activity unless the sample is consumed entirely for analysis. Thereafter, the laboratory supervisor or project leader will determine if the sample will be archived.

When the holding time interval has passed and samples are approved for disposal, samples and the sample containers will be disposed of properly. It is the sole

responsibility of the laboratory personnel to ensure that all applicable regulations are followed in the disposal of samples or related chemicals. If the contracting officer should request return of a sample prior to the maximum holding time, it will be returned in a manner that meets Department of Transportation regulations.

8.0 CALIBRATION PROCEDURES AND PREVENTIVE MAINTENANCE

All laboratory instruments and equipment that are used for laboratory measurements will be maintained and calibrated for good operating conditions that meet laboratory accuracy requirements.

The calibration/maintenance techniques will be performed according to a specific calibration standard operating procedure (SOP) which has been specified by the manufacturer's recommendations, an analytical or agency requirement, or by good laboratory practices.

Analytical standards to be used for instrument calibration are obtained from sources that have demonstrated accuracy levels, and are properly stored to ensure accuracy integrity. Calibration chemicals will be logged in and assigned a specific shelf life based on chemical stability. Non-chemical standards, instruments and equipment used for calibration purposes will be re-certified per an established schedule.

Routine maintenance will be performed by qualified laboratory personnel with recommended parts and supplies kept in stock. For some items, the services of an outside vendor will be used. The correct operation of instruments/equipment repaired by vendor services will be verified prior to use on projects.

An established schedule for the routine calibration or maintenance of the instrumentation and equipment will be developed based on manufacturer's recommendations, operational experience, procedural requirements and good laboratory practices. A maintenance log-book will be established for each instrument in which all maintenance will be recorded. Calibration results, which serve as a measure of instrument condition, must be kept with the project data files.

9.0 ANALYTICAL PROCEDURES

For analysis mandated by regulatory agencies, specific methods have been designated for routine analyses with a particular range of concentrations and matrices. If methods are not stipulated, standard methods from a recognized authoritative source will be used for the tests. Analytical methods and reference sources routinely used at the Department of Fish and Game are listed in Appendix F.

9.1 Standard Procedures

All procedures developed and routinely used by the laboratory are documented with laboratory standard operating procedure (SOPs). In addition to the actual test procedure, SOPs include applicable references, acceptance limits, health and safety precautions, trouble-shooting guidelines, quality control requirements, sample preparation and documentation criteria, calculation methods and reporting protocol. Analysts are trained to perform sampling and testing tasks per the established SOP. Each analyst has access to a current copy of the procedure for the methods they perform. When it is not possible to perform an analysis per established procedures, the section supervisor is promptly notified and corrective action may be initiated.

9.2 Method Development

If a suitable standard method is not available, the laboratory technical staff will develop appropriate methods to meet the project requirements. Procedures developed within the laboratory are thoroughly validated to assure accurate, consistent results. Reference materials, replicate analyses, matrix spikes, and procedural blanks are some of the techniques applied to validate procedure development. Based on these techniques, project-specific acceptance limits can be developed, and SOPs can be written.

SOPs are reviewed and updated annually or when a change in procedure is made. The revisions are coordinated through the laboratory Quality Assurance Officer and are distributed to appropriate personnel. Prior versions of the SOPs are retrieved and archived or destroyed.

9.3 Analytical Methods

Analytical methodology described in one of the following approved methodology manuals will be used as guidelines for all analytical methods used:

- EPA Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020

- EPA Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods, SW-846, third edition, Update III, 1996

- EPA Test Methods for Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057

- EPA EMAP - Estuaries QAPP, EPA/600/x-93/xxx, May 1993

- Standard Methods for the Examination of Water and Wastewater,18th Ed., 1992

- Manual for Association of Analytical Chemists, 15th Ed.,1990

- USFWS, Patuxent Wildlife Research Center Analytical Manual (PWRCAM)

- Pesticide Analytical Manual (PAM Vol. I and II), USFDA

- Quality Assurance of Chemical Measurements by Dr. John Keenan Taylor

- Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, EPA-600/8-80-038.

Modifications of the above approved methods together with methodology developed by DFG personnel will be documented in SOPs. This methodology may be used with approval in advance, in writing, by the Contracting Officer or the Contracting Officer's Technical Representative. The limit of quantitation of any method which is used must meet a Method Detection Limit (MDL) consistent with the methodology being used. Analytical control will be maintained by strictly following written SOPs. If for some reason the SOP cannot be followed, deviations will be noted and reported in the data submital package. Deviations from the approved analytical methodology must meet requirements based on the method validation test outlined subsequently in this section.

9.4 Analytical Method Validation

9.4.1 Limit of Detection

Determine, for each method, the limit of detection (LOD), defined as the lowest concentration level that can be determined to be statistically different from a blank, and the limit of quantification (LOQ), defined as the level above which quantitative results may be obtained with a specified degree of confidence. Calculate the MDL and RL, the Federal Register, Vol. 49, No. 209, Friday, October 26, 1984 (Appendix G).

