

STANDARD OPERATING PROCEDURE  
***Request for Method Development and the Laboratory Specifications Form***

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**KEY WORDS**

QC, MDL, RL, method development, storage stability, spike, blank, confirmation, COC, budget.

**APPROVALS**

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Environmental Monitoring Branch organization and personnel, such as management, senior scientist, quality assurance officer, project leader, etc., are defined and discussed in SOP ADMN002.

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## **1.0 INTRODUCTION**

### **1.1 Purpose**

This standard operating procedure (SOP) discusses documenting analytical guidelines for Environmental Monitoring Branch studies. These guidelines may include some or all of the following: method development, method validation, continuing quality control, reporting procedures and budget.

### **1.2 Definitions**

Quality assurance (QA), quality control (QC) and method development terminology are defined in QAQC001.00.

## **2.0 PROCEDURES**

### **2.1 Request for Method Development or Method Modification**

- 2.1.1 Upon initiating a study and prior to writing a protocol, contact the Environmental Monitoring (EM) QA officer/laboratory liaison (liaison) to find out if methods are available for the expected analyses to be performed and to see if the laboratory has the capacity to conduct the analyses.
- 2.1.2 The attached Analysis Request Form (Attachment 1) may be used as a reference to determine what kind of information the liaison may need to begin consulting with the lab. The form can be filled out and given to the liaison. If method development is necessary, ample time must be given to the lab to validate the method. This may take as little as one month. However, usually more time is generally needed to develop difficult methods and to fit development into the lab's schedule.
- 2.1.3 Lab availability for method development and analysis is based on decisions made at supervisor priority meetings and in consultation with the laboratory supervisor. The project leader will be informed of lab availability by the lab liaison.

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#### **2.2 Laboratory Specifications Sheet**

- 2.2.1 The EM lab liaison shall negotiate with the project leader and the chemistry laboratory supervisor all requirements necessary to perform analyses for a study.
- 2.2.2 Attached is an example of an EM Analytical Laboratory Specifications Form (Attachment 2). This form is used by the liaison to document requirements for analysis. Some or all of the guidelines included in this form may be required depending on the specifics of the study. The Laboratory Specifications Form may be modified to suit the needs of the study.
- 2.2.3 Once the requirements are described and the project leader has approved them by signing the Lab Specifications Form; the liaison and the chemistry supervisor must each sign the form. The signed form will be maintained in the lab liaison study file for future reference. The chemist keeps a copy as a reference during the study. Any modifications of the original requirements must be approved by the project leader, laboratory supervisor and initialed by the QA/lab liaison on the form. A copy of the lab specifications form, with changes should be sent to the project leader.

#### **3.0 STUDY-SPECIFIC DECISIONS**

##### **3.1 Method Detection Limit (MDL) and Reporting Limit (RL) Determination**

- 3.1.1 SOP QAQC001.00 describes MDL determination procedures and calculation. The SOP also explains RL determination. For every new analyte and matrix combination needed for a particular study, the spike level and the number of repetitions, and matrix should be written on the Lab Specifications Form in the MDL determination section as specified in SOP QAQC001.00.
- 3.1.2 If the method is complete (ready for use) for the analyte of interest, "complete" is written in the MDL determination section. Due to time constraints oftentimes the method Standard Operating Procedure is written by the lab after analysis of samples has begun. If necessary, the laboratory can provide a summary of the method to a project leader. If the method SOP is completed prior to initiation of the study, the lab liaison can provide a copy when requested.

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#### **3.2 Validation of the method**

- 3.2.1 The validation procedure is described in QAQC001.00. The spike levels (at minimum 3 levels), and number of repetitions (at minimum 3 repetitions) must be written on the form. If validation for the analyte/matrix combination is complete, "complete" can be written in this section of the form.

#### **3.3 Continuing Quality Control**

- 3.3.1 General continuing QC and optional QC are described in QAQC001.00. Some of the optional QC are similar to QC procedures that are a standard practice at laboratories. The lab liaison needs to be familiar with the lab's standard QC procedures by requesting the laboratory's SOPs for QC. If optional QC is needed for a particular study, the procedures must be stated on the Lab Specification Form.
- 3.3.2 At minimum the number of matrix spikes per extraction set, and whether they are duplicate (spiked at same level) and spike level should be written on the form. The type of matrix, and the matrix location should be specified (e.g. American River water, Auburn ground water).
- 3.3.3 If validation for the analytical method has been previously completed, the control limits that were based on the validation recoveries should be inserted on the continuing QC page or attached to the Lab Specifications Form. Control limits are also defined in QAQC001.00.

#### **3.4 Confirmation method**

- 3.4.1 If a confirmation analysis, or a confirmation instrument is necessary for a project or program, then the lab liaison must be notified. The same procedures for MDL determination and validation must be followed for the confirmation method. Analytical confirmation is described in QAQC001.00.
- 3.4.2 The lab can develop a confirmation method, or confirm with another instrument, as they deem necessary if the need for a confirmation is not specified in the Lab Specifications Form.

