US Army Corps of Engineers ®
Louisville District

LOUISVILLE CHEMISTRY GUIDELINE (LCG)

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ENVIRONMENTAL ENGINEERING BRANCH
LOUISVILLE DISTRICT

June 2002

LOUISVILLE CHEMISTRY GUIDELINE (LCG):

- **CHEMICAL ANALYSIS CRITERIA**
- **DATA REPORTING**
- **DATA ASSESSMENT**
- **DATA VALIDATION**
- **CHEMICAL QUALITY ASSURANCE REPORT (CQAR)**
- **CHEMICAL DATA QUALITY ASSESSMENT REPORT (CDQAR)**

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FORWARD

LOUISVILLE CHEMISTRY GUIDELINE

This is an evergreen document that is intended to summarize the requirements for evaluating environmental chemistry data quality and to help ensure proper preparation of the appropriate documentation for HTRW projects.

There are no restrictions on the distribution or reproduction of this document. However, it is the responsibility of the User to maintain the most current version. Revisions and additions to this document will be posted on the USACE Louisville District web page. Users are encouraged to frequently visit the web page to obtain current updates and make suggestions for further improvement of this document.

It is the intent of Louisville District to ensure that all laboratories and contractors who are involved in any environmental chemistry process (with Louisville District) are familiar with and follow the guidelines contained herein. All laboratories and contractors are required to complete the following Ethics and Data Integrity Agreement and keep the signed agreements on file.

If you have any suggestions, revisions, additions, or need clarification of any part of this document, please contact the author, *name removed* (webmaster.celrl@LRL02.usace.army.mil or 502-315-6324).

ETHICS AND DATA INTEGRITY AGREEMENT

_____
(Laboratory/Company)

I. I, _____ *(Name),* state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at _____ *(Laboratory/Company).*

II. I agree that in the performance of my duties at _____ (*Laboratory/Company*):

a. I shall not intentionally report data values that are not the actual values obtained;
b. I shall not intentionally report the dates and times of data analysis that are not the actual dates and times of data analyses; and
b. I shall not intentionally represent another individual's work as my own.

III. I agree to inform _____ *(Laboratory/Company)* of any accidental reporting of non-authentic data by myself in a timely manner.

III. I agree to inform _____ *(Laboratory/Company)* of any accidental or intentional reporting of non-authentic data by other employees.

_____
(Signature)

_____
(Date)

REVISIONS AND ADDENDA

Revisions to this LCG will be noted below and incorporated into this document as warranted. All revisions and addenda will be reviewed by the USACE Louisville District Senior Chemist prior to incorporation.

VERSION NO.	DATE OF INCORPORATION	BRIEF DESCRIPTION OF REVISION	USACE SENIOR CHEMIST INITIALS
2	September 2001	Document revised per comments solicited from various laboratories and USEPA.	SAM
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1.0 INTRODUCTION

This document summarizes the environmental data assessment required for Louisville District projects. Primary and QA laboratories must be validated by Corps of Engineers HTRW-CX branch in order to conduct sample analysis. Louisville District requires data not be reported based on the Method Detection Limits (MDL), but must be reported on the basis of established reporting limits. A procedure for establishing the reporting limit and its relation with action limits is outlined. Since the terms of data review, data verification and data validation has been used interchangeably, by different organizations, they are defined herein. Data Review steps have been defined in relation to laboratories, A/Es, and independent 3rd party Data Validators. These steps are summarized on the Flow Chart presented on page12.

A/Es and laboratories request and/or conduct MS/MSD for the organic and inorganic analysis. However, Louisville district does not require MS/MSD for metal analysis. Rational for analyzing or not analyzing MS/MSD for organic and inorganic analysis is discussed in Section 4.0.

Louisville Chemistry Guideline (LCG) is written to describe the process of data review by the laboratory, data verification by the A/E, comparison of QA and QC data (primary), data validation by an independent validators, and the final report describing the acceptance/rejection of the data. In order to avoid the situation of rejecting all the data, it is expected that all parties involved in Louisville District projects implement the LCG. This document has been divided into six sections as described below.