9.4.2 Standard Reference Material Test

To the extent that a standard reference material (NIST or National Research Council of Canada) can be obtained and when appropriate or required, it will be analyzed with each set of samples or every twenty samples for sets greater than twenty. To validate a new or non-standard method for limited use, comparability data will be generated.

All results must be within 65-135% of the 95% certified confidence interval for the reference materials unless otherwise stated.

9.4.3 Sample Duplicate Test

For each set of samples, analyze one sample in duplicate for each analyte in question or one duplicate for every twenty samples.

9.4.4 Spike Recovery Test

One spike recovery test will be run for each set of twenty samples for each analyte on each matrix type analyzed. For method validation, each matrix will be fortified with analyte at the reporting limit. In general, recovery values for sample spikes must be greater than 50 percent for method validation unless otherwise stated.

9.4.5 Method Documentation

Maintain a file containing all validation and modification reports. Upon completion of the validation tests, prepare a data report detailing the results.

9.5 Round-Robin Studies

The DFG laboratories will participate in round-robin studies and/or other standard reference sample programs as an on-going laboratory QC effort.

9.6 Organization of Laboratory

Qualifications of personnel is acknowledged to be very important to the laboratory. When hired, chemists and technicians must have knowledge of laboratory protocol. Such experience can be obtained from laboratory experience in another facility or satisfactory completion of suitable college course work. Anyone conducting analytical procedures in the laboratory is responsible for the accuracy of those procedures and is answerable to the Laboratory Director. New personnel will be trained by a qualified analyst and will report to the appropriate

Project/Section Leader. All personnel are expected to be familiar with and carefully follow the appropriate laboratory standard operating procedures (SOPs) developed for use in the laboratory when conducting analyses on samples received.

9.7 Laboratory Operating Practices

9.7.1 Sample Receipt

The following procedure will be followed immediately upon receiving a sample shipment:

- Record sample number in sample log book and check that the sample number is clearly marked on the sample container and on accompanying custody forms or sample worksheets.
- Record the requested analytical information in the sample log book.
- If sample is not going to be run immediately, follow appropriate sample storage procedures.
- Copies of COC will be distributed to the appropriate laboratory staff.

9.7.2 Laboratory Procedures

The individual analyst upon receiving a sample shipment will proceed as follows:

- Check the sample collection date and analysis holding time. This will determine the priority for sample preparation and analysis.
- Record date and procedure to be used.
- Record all pertinent information regarding instrument and/or materials in the lab book or on the data sheet.
- Examine glassware routinely to confirm cleanliness.
- Check to make sure that the reagents used are the correct grade and type for the analysis to be done.
- If a new lot of reagent is put into use, reagent blanks or other checks should be run to demonstrate continuity of the required quality.

- Follow designated procedure (SOP) exactly, including all quality control requirements.

9.7.3 Record Keeping

The following logs will be maintained by laboratory personnel:

- Sample log: record date of receipt, number of samples, laboratory sample number, analyses to be completed.

- Run log: record each analytical run by run number, date, and analysis. Data printouts shall be referenced Laboratory number (log-in or L#).

- Standards preparation log: record all weights and volumes used to prepare standards, solvent, source and purity or concentration of neat or concentrated standards, date prepared, final concentration, and preparer's initials. All standard storage containers will be labeled to reference the standard preparation log.

9.7.4 Reports

Procedures for analytical reporting will be as follows:

- Retain all appropriate computer printouts and strip chart recordings in a binder (be sure information includes sample number, date and time).

- Have all information clearly recorded so that a written data report can be made and reviewed.

- Final data reports shall be given to the Laboratory Director for review and signature.

9.7.5 Instruments

Instrument check procedures will be implemented as follows:

- The instrument is calibrated for the range in which work is intended. The calibration must meet required linearity or curve specifications for the method (eg. $R^2 \geq 0.995$)

- The accessory equipment (such as syringes or autosampler tubes) must be the correct ones for the instrument and method being used and they must be CLEAN.

- The reagents being used are the proper ones, and they have been checked for interferences by running a blank.

- Analytical results (raw data) are stored in hardcopy or electronically with the project data files.

- If the instrument or other equipment does not appear to be working properly, contact the section supervisor IMMEDIATELY.

9.7.6 Quality Assurance Procedure

The following quality assurance procedures form the foundation for quality control practices in the laboratory. These procedures will be practiced as a matter of routine unless superceded or modified by specific quality assurance requirements of a given project.

9.7.6.1 Chemical Analysis – General

- At least one method blank will be run for each set of samples of one matrix type to determine whether interferences are introduced. Method blanks will be run through the complete analytical method along with the sample set. Blanks shall be run with a minimum frequency of one blank per 20 samples unless otherwise specified.

