STANDARD OPERATING PROCEDURE
**Chemistry Laboratory Quality Control**

## KEY WORDS

QC; method detection limit; MDL; reporting limit; RL; confirmation; method development; method validation; storage stability; split; spike; blank; laboratory specifications.

## APPROVALS

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## 1.0 INTRODUCTION

### 1.1 Purpose

This Standard Operating Procedure (SOP) discusses the chemistry laboratory quality control (QC). These guidelines describe method development as well as continuing quality control procedures that should be followed for all Environmental Monitoring (EM) Branch studies.

### 1.2 Definitions

1.2.1 **Analytical Confirmation** refers to an analyte that has been unequivocally identified. For an analytical method that is nonspecific (e.g., gas chromatography with a flame photometric detector) analytical confirmation requires a second analysis that has a change in both the separation and detection principle. An analytical method that is specific (e.g., mass spectrometry) meets the analytical confirmation criterion and a second analysis is not required.

1.2.2 **Blank** refers to a sample with no detectable amount of pesticide. Blanks are used to check for contamination or to prepare QC samples (e.g., **blank-matrix, reagent blank,** and **field blank** samples).

1.2.3 **Blind Spike** refers to a blank-matrix sample, which has been spiked and submitted to the lab disguised as a field sample.

1.2.4 **Extract** refers to the final solvent, which contains the pesticide residue.

1.2.5 **Extraction Set** refers to a single group of samples extracted and processed at the same time.

1.2.6 **Instrument Detection Limit (IDL)** is 1-5 times the signal-to-noise ratio depending on the analytical methods.

1.2.7 **Method Detection Limit (MDL)** refers to the USEPA definition (40CFR, Part 136, Appendix B). “The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix….”

1.2.8 **Reporting Limit (RL)** is 1 - 5 times the MDL depending on the analytical method and matrix. The MDL can vary from sample to sample because of matrix effects. Ideally, the RL will not change, will be set high enough to account for matrix effects, yet low enough to be useful.

1.2.9 **Spike** refers to a known amount of pesticide added. These QC samples are used to check the precision and accuracy of a method.

1.2.10 **Split** refers to one homogeneous sample divided into several aliquots, with the different aliquots analyzed by different laboratories. These QC samples are used to check the specificity and precision of a method.

1.2.11 **Standard** refers to the laboratory analytical standard.

## 2.0 GENERAL PROCEDURES

These guidelines are meant to be a starting point; a specific study may require more or less QC than is given here. The procedures outlined here are the QC measures which should be reported. Performing other QC procedures such as frequency of standard injections and calibrations are left to the chemist's discretion.

### 2.1 General Method Development

Many times the method development will be a negotiation between the project leader and the laboratory. The project leader can suggest some method performance goals (e.g., specificity, reporting limit, etc.), but the goals need to be balanced with laboratory cost and time constraints. The method performance should be consistent with the study objectives.

2.1.1 **Standard** – Standard solutions should be validated prior to use by checking for chromatographic purity or verification of the concentration using a second standard prepared at a different time or obtained from a different source.

2.1.2 **Method Detection Limit Determination** - The MDL is determined by the USEPA method (40 CFR, Part 136, Appendix B). The complete procedure is given in Appendix 1. Briefly, the MDL is determined by analyzing at least 7 low-level matrix spikes (generally 1 - 5 times the IDL) and performing the following calculation:

$$MDL = t \times S$$

Where:

t = Student's t value for 99% confidence level (single-tailed) and n-1 degrees of freedom
S = standard deviation

2.1.3 **Reporting Limit Determination** - The RL is determined by the chemist and set at 1 - 5 times the MDL depending on the matrix and instrument.

2.1.4 **Method Validation** - At the onset of a study, an acceptable range of spike recoveries will be established. This range will be established by analyzing blank-matrix spike samples. Two to five replicate analyses at two to five different spike levels will be used to determine the mean percent recovery and standard deviation. Number of replicates and spike levels will be chosen by the EM Quality Assurance (QA) officer. Warning limits will be established at the mean percent recovery plus/minus 1 - 2 times the standard deviation. Control limits will be established at the mean percent recovery plus/minus 2-3 times the standard deviation. Any subsequent spiked samples outside the control limits may require the set of samples associated with that spike to be reanalyzed.