Section I, Chemical Analysis Criteria: acceptance criteria for analyses with flagging criteria have been summarized in tables. All parties involved in a project for the Louisville District should be aware of these Method Quality Objectives (MQOs). It is very important that these tables be given to the laboratory analyst, so he/she becomes aware of Validators' technical judgment on the data that he/she produces.

Section II, Data Reporting: a summary of the hard copy deliverable is presented. It is expected that laboratories include detailed information in the case narrative.

Section III, Data Assessment: this chapter was entered from the *US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997*. Steps of data review were entered in this chapter to correspond to the summary outlined on the Flow Chart, page 11.

Section IV, Chemical Quality Assurance Report (CQAR): this chapter was entered from the *US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997*. This CQAR is a Corps of Engineer tool that is used to compare the results from the QA sample analysis to the primary sample analysis. A reporting format is presented in this section.

Section V, Data Validation Report (VR): Since sample analyses are conducted based on SW-846, a guideline is provided to validate the data accordingly. A Scope of Work for data validation is presented in this section. Furthermore, validation checklists were prepared in order to assure uniformity among Validators. The author emphasizes the importance of the Validators' technical judgment based on their expertise.

Section VI, Chemical Data Quality Assessment Report (CDQAR): this report is issued by Louisville District Chemist, and is based on the findings in the CQAR and/or the validation report.

2.0 DO's AND DON'Ts CHECKLISTS

Project Name:

Table 2-1 DO CHECKLIST	
Do Item	**Completed Y/N**
Have all personnel who participate in USA Corps of Engineers projects sign the attached ETHICS AND INTEGRITY AGREEMENT.	
The Initial Calibration Verification (ICV) must be conducted immediately after acceptance of initial calibration. THE ICV MUST BE A 2nd SOURCE and must be shown as such in the log book and furnished in final data package.	
Express results as µg/l and µg/kg for water and soil/sediment samples respectively for the following analyses: Volatile Organic, Semivolatile Organic, Pesticides, and PCB. Express results as µg/l for water samples and mg/kg for soil/sediment samples for explosive residues and metal analytes.	
Report percentage solid for soil and sediment samples on the data sheet.	
Report the following dates of samples on their corresponding analytical data sheet: Date Collected, Date Extracted, and Date Analyzed.	
Report surrogate recoveries and their QC limits on their corresponding analytical sheet.	
Dilute sample extracts/digestate to the calibration range when initial concentration levels are outside the upper limit of the calibration curve. Report results of the diluted and undiluted analyses in the data package.	
Report concentration of compounds that were quantified off the primary column and confirmed by the confirmatory column analysis for GC/ECD or HPLC methods. Report the higher of the two concentrations, unless a positive matrix interferents is acknowledged. The QC criteria for the confirmation column must also be met (including ICAL, CCV etc.)	
Include the following information for all MS/MSD and LCS conducted: theoretical concentration of solutes spiked to sample matrices, concentration of the analytes present in the matrices before spiking, concentrations of determined solutes (recovered) after spiking, % recovery, and RPDs.	
A pre-digested metal spike and a matrix duplicate are the required QC analysis for metals. Also, conduct duplicate (Field, Lab) analysis, and calculate RPD. When a pre-digested matrix spike displays recoveries outside the control limits, performance of a serial dilution and/or post-digestion spike is required and possibly the method of standard additions.	
Ultrasonic method is only allowable for specific site when laboratories provide a proof of comparability between the EPA3550 and 3540/3545 methods. A pilot study of at least 20 samples (exclusive of EPA8330 for explosives analysis) must be conducted to prove. The following must be met. 1) The sonication method must detect the same compounds as the soxhlet method. 2) The standard deviation between concentrations must be within ±2 standard deviations with the approval of the District Chemist.	
Control charts for the following: Surrogate Recoveries, Blank Spike Recoveries, LCS, and QCMRL for the period of sample analyses, should be available on request.	