- At least one sample will be fortified or one SRM of similar matrix to that of the samples will be used and run with each set of samples to measure the analytical accuracy. When appropriate, the sample will be spiked with the analyte(s) at the expected level in the sample or at mid- range of the standard curve. A minimum of one fortified sample or SRM will be run with each sample set of 20 samples or less.

- At least one sample or fortified sample will be prepared in duplicate and analyzed with each set of samples of one matrix type to measure analytical precision. A minimum of one duplicate will be analyzed for each set of 20 samples or less.

- Prepare new standards as needed according to the guidelines in section 8.0. Record all the required information in the standards preparation log. Freshly-made standards should be compared to the response of existing calibration standards or reference standards before using in order to verify the reliability of the new standard.

- When washing glassware, follow procedures outlined in method SOPs.

- All QA procedures used will be documented and stored with the results in the project files.

- Additional periodic checks of accuracy or precision will be required as deemed necessary by laboratory supervisor.

9.7.6.2 Review of Quality Control/Analytical Data

- All quality control/analytical data will be reviewed for correctness of the analytical, calibration, and data reduction procedures used, and initialed by the section supervisor, QAO or laboratory director before the accompanying data may be reported.

- If after being reviewed, a set of data are determined to be out of control, the laboratory director shall be notified and an appropriate course of corrective action will be prescribed. (See sections 11, 14, and 15 for data evaluation criteria and corrective measures.) The analyst shall keep records of the corrective measures taken. No additional analytical data will be generated until the problem has been identified and corrected.

9.7.7 Laboratory Safety

- Safety glasses will be worn in designated laboratory areas at all times. Laboratory visitors will be issued safety glasses when necessary.

- Lab coats or aprons will be worn while working with any solvents, acids, caustics, condensers, or designated instrumentation.

- Walkways, exits, and safety shower/eyewash stations will be clear of debris at all times.

- No horseplay of any kind will be tolerated.

- No food or drink will be allowed in laboratory areas.

- Additional safety and emergency procedures outlined in the Standard Operating Procedures will be followed.

- Refer to laboratory Injury and Illness Prevention Plan for additional safety measures.

9.7.8 Laboratory Cleanliness

- Glassware will be washed and prepared for use in accordance with the procedures set forth the method SOPs.

- Clean glassware will be returned to its proper place as soon as it has been cleaned appropriately and properly capped.

- Counter tops will be kept clean.

- Spills will be cleaned up immediately.

- Broken glassware will be placed in a specified container.

9.7.9 Records

Records on all relevant data are to be easily located in files that pertain to a specific analysis, question, or project.

10.0 DATA REDUCTION, VALIDATION AND REPORTING

10.1 Analysis

The conversion of raw data into functional values and the presentation of these values is a critical process in the laboratory function. In order to assure the production of data that is scientifically valid, defensible, comparable and of known precision and accuracy, the following steps are required.

10.2 Validation

Reduction of raw data is accomplished using established techniques. The calculations required to perform the reduction of raw data are performed manually or with the aid of automated data processing systems, as specified by the SOP for the particular testing method. If manual processing is to be used, the SOP will provide the calculation method and the units for reporting derived values. For automated data reduction, the accuracy of calculations will be verified through the use of standards or raw data inputs of known values.

Raw data, related quality control information and derived values are carefully evaluated prior to final reporting. The initial evaluation is performed by the analyst/specialist performing the work. Statistical methods, such as precision and accuracy acceptance limits and/or control charts, are employed to assess data acceptability. The laboratory director provides a second evaluation of the data and conclusion contained in the final report.

10.3 Reporting

Analytical reporting limits will be experimentally developed either as instrument detection limits or method detection limits (MDL). For applications requiring a greater degree of statistical confidence, the reporting limit (RL) will be used to establish a minimum reporting limit. The minimum reporting level applied will be based upon project requirements and proven laboratory capabilities.

10.4 Final Reports

The final reports contain an outline of the scope of the project, sample identification, methodologies performed, a discussion of any unusual circumstances regarding the project, and tabulated analytical results. This report is reviewed and may be signed by the analyst or when multiple analysts are involved, each analyst's initials are recorded on the report and the report is signed by the lead chemist and/or the technical reviewer and laboratory director.

10.5 Record Keeping and Maintenance

Instrument logbooks will be maintained with each instrument. A record will be made of any conditions or incidents the analyst encounters which are in any way unusual, or deviate from the SOP.

When maintenance is required, a record will be made of the symptom, the repair performed, and the individual performing the repair.

All observations, electronic records, and most printouts, and other raw material generated in the course of any analysis will be saved. They will be filed with reference to laboratory log number, date, batch number, analyst, and other information deemed pertinent. Also recorded in the laboratory notebook or bench sheets will be all weights or other types of raw data generated in the laboratory but not printed on a hard copy by the data generating device. All documentation in the laboratory notebook or bench sheets will be made in ink. Corrections to notebooks or other data records will be made by crossing a single inked line through the error, entering and initialing the correction, and recording the date.