2.1.5 **Storage Stability** - Storage stability needs to be evaluated on a case-by-case basis, so no specific test design is specified. However, in general the test should be run for the longest anticipated holding period, with at least four sampling intervals and two replicate samples at each sampling interval. Other factors may also need to be incorporated into the storage stability tests, such as pH, temperature, and container type. The project leader is responsible for specifying the design of the storage stability test.

## 2.2 General Continuing QC

These analyses are to be done by the main lab on a continuing basis. Each extraction set should consist of 5-20 actual samples. Exact frequency of QC analyses and spike levels are chosen by the project leader.

2.2.1 **Reagent Blanks** - 1 - 2 per extraction set

2.2.2 **Blank-Matrix Spikes** - 1 - 3 per extraction set

2.2.3 **Analytical Confirmation** - 0 to100% (normally 10%) of positive samples confirmed

2.2.4 **Split Matrix Samples** - 0 to 100% (normally 10%) of the actual samples should be split into two aliquots, one aliquot analyzed by the main lab, and one by the QC lab. For studies that cannot have actual samples split or for which only a few positives are anticipated, blind spike samples may be used.

2.2.5 **Blind Spikes** - 0 to 100% (normally 10%) of the actual samples should be accompanied by laboratory-spiked samples disguised as real samples. These should be done only for matrices that can be accurately spiked.

## 2.3 Optional Continuing QC

The following analyses should be considered but may not be routinely performed unless specified by the project leader.

2.3.1 **Internal Standard** - a chemical not expected in the samples can be spiked into all samples or extracts. This is particularly useful for quantifying mass spectrometry data.

2.3.2 **Replicate Sample Analyses** - analyzing multiple aliquots of a single sample will give a better estimate of the method precision.

2.3.3 **Replicate Extract Analyses** - multiple analyses of a single extract will give a separate estimate of the precision of the extraction and analysis processes.

2.3.4 **Split Extract Analyses** - analyzing a single extract with more than one lab is useful for checking discrepancies between laboratories.

2.3.5 **Reference Material** - a stable sample that contains the analyte(s) of interest and has been analyzed many times so that the concentration(s) are known. Analysis of this material may give a better estimate of the method's accuracy than spiked samples. Also useful for method development.

2.3.6 **Standards Exchange** - exchanging analytical standards between the primary and QC lab is useful for checking discrepancies in split samples.

## 3.0 GROUNDWATER STUDY QC PROCEDURES

### 3.1 Groundwater Study Method Development

The general method development procedures should be used and the method must be an unequivocal method. If it is not possible to develop an unequivocal method, the project leader must be consulted.

### 3.2 Groundwater Study Continuing QC

The following specific continuing QC should be used in place of the general continuing QC:

3.2.1 **Reagent Blanks** - 1 to 2 per extraction set

3.2.2 **Blank-Matrix Spikes** - 1 to 3 per extraction set

3.2.3 **Blind Spikes** - 1 blind spike should be submitted for every 3 - 50 groundwater samples.

3.2.4 **Field Blanks** - Field Blanks are only analyzed for positive samples.

## 4.0 AIR STUDY QC PROCEDURES

### 4.1 Air Study Method Validation (trapping efficiency)

In addition to the general procedures, the trapping efficiency should be determined. This normally involves collecting a series of 2-stage air samples. The top stage sampling tube contains glass-wool and is spiked. The bottom stage consists of the normal sampling tube. The 2-stage sample is placed on an air sampler and run for the appropriate amount of time. Both stages are then analyzed to determine the proportion of the spike trapped in the bottom stage. The test should consist of two to five replicate analyses at two to five spike levels. Samplers should run for various lengths of time, if necessary. To determine the precision of the spiking technique, five sample tubes with glass wool should be spiked and analyzed. Oxidation products should also be analyzed to determine the rate of conversion. Exact test specifications are chosen by the project leader.

### 4.2 Air Study Continuing QC

In addition to the general procedures, one reagent spike should be analyzed with each extraction set. The air sampling matrix will occasionally give an enhanced detector response. In general, it is not possible to split air samples, so split matrix analyses are not usually done.