Table 2-1 DO CHECKLIST	
Do Item	**Completed Y/N**
Make efforts to analyze samples within the first half of the holding time, so repeat analyses if needed can be conducted in the second half of that holding time.	
Include Chain of Custody and Cooler Receipt Forms in the data packages.	
Include the Quality Assurance Officer/Manager signature and Laboratory Manager signature in each data package and report.	
Data for Inorganic and Organic analyses reported at the MRL. Lab should indicate MDLs even if report is on CLP forms.	
Auto Trail must be on for GC/MS analyses.	
Data Validator must initial and date pages of data results that have been validated	
Manual integration – Laboratory must report manual integration in case narrative	
Manual integration should be reviewed by an Independent 3rd Party Validator	
Manual integration – a "before" and "after" hard copy, with the reason, date and signature of the analyst must be provided	

Project Name:

Table 2-2 DON'T CHECKLIST	
Don't Item	**Completed Y/N**
Do not report analytical data per wet weight.	
Do not use ultrasonic methods for organic soil extraction (EPA 3550B), exclusive of explosive analysis (EPA 8330). Use EPA 3540 or 3545, these methods are preferable over the sonication method EPA 3550, unless pilot study provides replicate results.	
Do not conduct MSD or LCSD for metal analysis.	
Do not report MS/MSD and/or LCS results for organic analyses without their acceptable QC limits.	
Do not report analytical data with elevated reporting limits due to dilution with results that are below detection limits (BDL). The elevated reporting limits would be acceptable when undiluted matrix results are reported, and attempts were made for cleaning the sample matrix.	
Validators must not alter the reported results by crossing out data and changing. If the form has incorrect data, have the laboratory revise. If qualifying data, only place qualifier.	

3.0 ENVIRONMENTAL DATA ASSESSMENT GUIDELINE

The purpose of this document is to summarize the requirements set forth by US Army Corps of Engineers (USACE), Louisville District for evaluating data quality and for providing the appropriate Analytical Reports. The goal of USACE-Louisville District is to obtain analytical data of definitive quality that meet all project specific requirements.

3.1 Laboratory Validation

The Louisville District requires environmental laboratories be validated by the USACE-HTRW-CX, Omaha, Nebraska. The Louisville District and/or A/E firms are the laboratory contract holders. The District Chemist initiates the validation process for both the Louisville District and A/E firms. There are two types of environmental laboratories for HTRW projects:

3.1.1 Primary Laboratory

Primary samples constitute 100% of the samples collected and may include field duplicates (normally10%), to be analyzed by a certified laboratory. This laboratory is called a Primary or QC laboratory. Primary Sample Analysis Data is usually called QC data. QC data should not be confused with the laboratory QC analyses performed during analysis such as method blanks, duplicates, and spiked samples. See Flow Chart-I, page 12.

3.1.2 QA Laboratory

Samples that are split off primary or duplicate samples (normally10% of primary) are called QA samples, and are to be analyzed by another certified laboratory. This laboratory is called a QA laboratory. See Flow Chart-I, page 12. Section IV: chemical Quality Assurance Report discussed the minimum requirements for QA Split Sample Analysis.

Approval of primary and QA laboratories must be obtained from the Louisville District Chemist.

3.2 Chemical Analysis

The analytical testing of environmental samples must be conducted according to SW-846 and the USACE SHELL. In order to assure that the Analytical Data are scientifically and legally defensible, the Louisville District prepared this document as a guideline for A/E, environmental laboratories, and data validation contractors. Its purpose is to generate data for the intended usage on the first attempt.

The laboratory should review this document and be aware of the QAPP/SAP (project specific), EM 200-1-3 (available on http://www.usace.army.mil/inet/usace-docs/eng-manuals/em.htm),

USACE Shell for Analytical Chemistry Requirements (Document CEMP-RT-025 available on http://www.usace.army.mil/inet/usace-docs/review/cemp.htm), and SW-846 in order to determine project-specific DQOs and project requirements.

The Chemical Analysis Criteria, Section I, introduces a procedure for setting up Method Reporting Limits (MRL), and summaries of Method Quality Objectives (MQO) for Louisville District.

Laboratories must adopt the analytical requirements outlined in the MQO tables for any of the requested analyses (see Section I). In addition to the practiced QC requirements in SW-846, there are three additional QC requirements that must be implemented in all analytical analyses for Louisville District as indicated below.