The records to be maintained in the laboratory include such items as sample tracking records, notebooks, bench sheets, instrument read-out records, computer printouts, quality control data, and raw data. The records will be maintained for the life of the project and they will be provided to the organizations contracting for the research or studies upon request.

11.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

Quality control checks are routinely performed in the WPCL operations. These checks may be increased or modified to meet the needs of a particular analysis or project.

11.1 QA Samples

Internal quality assurance samples (fortified samples and duplicates, appropriate reference materials, duplicate samples, and method or procedural blanks) will be analyzed with each set or every twenty analyses being performed. These internal quality assurance analyses are conducted for the parameters being monitored by that analytical procedure. In addition, the compounds contained in the quality assurance sample will be representative of those compounds being monitored. Accuracy is measured by calculating percent recovery for laboratory control spikes (fortified reagent sample) and matrix spikes (fortified samples) and certified reference materials (CRMs or SRMs). Accuracy is also determined for CRMs by comparing the analysis results with the certified values. CRM results are acceptable if they are within 65-135% of the 95^{th} percentile confidence interval of the consensus values for the certified materials.

The results of all QA analyses and the percent recoveries for fortified samples and reference materials will be calculated and documented.

11.2 Duplicate Samples

One duplicate sample and/or a matrix spike duplicate or laboratory control spike duplicate will be analyzed for each set of twenty samples analyzed. The relative percent difference for each constituent is calculated as follows:

$$RPD = \{(D_1-D_2)/[(D_1+D_2)/2]\}x100$$

Where, RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value (duplicate)

The results of all duplicate determinations and the calculated relative percent difference will be reported with the data sets. For RPD, use a control limit of 25 percent unless otherwise specified by a project specific QAPP.

If either sample value is less than the MDL, the notation of "ND" (not detected) will be reported. If the precision falls outside the control limits, the analysis results will be reported with the appropriate data qualifier.

11.3 Fortified Matrix (MS/MSD) Sample Analyses

When required, matrix spike and matrix spike duplicate analyses will be conducted at a rate of five percent. The spike will be added prior to any digestion, extraction, or distillation steps as a check on the sample preparation and analysis. An amount of analyte will be added to the sample that is five to ten times the reporting limit for the analyte of interest. Recovery values are calculated as follows:

$$\text{Recovery} = [(D_a - D)/D_s] \times 100$$

Where, Recovery = Percent Recovery

D_a = Analysis value of fortified sample
D = Analysis value of sample without spike
D_s = Amount of spike added

Recovery values for fortified samples must be greater than 50 percent except where a specific method (SOP) or project specific QAPP require a different acceptable range. Exceptions shall be noted in the project specific data quality objectives. When a specific method and analyte require a different acceptable recovery range, as determined by actual spike recovery runs, the acceptable range shall be noted in the Standard Operating Procedure for that method. If the recovery falls outside of the acceptable recovery range, the analysis results will be qualified or rejected. If the results are rejected, the batch of samples associated with the rejected results may need to be re-analyzed. When sample concentrations are less than the MDL, the value of "0" will be used as the sample result concentration for purposes of calculating spike recoveries. All fortified sample results will be reported with the data package.

If the percent recovery for matrix spike is unacceptable, there might be an interference due to the matrix. The sample will be diluted to lower the interference and re-analyzed. If dilution doesn't work, the method of standard additions will be used. If matrix interference is determined to be the cause of unacceptable recoveries, the data will be qualified.

11.4 Method Blanks

Method blanks will be analyzed at a minimum of once for every batch of samples. Blank concentrations should not exceed the reporting limit for the analyte. If blank values exceed the reporting limit, the source of the contamination should be investigated and corrected, and the results associated with the contaminated blank re-analyzed or qualified. All blank analysis results will be reported with the data package.

12.0 SYSTEM AUDITS

The system audit is an on-site review which provides a qualitative appraisal of a project data set.

12.1 System Audit

The Quality Assurance Officer or person acting in that capacity conducts a QA/QC evaluation of selected project data reports prior to reporting data. This evaluation includes a review of QC data. Findings are reviewed by the laboratory director. The project lead chemist may be required to re-analyze samples associated with the unsatisfactory findings.

13.0 PREVENTATIVE MAINTENANCE

Maintenance on analytical instruments will be performed by WPCL chemists or by manufacturer's service personnel. An inventory of critical spare parts (for gas chromatographs - septa, syringes, column ferrules, backup column, etc.; for atomic adsorption spectrophotometers -- AA lamps, quartz cell, plastic tubing, etc.) will be maintained on hand.