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#### **3.5 Storage Dissipation or Stability Study**

- 3.5.1 A storage dissipation study should be designed for any new matrix/analyte combination unless the stability is well documented. The storage stability study is described in QAQC001.00. The number of replicates, spike level, analysis intervals and length of the study should be noted on the Lab Specification Form. All other specifics such as type of bottle, pH, and temperature should also be noted on the form.

#### **3.6 Reporting requirements**

- 3.6.1 Reporting requirements and additional specifications are to be written in the reporting procedures section of the Laboratory Specifications Form. Reporting requirements could include any QA requirements, number of significant figures, reporting of trace detections and reporting units.

#### **3.7 Turnaround time**

- 3.7.1 Turnaround time can include date MDL and validation must be completed, maximum number of days prior to sample extraction, and maximum time allowed to report results to the lab liaison. All turnaround deadlines begin at receipt by the lab of either the laboratory specifications sheet for MDL and validation, or by the receipt of samples for extraction and data reporting. All turnaround times must be noted in the lab specifications sheet. If the laboratory cannot meet the deadlines listed in the lab specifications sheet it must be reported to the lab liaison. The lab liaison must report delays to the project leader.
- 3.7.2 The MDL and validation must be completed by the time a study is set to begin. The laboratory should be consulted to determine how long method development might take. The amount of time varies based on chemist and instrument availability, the chemistry of the analyte and priorities set by Environmental Monitoring supervisors.
- 3.7.3 The storage time prior to extraction depends on the stability of the analyte. For stable analytes, the extraction deadline can be expressed as a number of business days and depends on lab availability and the needed data turnaround time. Generally stable surface water analytes are given 10 business days and for stable

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ground water analytes the lab is given 10 to 20 business days to extract by. Unstable analytes must be extracted within 1 to 3 business days. When samples need to be analyzed within 24 hours the project leader must ensure samples will be delivered to the lab as soon as possible after sampling and early in the week so that samples can be extracted before a weekend. Samples should be collected early in the day if same day extraction is needed.

- 3.7.4 Data reporting turnaround time depends on the needs of the project leader and laboratory workload. Generally the lab liaison should receive the data by 4 to 8 weeks. Rush turnaround times can be requested.

### **3.8 Budget**

- 3.8.1 The budget section is optional. It is useful for both the liaison and the branch's accounting purposes.

# Analysis Request Form

Attachment 1.

A laboratory specification sheet will be developed by the lab liaison from information supplied on this form.

*If method validation has not been completed for a particular analyte/matrix combination, then a new set of validation samples will need to be analyzed. The usual minimum time for this process is one month and can be much longer for difficult analytes or matrices. To expedite your study, this form needs to be filled out promptly. Then you will be given the best estimate of when the lab will be ready to accept your samples.*

Project Leader: \_\_\_\_\_

Date: \_\_\_\_\_

Phone number: \_\_\_\_\_

Description of Project/Study: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Time frame for sampling: \_\_\_\_\_

---	Matrix 1	Matrix 2	Matrix 3	Matrix 4	Matrix 5	Matrix 6
Matrices						
Analytes or Screen*						
Number of samples (Per week, month, event etc.)						
Any Field or Rinse Blanks?						
Expected concentrations						

\* Please list all analytes that should be included in the selected screen. May be written in comment section below.

Comments and Concerns (incl. QA or QC, turnaround time etc.): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Approved by: \_\_\_\_\_  
lab liaison

Date: \_\_\_\_\_

**CALIFORNIA DEPARTMENT OF PESTICIDE REGULATION  
 ENVIRONMENTAL MONITORING PROGRAM  
 ANALYTICAL LABORATORY SPECIFICATIONS**

Project No. \_\_\_\_\_  
 Lab Project Manager \_\_\_\_\_  
 Project Chemist \_\_\_\_\_  
 EM Project Manager \_\_\_\_\_  
 EM Lab Liaison/ QA Officer \_\_\_\_\_

Lab \_\_\_\_\_  
 Phone \_\_\_\_\_  
 Phone \_\_\_\_\_  
 Phone \_\_\_\_\_  
 Phone \_\_\_\_\_

**Type of Analysis:**

Sample Type	Analysis For	Reporting Limit	Number of Samples
1	_____	_____	_____
2	_____	_____	_____
3	_____	_____	_____
4	_____	_____	_____

Methods Development: \_\_\_\_\_  
 Sample Storage: \_\_\_\_\_  
 Sample Extraction: \_\_\_\_\_  
 \_\_\_\_\_  
 Analytical Standard Source: \_\_\_\_\_  
 Instrumentation: \_\_\_\_\_  
 Confirmation Method: \_\_\_\_\_  
 Continuing QC: \_\_\_\_\_  
 Sample Disposition: \_\_\_\_\_  
 Extract Disposition: \_\_\_\_\_  
 Reporting/Turnaround: \_\_\_\_\_  
 Cost of Analysis: \_\_\_\_\_

**Other Specifications:**

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**Approval:**

\_\_\_\_\_  
 Project Leader: \_\_\_\_\_ Date \_\_\_\_\_

Note: all modifications after this document is signed, must be approved by all parties and initialed by lab liaison.