3.2.1 Method Reporting Limit (MRL)

A procedure for setting the MRL is presented in Section I. The MRL is set based on the Method Detection Limit (MDL), action level, and risk assessment levels. It should be set at ≥ 3MDL and below one half of the action level for any specific site cleaning. Also, MRL must be set below the risk assessment levels. If 3MDL for a particular analyte is above the risk assessment level, the laboratory must contact the District Chemist before proceeding with the analysis. The MRL Tables are located in the Appendix.

3.2.2 QC/MRL Check

A QC sample at the MRL concentration must be analyzed at a low level of the calibration curve. This standard is run before and after analyzing USACE samples. The QC/MRL is a low-level standard that is not exposed to either digestion or extraction. The acceptance criteria for the QC/MRL are indicated in the tables, Section I.

3.2.3 QC/MDL Check

A QC sample of known concentration must be analyzed to verify instrument sensitivity at the MDL on a quarterly basis for every instrument used to run USACE samples. The QC/MDL concentration is at ≅ 2 MDL. The QC/MDL is a low-level standard that is not exposed to either digestion or extraction. The acceptance criteria for the QC/MDL are indicated in the tables, Section I.

Data Reporting: It is preferable to report the analytical data on CLP forms even though SW-846 methodology was employed in producing such data. There are items that must be reported on the same analytical data sheet, % solids for soil/sediment samples, sample weight or volume, dilution factors, and surrogate recoveries with their QC limits. See Section II for data reporting requirements.

3.3 Contaminants of Concern VS. Non-contaminants of Concern

Contaminants of concern pertain to VOC and SVOC analyses. Any analyte that may be present at a site should be considered a contaminant of concern. The contaminants of concern must meet the primary evaluation of data as presented in the MQO Tables 1 and 2. No sporadic failure is allowed for the contaminants of concern.

3.4 Reporting of PCB and Pesticide Results
Results for either PCB or Pesticide analysis must be reported from both columns.

3.5 Data Assessment Process
Review of the analytical data will normally be conducted incrementally on Sample Analytical Groups instead of the entire analytical data at the end of the project. Analytical results for samples that are sent to primary and/or QA laboratory will be reviewed when their data packages are ready for release to the client (A/E or Louisville District). Size and frequency of the Sample Analytical Groups will be determined at project initiation. There are four steps for review to achieve acceptable data for risk assessment purposes. These steps are defined below, and are designated on the attached Flow Chart-I, page 11.

3.5.1 Laboratory Data Review (Steps 1 & 1A)
Primary- and QA-Laboratories review their data before releasing data packages/reports to the A/E or to Louisville District. The review process is the same for both primary and QA laboratories. Accordingly, STEP-1 is designated for review by the primary laboratory and STEP-1A is designated for review by the QA laboratory.

3.5.2 Process
The review may be variable as a function of the laboratory Quality Assurance Plan, and the type of work that is being performed. At a minimum, review should include routine quality control (QC) data, but may also include Special Reporting Requirements. The data review requirement should be determined prior to the start of the analysis, i.e., during project scope set up and negotiations.

3.5.3 Product
Analytical Reports must contain the analytical results with laboratory QC data. The reports will have the following items:

Cover Page: the cover page must contain the Project Name, and a statement indicating the authenticity of the data. Laboratory Director and Quality Assurance Manager/Officer must enter their signatures with dates on this page.

Case Narrative: a case narrative describing any non-conformances with SW-846 methodology and/or the -Tables of Method Quality Objectives (attached).

Summary Surrogate Recovery Sheet: This sheet must precede the analytical results of each method. The results of each employed method must be presented in the following order for each applicable method:
- Volatile Organic Data Sheet (EPA8260B)
- Semivolatile Organic Data Sheet (EPA8270C)
- Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) (EPA8280A or 8290)

- PCB Data Sheet (EPA8082)
- Pesticides Data Sheet (EPA8081)
- Explosive Residues Data Sheet (EPA8330, and/or EPA8095)
- Herbicides Data Sheet
- Wet Chemical Data Sheet
- Metal Data Sheet (EPA6010, 7000)
- Laboratory Quality Control Section: Method Blanks, LCS, MS, MS/MSD, Pre-Digested, Post-Digested Metal Spikes Serial Dilution.