Fume hoods will be checked quarterly. The results and the ventilation capacity across the face of the fume hood and the inspection date will be posted on an exterior wall of the fume hoods.

Equipment manuals containing trouble-shooting SOPs will be kept near the instruments.

Instrument operators are responsible for daily maintenance and for maintaining instrument logs. These logs will contain the date, operator's initials and description of routine maintenance procedures. Each entry into the run log will be initialed by the individual making the entry.

14.0 ROUTINE ASSESSMENT OF DATA PRECISION, ACCURACY, AND COMPLETENESS

14.1 Precision

Precision shall be assessed with each sample set for each analysis type. Precision will be expressed in terms of relative error as the percent deviation of the duplicate results from the original results obtained. The equation for determining precision is:

$RPD = (D_1-D_2)/[(D_1+D_2)/2]x100$

Where RPD = Relative Percent Difference
D_1 = First Sample Value
D_2 = Second Sample Value (duplicate)

14.2 Accuracy

Accuracy will be assessed on a regular basis in each set of samples for each analysis type by comparison of the analytical results of internal QA samples provided or approved by the QA Officer with accepted concentrations. Accuracy will be expressed in terms of percent recovery. Percent recovery is calculated as follows:

$Percent\ Recovery = [(D_a-D)/D_s]x100$

Where, D_a = Analysis value of fortified sample
D = Analysis value of sample without spike
D_s = Amount Spiked

14.3 Completeness

Completeness shall be assessed for each sample set and for each analysis type. The comparison for completeness will consist of a comparison of the number and type of analyses scheduled to be performed with those analyses successfully completed. Completeness shall be expressed as the percentage of analyses successfully completed relative to the number of analyses scheduled to be performed for each analysis type.

15.0 CORRECTIVE ACTION

Corrective action includes a variety of activities starting with the individual analyst applying the elements of quality control to a particular task. At this level, the corrective action takes the form of problem identification based on spike, calibration, or recovery results that exceed acceptance limits. Appropriate action taken at this stage includes checking calculations or calibrations, preparing new standards or spiking solutions, re-analyzing samples or re-extracting and re-analyzing samples. If the above actions do not correct the matter, the laboratory director is notified. Project work requiring the use of the problem method or defective instrument will be suspended until the problem has been resolved.

A review of data from reference standards, blind duplicates and standards may also indicate a necessity for corrective action. In these instances, corrective action for out-of-limit values are normally requested by the Quality Assurance Officer.

The data generated during the period of problem identification will not be reported, unless additional analysis is not possible due to restricted sample availability, or time constraints. In this case, the result will be reported with qualifications that have been clearly identified and approved by project officials.

All corrective actions are recorded in laboratory notebooks, instrument logbooks, or electronically with project data packages. These records are maintained at least eight years (unless otherwise required by project specific QAPP) in files at the laboratory. These are always available for review during external audits. These records include the analyst's comments on the corrective action such as calibrations, preparing new standards or spiking solutions, or re-analyzing samples and the results of corrective action.

The final disposition of documents is consistent with agency record-keeping procedures. Paper copies of all laboratory notebooks, benchsheets, instrument logbooks, and electronic data packages are part of the permanent archives.

16.0 QUALITY ASSURANCE COMMUNICATION WITH MANAGEMENT

Communication with all levels of management concerning quality subjects is an ongoing process and routine quality issues are communicated to appropriate levels of management. The results of performance audit evaluation sample analyses are provided to the laboratory director, as well as to the lead chemists and analysts. If significant QA problems are experienced or observed in any aspect of lab operations, the laboratory director is promptly notified.

17.0 STAFF TRAINING AND DOCUMENTATION

17.1 Hiring Process

People often begin their careers at the Water Pollution Control Laboratory (WPCL) as temporary employees. Under these circumstances, screening and reference confirmation is undertaken by the laboratory director and/or lead chemists. If/when the employee becomes permanently hired by CDFG or SJSUF, she/he completes and signs the agency employment documents, and those papers are retained in the employee's personnel file.

New employees hired by CDFG and SJSUF provide documentation of skills they already possess via publications, detailed resumes, letters of recommendation, and self-certification. Written management and/or peer performance reviews are maintained in the individual's personnel files. Every staff member is formally evaluated annually using a combination of self and supervisor evaluation. Job descriptions are reviewed and may be updated at that time.

17.2 Safety Training and Meetings

On a yearly basis, all staff attend at least one safety seminar. In addition, staff attend the laboratory's regular safety meetings. The agendas from all staff meetings which include health and safety concerns will be signed by the attendees, and copies will be retained in the safety meeting file.

18.0 Glossary

Accuracy - combination of bias and precision of an analytical procedure, which reflects the closeness of a measured value to a true value.

Bias - consistent deviation of measured values from the true value, caused by systematic errors in a procedure.

Calibration check standard - standard used to determine the state of calibration of an instrument between periodic recalibrations.

