

## **APPENDIX I**

### **Quality Assurance/Quality Control**

The quality assurance objectives for measurement of data are unique to the particular program for which the data are collected and utilized. They describe the overall uncertainty that the data user is willing to accept in order to make decisions for environmental or other concerns. This uncertainty describes the data quality that is needed, which are usually expressed in terms of precision, bias, representativeness, comparability, and completeness.

The participating laboratories will use approved and recognized test methods, and comply with uncertainty requirements of the method. Quality control samples are measured, uncertainties are assessed, and results must be within the range prescribed by the methods. Internal acceptance criteria are established by analyzing laboratory control samples on a daily basis. The participating laboratories will strive to meet the QA/QC goals described in this section and, therefore, be able to attest to the integrity of the sampling and analytical process.

The following QA/QC procedures will be conducted for shoreline sample collection, laboratory analyses, and data management to ensure the production of reliable and defensible data.

#### Sample Collection

Only trained laboratory staff will be assigned to collect samples using proper sampling procedures, appropriate sampling equipment, required containers, and proper preservation techniques.

General guidelines for sample collection by laboratory staff are as follows:

- Assure sterility check on sample bottles and avoid contamination.
- Label sample containers with sample date, sample time, sampling point, sample type (grab/composite), preservatives added (if needed), the name of the sampler, and analyses needed.
- Use aseptic technique when collecting samples to prevent contamination (e.g. the inner surfaces or lip edges of the bottle or cap are not to be touched).
- Avoid collecting sample in multiple sweeps and no refilling of the sample bottle.
- Once the sample is collected, immerse at least one-third of the sample bottle in ice.
- Do not exceed maximum allowable transport time (time of sample collection to sample analysis) of 6 hours.
- Once received, log the samples into the laboratory system as soon as possible, assigned a unique login number, and properly stored.
- Sample preparation steps done prior to analysis, such as sample preservation are described in individual test SOP's.

#### Sample Handling

## Chain-of-Custody

The purpose of the chain-of-custody is to establish detailed written and legal documentation of all transactions in which samples are transferred from the custody of one individual to another. The custody procedure is also used whenever samples are submitted to a laboratory within the division or to a contract laboratory. The chain-of-custody begins at the sample collection site and includes couriers or messengers who handle the sample in transit. It follows the sample in the laboratory until its ultimate disposal. It is a form of proof used to establish the authenticity and integrity of the sample, since the results will be used to show compliance with the TMDL requirements, i.e., numeric targets and wasteload allocations.

A Chain-of-Custody (COC) must accompany each sample submitted to a participating laboratory. If a COC has not been filled out prior to delivery of the sample, a form will be provided to the delivery person prior to acceptance of said sample. The COC will be reviewed to make sure that all of the needed information has been supplied. As an example, the Chain-of-Custody Form being used at EMD is attached (Appendix E).

Samples that are collected by EMD's Microbiology Unit staff for bacteriological testing are delivered directly to the microbiology laboratory. A COC sheet is not required since technically there is no sample exchange, i.e., the sample collection staff and the analytical staff are the same.

## Sample Holding & Preservation

Samples must meet EPA holding time requirements for each testing parameter. The sample refrigeration and holding time of six hours until analyses are performed are crucial for microbiological testing. Microbiological samples must be handled and stored under contamination free environments.

After the sample is received, the participating laboratory will enter the sample information into the Laboratory Information Management System (LIMS) or comparable database and a unique laboratory registration number will be generated for that sample.

## Sample Disposal

After the analyses are completed the sample will be retained as legal evidence or legally disposed of as determined by the microbiological analysis of the sample. Analyzed samples and standards used in analyses are disposed of in accordance with the laboratories written procedures, e.g., EMD's Chemical Hygiene Plan.

## Analytical Procedures

### Analyses

Analyses performed at EMD laboratories are generally driven by regulatory concerns and plant operations' requirements. There are many different analytical methods applicable to

environmental analyses. EMD's methods are generally based on those specified by EPA, Federal and State regulatory agencies, or professional organizations. As a guide, references for the microbiological procedures are listed below.

"Standard Methods for the Examination of Water and Wastewater," 18<sup>th</sup> edition, 1992, APHA, AWWA, WPCF, Washington, DC.

"Microbiological Methods for Monitoring the Environment, Water, and Wastes," EPA-600/8-78-017.