Analytical Data Packages: The laboratories will provide data packages that will contain the raw chromatogram, with a copy of the analytical data sheet attached to the Sample Delivery Groups (SDGs).

3.6 Data Verification (Step-2)

An independent entity or laboratory contract holder for the primary laboratory (A/E) performs this process.

3.6.1 Process

This process must evaluate the Completeness, Consistency, and Compliance of a data package against the Quality Assurance Project Plan (QAPP). This process requires a Definitive Data Package, see Section III, *US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997*.

A/Es will extend the data assessment process to **include additional data verification.** This verification process will include the following: Calibration Criteria, Internal Standard Areas/Peaks, Calibration Blanks, results of Initial Calibration Verification (ICV), results of Continuing Calibration Verification (CCV), results of Method Reporting Limit QC (MRL/QC) recovery, results of Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSDs), and/or Matrix Spike/Matrix Spike Duplicate (MS/MSD), results of surrogate recoveries, and results of duplicates. The reviewer performs verification of 100% of the primary sample results.

3.6.2 Product

The reviewer might assign and/or change qualifiers that were assigned by the laboratory to fit their findings, without re-calculating the positive hits in the data. This will result in a Data Verification Report (typically submitted in a summary table(s) format), analytical results and quality control summary tables.

3.7 Data Assurance (Step-3)

The Louisville District or an independent contractor (not primary lab) performs this process that produces the CQAR.

3.7.1 Process

The intent of data assurance is to provide a complete assessment of the quality of the data by examining primary samples (confirmatory), 10% duplicates, and their 10% split samples (QA) via comparison of the QA sample results to the duplicate and/or primary sample results. Examination of the primary (confirmation) sample data, and their 10% split samples (QA) provides the data user with a degree of the acceptance and usability of the Chemical Data Quality. The finding should be summarized in a report format labeled Chemical Quality Assurance Report (CQAR).

3.7.2 Product

A detailed description of the CQAR preparation is provided in Section IV of this document, Chapter 4 of the *US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997*. The CQAR is a document that is prepared by an independent entity that is not involved directly in the analysis of the primary samples. In order to assure an acceptable quality of primary sample results, the CQAR will normally be divided into sampling event CQARs over the duration of the project. Any nonconformance with the sampling analysis plan (SAP) will be related to the A/E, and to the laboratories for corrective actions. Corrective actions will be implemented in order to avoid such deficiency in the subsequent phases of analysis. This approach allows in real-time, determination of the lab analytical performance, allows immediate determination of data integrity, and data usability. At the end of the project all the CQARs will be assembled into one final CQAR.

3.8 Data Validation (Step-4)

Louisville District is responsible for the data validation, and it may contract with a 3rd party to validate (10%) of the raw data. See a memo by Office of Council in Appendix A for definition of the independent 3rd party.

3.8.1 Process

Data validation may be done concurrently with the CQAR. It is recommended for data validation to be ongoing throughout the duration of the project. The Validator must first determine if overall data quality problems exist or if data quality problems are specific to a given matrix/method. If the rejected data are deemed to be part of an overall quality problem, the Validator randomly chooses 10% of the data package to evaluate and so on until a level of confidence is reached to accept or reject the data (up to 100%). If the rejected data are deemed systematic to a particular matrix/method, the Validator chooses 10% from that specific matrix/method to evaluate. Comprehensive data packages are required for such process, see Section III, *US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997*.