Confidence coefficient - the probability, %, that a measurement result will lie within the confidence interval or between the confidence limits.

Confidence interval - set of possible values within which the true value will lie with a specified level of probability.

Confidence limit - one of the boundary values defining the confidence interval.

Detection limits - Various limits in increasing order are:

Instrument detection limit (IDL)-the constituent concentration that produces a signal greater than five times the signal/noise ratio of the instrument. This is similar in many respects, to "critical level" and "criterion of detection." The latter limit is stated as 1.645 times the s of blank analyses.

Lower limit of detection (LLD) - the constituent concentration in reagent water that produces a signal 2(1.645) s above the mean of blank analyses. This sets both Type I and Type II errors at 5 %. Other names for this limit are "detection limit" and "limit of detection" (LOD).

Method detection limit (MDL) - the constituent concentration that, when processed through the complete method, produces a signal with a 99% probability that it is different from the blank. For seven replicates of the sample, the mean must be 3.14s above the blank where it is the standard deviation of the seven replicates. The MDL will be larger than the LLD because of the few replications and the sample processing steps and may vary with constituent and matrix.

Limit of quantization (LOQ) - the constituent concentration that produces a signal sufficiently greater than the blank that it can be detected within specified limits by good laboratories during routine operating conditions. Typically it is the concentration that produces a signal 10s above the reagent water blank.

Duplicate - usually the smallest number of replicates (two) but specifically herein refers to duplicate samples, i.e. two samples taken at the same time from one location.

Internal standard - a pure compound added to a sample extract just before instrumental analysis to permit correction for inefficiencies.

Laboratory control standard - a standard, usually certified by an outside agency, used to measure the bias in a procedure. For certain constituents and matrices, use National Institute of Standards and Technology (NIST)* Standard Reference Materials when they are available.

Precision - measure of the degree of agreement among replicate analyses of a sample, usually expressed as the standard deviation.

Quality assessment - procedure for determining the quality of laboratory measurements by use of data from internal and external quality control measures.

Quality assurance - a definitive plan for laboratory operation that specifies the measures used to produce data of known precision and bias.

Quality control - set of measures within a sample analysis methodology to assure that the process is in control.

Random error - the deviation in any step in an analytical procedure that can be treated by standard statistical techniques.

Replicate - repeated operation occurring within an analytical procedure.

Surrogate standard - a pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

Type I error - also called alpha error, is the probability of deciding a constituent is present when it actually is absent.

Type II error - also called beta error, is the probability of not detecting a constituent when it actually is present.