### Standard Operating Procedures (SOPs)

Routine analyses are defined in Standard Operating Procedures (SOPs), which are detailed descriptions of how to use and what to expect from a method. They contain method-specific QC criteria (i.e., instrument calibration, reagent blank, method blank, calibration standards, etc.), and QC requirements such as duplicate analysis, spike recoveries, holding time, etc. EMD follows a standardized SOP format, its content and application is presented in Appendix H of this document.

### Microbiological Analyses

The following methods and target organisms are used in analysis of shoreline samples:

- Membrane Filtration
  - Total coliform
  - Fecal coliform
  - *Enterococcus*
  
- Chromogenic Substrate
  - Total coliform
  - *E. coli*
  - *Enterococcus*

For the SMB Beaches Bacterial TMDL Monitoring Program, the chromogenic substrate method will be used in the determination of total coliforms/*E. coli*, while either the chromogenic substrate method or membrane filtration will be used for total coliforms, fecal coliforms, and *Enterococcus*.

The following QA/QC checklist is applicable for the chromogenic substrate and membrane filtration methods.

### Chromogenic Substrate

- QC Checks on Idexx Reagent
  - Colilert-18 and Enterolert –sterility check performed with each use; autofluorescence, positive and negative controls; performed on each new lot of reagent

- Monthly QC verification of at least 10 positive wells/target organism
- Quanti-trays:
  - Leak test performed on each new lot of trays
- DI Water
  - Sterility check performed with each autoclaved batch
  - Heterotrophic plate count performed monthly
  - Amm-N, Org-N, and TOC performed monthly
  - Heavy metals, total and single, performed annually
  - Total chlorine performed with each use
- Equipment and Laboratory Environment:
  - Incubator temperatures checked twice daily (morning and late afternoon)
  - Refrigerator temperatures checked twice daily (morning and late afternoon)
  - Thermometers calibrated semiannually
  - Autoclaves calibrated semiannually; preventative maintenance performed quarterly
  - Air and Rodac testing for laboratory air and surface environments performed monthly.
  - Balances calibrated semiannually; weight check with each use
  - PH meters- calibrated semiannually; standardized with each use
  - Quanti-tray sealers checked and cleaned weekly
- Personnel QA checks
  - Reagents blanks
  - Sample duplicates (done on 10% of the samples per month)
  - Standard sample analysis and comparison count performed monthly

## **Membrane Filtration**

- QC Checks on Media (mEndo, mFC, mE, EIA; phosphate buffered water):
  - mEndo, mFC, mE, EIA: pH, sterility check and positive, and negative controls with each new batch
  - Phosphate buffered water: pH and sterility check with each new batch
  - Monthly QC verification of at least 10 positive colonies/target organism
- Equipment and Laboratory Environment:
  - Incubator temperatures checked twice daily (morning and late afternoon)
  - Refrigerator temperatures checked twice daily (morning and late afternoon)
  - Thermometers calibrated semi-annually
  - Autoclaves calibrated semi-annually; preventative maintenance performed quarterly

- Air and Rodac testing for laboratory air and surface environments performed monthly.
  - Balances calibrated semi-annually; weight check with each use
  - PH meters- calibrated semi-annually; standardized with each use)
  - Residue on glass- performed annually for glassware and Petri dishes
- Personnel QA checks (performed by all technical lab staff)
- Reagents blanks
  - Sample duplicates (done on 10% of the samples per month)
  - Standard sample analysis and comparison count performed monthly for MF analysis:

## **System and Performance Audits**

An audit is a periodic check to ensure that the laboratory operates according to the policies and procedures described in the Quality Assurance Manual, complies with good laboratory practices, and meets the requirements of regulatory agencies. It may be either a system or a performance audit.

### System Audit

A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data. It is an on-site inspection of the laboratory's system of operations. It may be an internal or external audit. Internal inspections may be performed by quality assurance personnel. External audits are generally laboratory certification-related activities.

#### 1. Internal

Periodically, the QA Officer (or designee) audits the laboratories and reports the results to the Division Manager (or laboratory director), laboratory managers, and unit supervisors.

#### 2. External

State-certified laboratories are site visited every two years by auditors from the Environmental Laboratory Accreditation Program (ELAP) of the California Department of Health Services (CA DOHS). Accreditation is by scientific discipline or field of testing. Non-compliances with good laboratory practices are identified and reported as deficiencies and are subject to corrective action before accreditation is renewed.