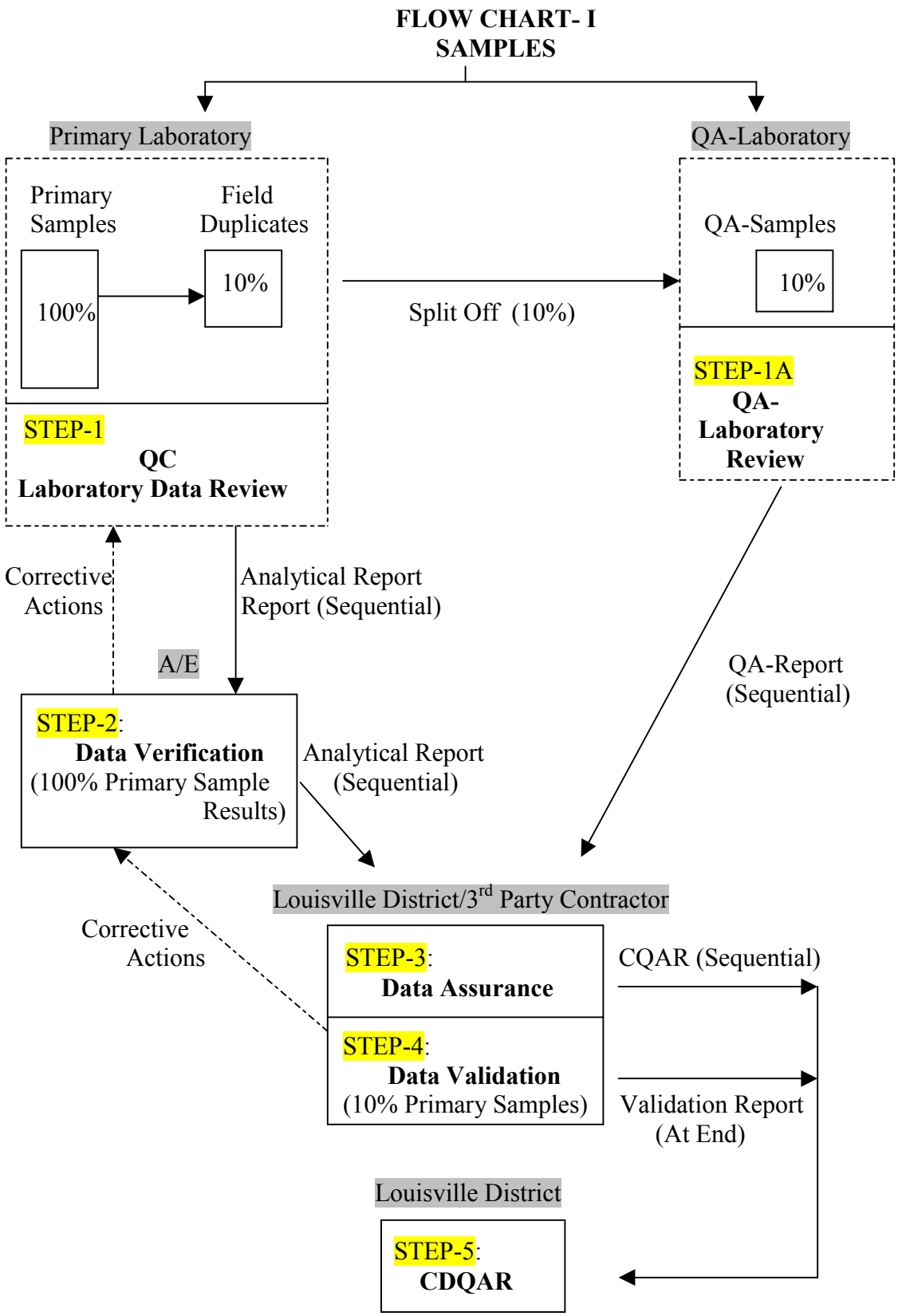
3.8.2 Product

Validators choose randomly the 10% of the raw data for validation. Full data validation consists of validating the data using the Guidelines in Section V and recalculating the positive hits above the MRL. Validator will qualify the data as "U" for levels below the MRL, "J" for estimated values, and "R" for rejected values. If serious problems are encountered during the validation

process, validation should be conducted on the next 10% of the raw data, and so on till a level of confidence is reached to accept or reject the data. The Validation Guidelines, and a Data Validation Checklist, are presented in Section V.

3.9 CDQAR (Step-5)

In order to determine whether the data is acceptable or not, two elements may be required, CQAR and/or Validation Report. If there are no major discrepancies between the QC and QA data provided in the CQAR, and also, the full data validation determined that the data is acceptable based on the level of confidence (normally at ≥ 90% usable data), then the data is considered acceptable. To this end, Louisville District Chemist prepares the CDQAR as outlined in Section VI of this document, *US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997*. A checklist is provided as part of Section VI.