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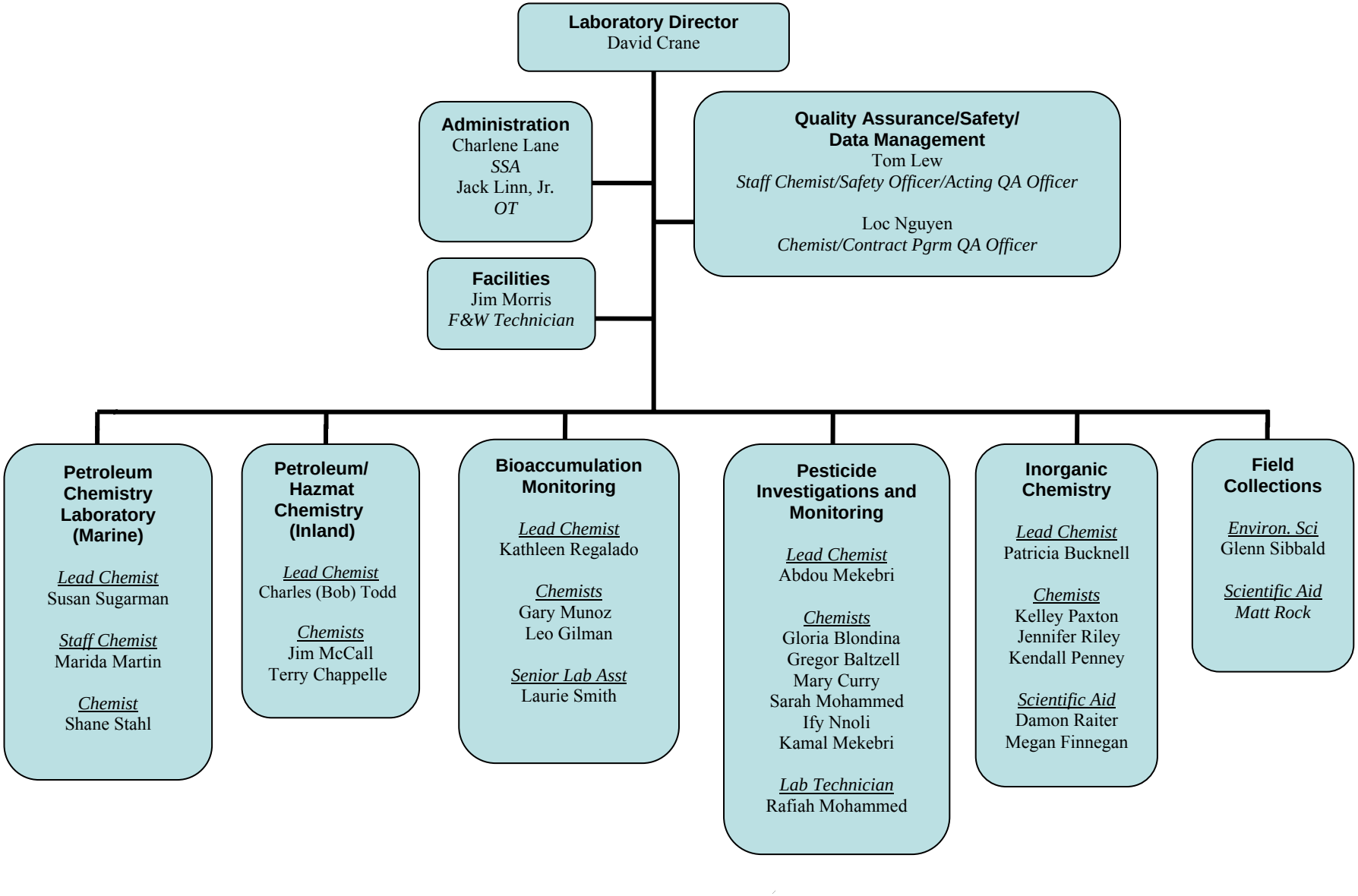
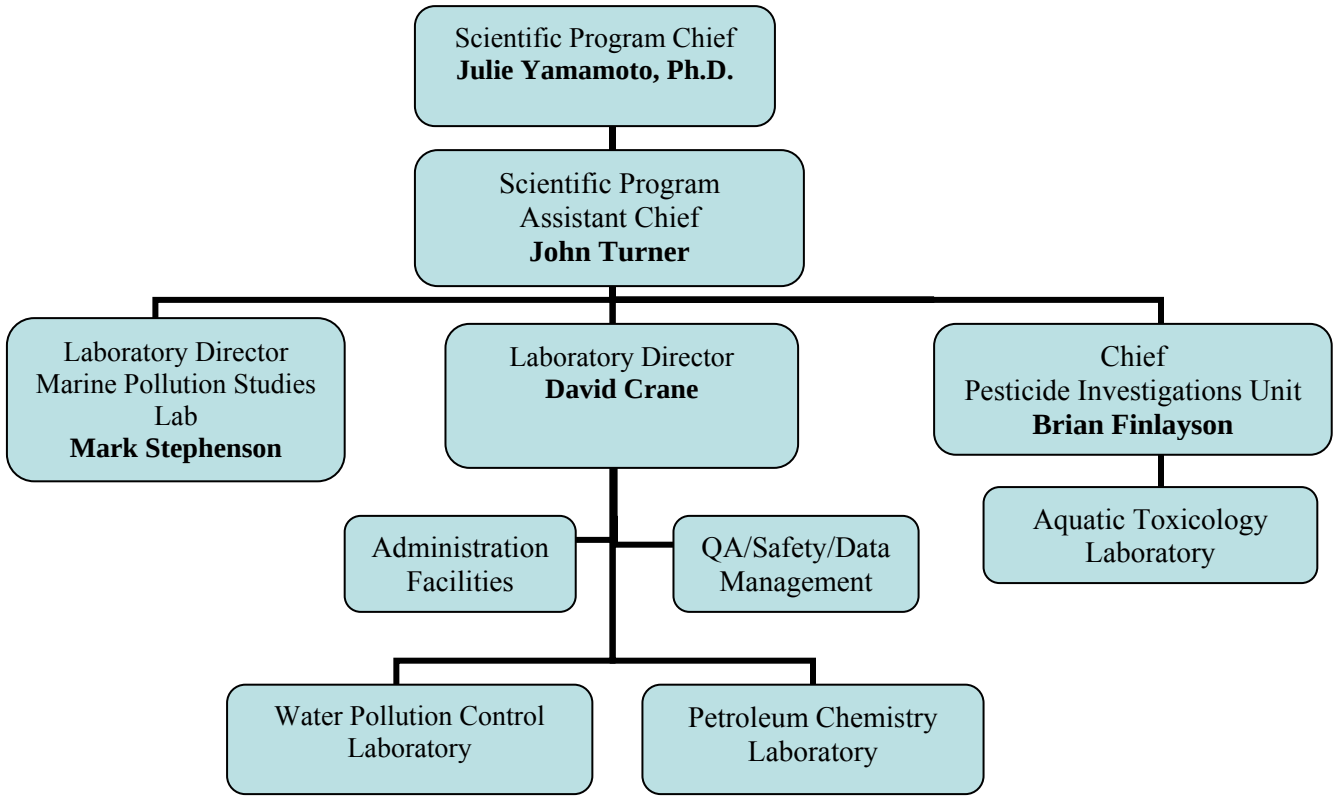
*Formerly National Bureau of Standard (NBS).