## 5.0 CALCULATIONS

### 5.1 Calculating the Method Detection Limit

The MDL is determined by performing the following calculation:

MDL = t x S

Where:

t = Students t value for 99% confidence level (single-tailed) and n-1 degrees of freedom

S = standard deviation

### 5.2 Calculating Warning and Control Limits

The method validation data are used to set warning and control limits. Warning limits will be established at the mean percent recovery plus/minus 1 - 2 times the standard deviation. Control limits will be established at the mean percent recovery plus/minus 2 - 3 times the standard deviation. Any subsequent spiked samples outside the control limits may require the set of samples associated with that spike to be reanalyzed.

## 6.0 REPORTING REQUIREMENTS

These reporting requirements pertain only to the QC data. There may be other reporting requirements specified in the Analytical Laboratory Specifications Form (Appendix 2).

### 6.1 Reporting Method Development Results

The following should be reported by the lab to the EM QA officer prior to the start of any field sample analyses: the spike level and concentration detected for each sample of the MDL determination, the method validation, and the storage stability. The EM QA officer will review, summarize and submit the data to the project leader.

### 6.2 Reporting Continuing QC Results

The following QC results should be reported by the lab to the EM QA officer on a continuous basis: the concentration of all blanks, the concentration detected for all spikes, the amount added for all spikes. Any spiked samples outside the control limits may require the set of samples associated with that spike to be reanalyzed. The EM QA officer will review, summarize and submit the data to the project leader. In addition, the project leader may request to be notified if any problems arise during the course of chemical analysis.

### 6.3 Reporting Sample Results

The laboratory should not use any spike or blank data to adjust the **field** sample results, unless specified by the project leader. Any adjustments should be made by EM personnel.

## 7.0 STUDY-SPECIFIC DECISIONS

The project leader is responsible for the following specific decisions for each individual study. These decisions must be made for both the primary lab and the QC lab, if one is used. All decisions should be given to the EM QA officer who will document the decisions and transmit them to the lab using the Analytical Laboratory Specifications Form.

**7.1** Method performance goals - reporting limit, specificity, precision, accuracy, sample size, time to complete analysis, etc.

**7.2** Number of MDL spike samples

**7.3** Method validation spike levels and number of replicates

**7.4** Warning and control limit criteria (1 – 3x standard deviation)

**7.5** Storage stability test design

**7.6** Number or frequency of continuous QC spike analyses

**7.7** Concentration of continuous QC spike samples

**7.8** Number or frequency of analytical confirmation

**7.9** Number or frequency of split analyses

**7.10** Use, selection and concentration of an internal standard

**7.11** Number or frequency of replicate sample analyses

**7.12** Number or frequency of blind spike analyses

**7.13** Concentration of blind spike samples (also select analyte(s) if multi-residue method)

**7.14** Number or frequency of replicate extract analyses

**7.15** Number or frequency of split extract analyses

**7.16** Number or frequency of standard reference material analyses

**7.17** Trapping efficiency test design

**7.18** Number or frequency of reagent spike analyses

## 8.0 REFERENCES

California Department of Pesticide Regulation. 1988. Chemistry Laboratory Quality Control Guidelines. Environmental Hazards Assessment Program.

**APPENDIX 1** - U.S. EPA Method Detection Limit Determination

**APPENDIX 2** - Analytical Laboratory Specifications

## APPENDIX 1



### APPENDIX B TO PART 136-DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT-REVISION 1.11

*Definition*

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration ls greater than zero and ls determined from analysis of a sample In a given matrix containing the analyte.

*Scope and Application*

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well de- fined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

Tile MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument, independent.

*Procedure*

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/ noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2.Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free of water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte if interest.

Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL Is to be determined in reagent (blank) water, prepare a laborators standard (analyte in reagent water) at a concentration which Is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between **1** and 5 times the estimated method. detection limit) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

It the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as Is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the anelyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) Insure that the procedure *Is* being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly

lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in **4a.** Evaluate these data:

(l) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven-measurements for calculation of the MDL.

**(2)** If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4aor 4b.

5. Calculate the variance ($S^2$) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2/n\right]$$

where:

$$S=(S^2)^{1/2}$$

$X_i$ ; i=1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and $\sum$ refers to the sum of the X values from i=1 to n.

**6. (a)** Compute the MDL as follows:

$$MDL_S = t_{(n-1,\ 1-\alpha\ =0.99)}S$$

Where:

MDL = the method detection limit

$t_{(n-1,\ 1-\alpha\ =0.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution ($x^2$/ df).