FLOW CHART- I
SAMPLES
Primary Laboratory
QA-Laboratory
Primary Samples
Field Duplicates
100%
10%
Split Off (10%)
QA-Samples
10%
STEP-1A
QA-Laboratory Review
STEP-1
QC
Laboratory Data Review
Corrective Actions
Analytical Report Report (Sequential)
A/E
QA-Report (Sequential)
STEP-2:
Data Verification
(100% Primary Sample Results)
Analytical Report (Sequential)
Louisville District/3rd Party Contractor
Corrective Actions
STEP-3:
Data Assurance
CQAR (Sequential)
STEP-4:
Data Validation
(10% Primary Samples)
Validation Report (At End)
Louisville District
STEP-5:
CDQAR

4.0 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Analytical data are generated for matrix spike/matrix spike duplicate (MS/MSD) to determine precision and accuracy of the analytical methods and extraction methods on various matrices. In order to evaluate the benefits and applicability of MS/MSD, we need to understand the relation between the accuracy, precision, and MS/MSD results.

4.1 Precision and Accuracy

Accuracy is a reflection of the correctness of the results i.e. how closely the measured results are to the true value in the sample. Since, the true value in the sample is unknown, a known concentration of surrogates (non-target analyte) are spiked into the sample matrix. The extracted surrogates are calculated as percentage of the true value providing the accuracy of the measured value to the true value.

Precision is a reflection of how close multiple measurements are to each other on the same sample. Precision is a measure of the variability. Therefore, a precise set of measurement is compact and reflected by a small relative standard deviation (RSD). In the situation of the MS/MSD, precision is measured by the relative percent difference (RPD).

There are three possible outcomes of accuracy and precision measurements, as described below.

4.1.1 Precise and Accurate

The resulting measurements have percent recoveries close to the true value, and RPD levels close to the zero. The percentage recoveries are within the QC limits, and the RPD levels are under the maximum allowable QC RPD limits.

4.1.2 Precise and Less Accurate

The resulting measurements have percent recoveries close to or out of the QC limits (LL & UL), and RPD levels close to zero. The lack of accuracy is an indication of **bias** results. Bias results may be due to idiosyncrasies with the measurement such as improper adjustment of instruments and/or poor extraction/digestion techniques. The idiosyncrasies produce **Systematic Error** with the same sign (+, -) and magnitude.

4.1.3 Less Precise and Less Accurate

The resulting measurements have percent recoveries out of the QC limits, and RPD levels close to/or above the maximum allowable RPD QC limits. The lack of accuracy and precision is an indication of **bias** results with **random error** and will have various signs (+/-) and magnitude.

4.2 Matrix Spike/Matrix Spike Duplicate

MS/MSD analysis is a method of pairing the analytical results of the first test (MS) and the retest (MSD) to determine the **reliability** that is a measure of reproducibility, and the performance of the method on a particular matrix.

The MS/MSD is employed for organic analyses but not for metal analyses as described in the following paragraphs.

4.2.1 Organic Analysis

Sample preparation is conducted by extracting organic analytes from various matrices (aqueous, soil) by exposing samples to organic solvents. All samples are spiked with surrogate compounds in order to monitor the efficiency of extraction and determine the accuracy of the analysis. This preparation step is critical in setting up the MS/MSD. Since organic compounds are moved from the aqueous and/or soil matrix to an organic solvent during extraction, it is imperative to monitor the efficiency of that transfer via spiking the sample with organic compounds in addition to the surrogates that are similar to the analytes of interest. This approach enables the analyst and data user to *compare the transfer of the analytes in relation to the surrogates from its original matrix to the organic solvent, and to determine the matrix effect*. As stated earlier, the percent recovery of the surrogates from samples, in addition to the analyte recoveries from MS/MSD samples, indicates the accuracy of the method. Furthermore, pairing the MS and MSD results, and calculating the RPD determines the precision in measuring the concentration of analytes of interest. The MS/MSD provide information **on the precision of extraction and analysis**. **Results of MS/MSD should be applied in evaluating/validating that particular spiked sample, however, the results in conjunction with other QC information can be used to evaluate/validate the remaining samples in the batch**.