## Performance Audit

A performance audit is a review to evaluate the laboratory's analytical activities as well as the data produced by analysts. It verifies the ability of the laboratory to correctly identify and quantify compounds in unknown samples submitted by the auditing entity. The purpose of these audits is to determine the laboratory's capability to generate scientifically sound data.

### 1. Internal

Periodically, the QA staff submits unknown samples to most of the laboratories. These samples are usually from the inventory of previous Performance Evaluation (PE) samples from EPA. Analysis of these samples is also a corrective action requirement for Discharge Monitoring Report (DMR) and/or Water Pollution (WP) samples evaluated with "unacceptable results." The QA staff may also conduct intra- and inter-comparison studies.

### 2. External

All laboratory units, including the Microbiology laboratory, at EMD participate in mandatory QA Performance Evaluation (PE) Study Programs.

#### a. Mandatory PE Programs

- \* Water Pollution QA Study Program (WP) serves a dual purpose. It satisfies EPA's wastewater testing laboratory requirements and meets one of ELAP's laboratory certification criteria. Test samples are analyzed for parameters listed under each field of testing on our certifications and are specified in the WP Program following certified procedures. A laboratory can participate in a WP Study twice a year.
- \* For the Microbiology Performance Evaluation (PE) Study, Drinking Water/Wastewater Enumeration is required for ELAP certification. Like all the other PE programs, the samples are acquired from NIST-approved vendors and analyses are done for certified analytes.

#### b. Voluntary PE Program

The Microbiology Unit also takes part in the interlaboratory calibration studies with EPA. These programs are performance based.

## Assessment of Precision and Accuracy

Data quality may be assessed in terms of precision, accuracy, representativeness, completeness, and comparability. The latter three are usually determined outside of the laboratory operations and with limited involvement of laboratory staff. These measures are not included in this section. The internal quality control measures (i.e., precision and accuracy) that are performed in the laboratory to evaluate data quality are described in this section.

### Precision

Precision is the agreement among a set of replicate measurements without knowledge of the true value. It is the degree to which a measurement is reproducible. Precision, expressed as Relative Percent Difference (RPD), is determined for each laboratory unit by analyzing replicates of the same sample, a number of duplicate pairs, or matrix-spiked duplicate samples.

### Accuracy

Accuracy is a measurement of how close the result is to the true value. Each laboratory unit establishes its accuracy of measurement by analyzing QC check samples (spiked samples, standard reference materials from a reliable source, etc). The results of the QC samples are correlated to documented, certified values. Results of spiked samples are calculated as Percent Recovery. Actual Percent Recovery is compared to established reference data. The degree of closeness of the QC check sample contributes to the general assurance that the accuracy of the data is within acceptable limits.

## Corrective Action

Laboratory events and data that fall outside established quality acceptance criteria may require investigation or corrective action. The corrective action implemented depends on the type of analysis, the extent of the error, and whether the error can be determined and corrected. The purpose of the corrective action is to resolve the problem and to restore the system to proper operation. Investigative steps and corrective actions implemented are documented.

### Corrective Action Procedures

1. The initial corrective action procedures may be handled at the bench level. The unit supervisor is immediately notified of the deviation. The analyst reviews the sample preparation for possible errors and checks the instrument calibration, calibration and spike solutions, instrument sensitivity, etc.

2. If the error cannot be resolved by the analyst, the unit supervisor has the responsibility of resolving the problem with assistance, if needed, from the laboratory manager and/or the QA Officer.
3. The corrective action adopted may be determined by the analyst, the unit supervisor, the laboratory manager, the QA Officer, or through a consensus. If needed, the final decision for corrective action rests with the laboratory manager after consultation with the QA Officer.
4. The unit supervisor shall maintain an accurate and up-to-date record of corrective actions taken in the unit. A corrective action report form (included herein as an attachment) is available for use.
5. The laboratory manager shall periodically review corrective action records and plan for system improvement by involving analysts, unit supervisors, and QA personnel.

#### General Guidelines for Initiating a Corrective Action

1. Identify/define the problem.
2. Assign responsibility for investigating the problem.
3. Investigate and determine the causes.
4. Develop corrective action to eliminate the problem.
5. Measure the effectiveness of the corrective action.
6. Analyst, unit supervisor, laboratory manager, and the QA Officer meet to review and evaluate the process, if necessary.
7. Document the process by filling out the Corrective Action Report Form.