\_\_\_\_\_  
 CDPR Lab Liaison \_\_\_\_\_ Date \_\_\_\_\_ Lab Representative \_\_\_\_\_ Date \_\_\_\_\_



**CALIFORNIA DEPARTMENT OF PESTICIDE REGULATION  
ENVIRONMENTAL MONITORING PROGRAM  
ANALYTICAL LABORATORY SPECIFICATIONS**

**METHODS DEVELOPMENT AND VALIDATION**

The laboratory shall determine a method detection limit (MDL) and a reporting limit (RL) for each analyte. The laboratory shall also document their terms, definitions and procedures for determining MDL and RL in their approved analytical method. *The laboratory shall provide at least a draft copy of the analytical method before analyzing any field samples.*

**Method Detection Limit Determination**

Sample matrix: \_\_\_\_\_  
 Analyzed for: \_\_\_\_\_  
 Target Reporting Limit: \_\_\_\_\_  
 Other specifications: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Proposed Levels		
Matrix	Spike level	# Reps
1	_____	_____
2	_____	_____
3	_____	_____
4	_____	_____
5	_____	_____

**Reporting Limit Determination**

Reporting limit (RL) is decided by the laboratory. The RL is 1 to 5 times the MDL depending of the the analytical method and matrix.

**Validation**

Method # \_\_\_\_\_  
 Sample Matrix: \_\_\_\_\_  
 Analyzed For: \_\_\_\_\_  
 Reporting Limit: \_\_\_\_\_  
 Other Specifications: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Levels		
Sample Type	Spike Level	# Reps
1	_____	_____
2	_____	_____
3	_____	_____
4	_____	_____
5	_____	_____

**Control limits for continuing QC are established with validation recovery data at +/- 2 X the standard deviation for each analyte. *(Some analytes are at +/- 3 X, see QAQC001.00 for details)***

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CONTINUING QUALITY CONTROL

Blanks 1 blank matrix per extraction set

Reagent or Solvent Spikes \_\_\_\_\_

Matrix Spikes 2 matrix spikes per extraction set (duplicate)

Matrix	_____	Spike Level
Matrix	_____	Spike Level
Matrix	_____	Spike Level
Matrix	_____	Spike Level

Notes: \_\_\_\_\_

Replicate Matrix Analyses \_\_\_\_\_

Replicate Extract Injections \_\_\_\_\_

Confirmation Analyses \_\_\_\_\_

Other QC \_\_\_\_\_

If validation is complete, insert control limits here:

**QC Reporting Requirements:** Extraction date, sample numbers in set, report date, method SOP #, matrix spiked, analyte spiked, amount spiked, results, % recovery, R.L. and units.

**QC assessment:** Spike recoveries will be assessed based on control limits according to QAQC001.00 and based on an RPD limit of 30% between matrix spike duplicates. Problems with QC or the method should be brought to attention of the lab liaison.

Storage Dissipation Study \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**REPORTING PROCEDURES**

**Samples to be analyzed:**

Primary Samples	All primary samples to be analyzed
Backup Samples	Back-ups to be analyzed as needed
Field Blank Samples	A small number of blanks will be submitted for analysis

**Completing the Chain of Custody Record:**

1. Sign and date the box marked "Received for Lab by:".
2. Write in the Lab I.D. number in the appropriate space.
3. Results should be reported as follows:

*All water samples to be reported in ppb.*

*For surface water samples traces will be reported unless otherwise noted.*

*For all other matrices, no traces will be reported unless noted here.*

4. For those samples which contain no detectable amount write "none detected" or "ND" and indicate the reporting limit.
5. The chemist who analyzed the sample should sign and date in the appropriate space.
6. Write in the date of extraction and analysis in the appropriate space.

**Turnaround Time:** \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**Additional Specifications:** \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

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ENVIRONMENTAL MONITORING PROGRAM  
ANALYTICAL LABORATORY SPECIFICATIONS

BUDGET

Contract #: \_\_\_\_\_

Analysis	Number of Analyses	Cost per Analysis	Cost

**Total Cost =** \_\_\_\_\_

**Please send all reports and invoices to:**  
  
California Department of Pesticide Regulation  
3971 Commerce Drive  
West Sacramento, California 95691