APPENDIX A

ORGANIZATIONAL CHART


Laboratory Director
David Crane
Administration
Charlene Lane
SSA
Jack Linn, Jr.
OT
Quality Assurance/Safety/ Data Management
Tom Lew
Staff Chemist/Safety Officer/Acting QA Officer
Loc Nguyen
Chemist/Contract Pgrm QA Officer
Facilities
Jim Morris
F&W Technician
Petroleum Chemistry Laboratory (Marine)
Lead Chemist
Susan Sugarman
Staff Chemist
Marida Martin
Chemist
Shane Stahl
Petroleum/ Hazmat Chemistry (Inland)
Lead Chemist
Charles (Bob) Todd
Chemists
Jim McCall
Terry Chappelle
Bioaccumulation Monitoring
Lead Chemist
Kathleen Regalado
Chemists
Gary Munoz
Leo Gilman
Senior Lab Asst
Laurie Smith
Pesticide Investigations and Monitoring
Lead Chemist
Abdou Mekebri
Chemists
Gloria Blondina
Gregor Baltzell
Mary Curry
Sarah Mohammed
Ify Nnoli
Kamal Mekebri
Lab Technician
Rafiah Mohammed
Inorganic Chemistry
Lead Chemist
Patricia Bucknell
Chemists
Kelley Paxton
Jennifer Riley
Kendall Penney
Scientific Aid
Damon Raiter
Megan Finnegan
Field Collections
Environ. Sci
Glenn Sibbald
Scientific Aid
Matt Rock


Scientific Program Chief
Julie Yamamoto, Ph.D.
Scientific Program
Assistant Chief
John Turner
Laboratory Director
Marine Pollution Studies
Lab
Mark Stephenson
Laboratory Director
David Crane
Chief
Pesticide Investigations Unit
Brian Finlayson
Aquatic Toxicology
Laboratory
Administration
Facilities
QA/Safety/Data
Management
Water Pollution Control
Laboratory
Petroleum Chemistry
Laboratory

APPENDIX B

QUALIFICATIONS AND SPECIFICATIONS OF KEY PERSONNEL
(Resumes Available upon Request)

SPECIFICATION FOR KEY PERSONNEL

Specifications for QC Officer

1. Respected person, have authority
2. Laboratory experience 5 - 10 years
3. Safety committee candidate

Specifications for Organic Laboratory Project Leader

1. Laboratory experience 5 - 10 years
2. Gas chromatography experience 2 - 3 years
3. Mass spectral interpretation experience 2 - 3 years
4. Communication skills
5. Computer skills
6. Dedicated to improvement

Specifications for Inorganic Laboratory Project Leader

1. Laboratory experience 3 - 5 years
2. Atomic absorption experience 2 years
3. Communication skills
4. Computer skills
5. Dedicated to improvement

Specifications for Chemists

1. Bachelors degree in chemistry or related sciences
2. Organic extraction experience 1 year
3. Inorganic digestion experience 6 mo.
4. Synthetic organic residue experience 2 years

APPENDIX C

SOP's
(Standard Operating Procedures for specific methods are available on request)

APPENDIX D

SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIME

Samples Analyzed for Synthetic Organics
Polynuclear Aromatic Hydrocarbons or Petroleum Hydrocarbons

Sample Type	Sample Size	Containers	Preservation	Holding Time
Water	One gallon	Glass [1,2]	4° C, pH 5-9	7 days [3]
Animal	Whole	Al foil	-20° C	6 mo.
Vegetation	One pint	Al foil	-20° C	6 mo.
Sediment	One pint	Glass [1]	-20° C	14 days [2]

1. Previously rinsed with petroleum ether and dried, with Teflon liner in lids.

2. Sample must be extracted within the specified days and analyzed within 40 days of extraction.

3. PAHs are light sensitive, therefore, sample extracts and standards must be stored in foil wrapped containers.

Samples Analyzed for Trace Elements

Sample Type	Sample Size	Containers	Preservation	Holding Time
Water	500 ml	LPE[1]	HNO_3 to pH<2	6 mo.[2]
Animal	Whole	Plastic bag	-20° C	6 mo.
Sediment	One pint	LPE[1 1]	-20° C	6 mo.

1. Previously soaked and rinsed with 1N HNO_3.

2. Six months except mercury and TBT which are 28 days.

APPENDIX E

FORMS

APPENDIX F

ANALYTICAL METHODS AND REFERENCE SOURCES

APPENDIX G

METHOD DETECTION LIMIT AND REPORTING LIMIT