LCL= 0.64 MDL

UCL= 2.20 MDL

Where: LCL and UCL are the lower and upper 95%, confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of M D L formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step **4.**

(b) If this is the second or later iteration of the MDL calculation. Uses $S^2$ from the current MDL calculation and $S^2$from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituteing the larger $S^2$into the numerators $S^2$ and the other into the denominator $S^2{}_B$. The computed F-ratlo is then compared with the F-ratlo found in the table which is 3.05 as follows: if $S^2{}_\lambda/S^2{}_B<3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \left[\frac{6S_\lambda^2 + 6S_B^2}{12}\right]$$

If $S^2{}_\lambda/S^2{}_B$ >3.05, respike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c ) Use the $S_{pooled}$ as calculated in 7b to compute the final MDL according to the following equation:

$$MDL=2.681(S_{pooled})$$

where 2.681 ls equal to $t_{(12,\ i-\alpha\ =0.99)}$ .

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

LCL=0.72 MDL

Tables of Students' t Values at the 99 Percent

Confidence Level

| Number of replicates | Degrees of freedom (n-1) | $t_{(i-n,\ 0.99)}$ |
|---|---|---|
| 7 | 8 | 3.143 |
| 8 | 7 | 2998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |
| 16 | 15 | 2.602 |
| 21 | 20 | 2.528 |
| 26 | 25 | 2.485 |
| 31 | 30 | 2.457 |
| 81 | 60 | 2.390 |
| 00 | 00 | 2.326 |

## *Reporting*

The analytical method used must be specifically identified by number or title aid the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which

affect the method detection limit, these conditions must be specified with the **MDL** value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery,

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the anlyte in reagent water, do not report a value for the MDL.

[49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986]

## .APPENDIX C TO PART 136-INDUCTIVEIY COUPLED PLASMA-ATOMIC EMIS· SION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

### *1. Scope and Application*

1.1 This method may be used for the de- termination of dissolved, suspended, or total elements in drinking water, surface water and domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See Section 5.)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples appropriate steps must be taken to correct for potential interference effects. (See Section 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other element may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead the analyst is referred to the instruction provided by the manufacturer of the Particular instrument.

### *2. Summary of methods*

2.1 The method describes a technique for the simultaneous or sequential multi-element determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic•line emission spectra are produced by a radiofrequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate **corrections made.**

### *3. Definitions*

3.1 *Dissolved*-Those elements which Will passthrough a 0.45µm membrane filter.

3.2 *Suspended*-Those elements which are retained by a 0.45µm membrane filter.

3.3 *Total*-The concentration determined on an unfiltered sample following vigorous digestion (Section 9.3). or the sum of the dissolved plus suspended concentrations. (Section 9.1 plus 9.2).

3.4 *Total recoverable*-The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (Section 9.4).

3.5 *ln8trumental detection limit*- The concentration equivalent to a signal due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 *Sensitivity*-The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 *Instrument check standard*-A multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis, (See 7.6.1)

## APPENDIX 2

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

Project No. Lab
Lab Project Manager Phone
Project Chemist Phone
EM Project Manager Phone
EM Lab Liaison/ QA Officer 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 analytical method and matrix.

**Validation**

Method# _____
Sample Matrix: _____
Analyzed For: _____
Reporting Limit: _____
Other Specification : _____

**Levels**

| | Matrix | The Spike Levels in Set |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |

# Reps _____

**Control limits for continuing QC are established with validation recovery data at** +/- *2 X* **the standard deviation for each analyte.**

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ANALYTICAL LABORATORY SPECIFICATIONS

CONTINUING QUALITY CONTROL

Blanks

Reagent or Solvent Spikes

Matrix Spikes

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 OAQC001.01 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

**CALIFORNIA DEPARTMENT OF PESTICIDE REGULAT I ON**
**ENVIRONMENTAL MONITORING PROGRAM**
**ANALYTICAL LABORATORY SPECIFICATIONS**

**REPORTING PROCEDURES**

**Samples to be analyzed:**
Primary Samples
Backup Samples
Field Blank Samples

**Completing the Chain of Custody Record:**

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

4. For those samples which contain no detectable amount write "none detected" or "ND" and indicate the reporting limit.
5. The chemist who analyze d 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:**