4.2.2 Metal Analysis

Samples for metal analysis are prepared differently from the organic preparation. The samples are not spiked with surrogates, and are digested via addition of acid (s) to the matrix. Since the samples are not spiked with surrogates, there is no analyst's precision to be determined. However, there is the possibility of analytes to be lost during digestion, and also the possibility of low recoveries due to matrix effect.

Example 1:

Recovery of an analyte from MS is ≅ 20%
Recovery of the analyte from MSD is ≅ 20%

The analyst would not be able to determine the cause of MS/MSD low recoveries since they could be due to digestion problems and/or matrix effect.

Example 2:

Recovery of an analyte from MS is ≅ 20%
Recovery of the analyte from MSD is ≅ 85%

Since the digestion is performed under controlled environment, the discrepancy could be due to an error in spiking or an unforeseen event during the digestion process. The MS/MSD must be repeated.

Therefore, MS/MSD pairing is inapplicable to the metal digestion processes. However, pre- and post-digested spike pairing will provide information that might shed some light on digestion problems, or on the release of the analytes from the matrix. Spiking samples with known concentration of analytes before digestion (pre-digested spike) will provide information on the effect of the matrix in measuring the analyte concentration after digestion. The results of the pre-digested spike are usually confirmed by post-digested spike in presence of matrix effect. When both results of the pre- and post-digested spikes are low, one can conclude that it may be due to matrix effect as illustrated below.

Example 3:

Recovery of an analyte from pre-digested spike is ≅ 20%

Recovery of the analyte from post-digested spike is ≅ 20%

One can conclude that the low recoveries are due to matrix effect, and the analyst needs to perform serial dilution and method of standard addition techniques. The method of standard addition must consist of three spikes and a straight.

Furthermore, in case of pre-digested spike recoveries are low, but post-digestion spike recoveries are within acceptable limits as illustrated below:

Example 4:

Recovery of an analyte from the pre-digested spike is ≅ 20%

Recovery of analyte from the post-digested spike is ≅ 85%

The result would indicate the absence of the matrix effect, and indicate analyte might have been lost during digestion.

Determining precision of the analytical measurements is usually achieved by calculating the RPD for duplicate analysis. RPD results within acceptable limits would provide information on **the precision of the analysis**. **The Louisville District does not recommend performing MS/MSD on samples for metal analysis. The Louisville District requires pre-digested (MS) be performed. Post-digested spike must be performed when pre-digested spike is out of the acceptable limits (75-125%).**

4.3 Accuracy and Relative Percent Difference (RPD) Calculation

Accuracy is expressed as the percent recovery of surrogate and/or analyte spikes. Recoveries are calculated on net concentrations and are indicative of two factors:

(a) sample matrix effect
(b) sample preparation technique.

Matrix effect or poor sample preparation results in lower recoveries, assuming analytical instruments are properly functioning, and samples are properly extracted/digested, and in case of soil matrix, are well homogenized.

Precision, which is expressed as the RPD, is the measure of the variability of two or more measurements. Assuming instruments are properly functioning, and samples of soil matrix are homogenized, the RPD becomes a function of the preparation techniques.

There are several ways to calculate the RPD, i.e. using total concentration or net recoveries. The method of choice will depend on the intended use of the RPD results.

4.3.1 Total Concentrations:

When sample extracts are analyzed on two different columns for the purpose of evaluating different columns using the same detector, or similar columns using different detectors, the agreement between the quantitative results should be evaluated using total concentrations. The intended use, in this situation, is to determine the efficacy of the columns and/or the detector. Total concentrations are also used in the RPD calculation when comparing the results obtained from a primary sample with those obtained from a duplicate sample. The RPD is calculated on total concentrations as follows:

$$RPD = \frac{\left| C_1 - C_2 \right|}{(C_1 + C_2)/ 2} \times 100$$

Where:

C_1 = Measured total concentration off the first column or the first sample aliquot
C_2 = Measured total concentration off the second column or the duplicate sample aliquot

4.3.2 Net Recoveries:

When spiked samples are analyzed to determine the efficiency of the transfer of the spiked compounds/analytes from the aqueous to the organic media (e.g. MS/MSDs), the quantitative results must be expressed as the recovery of each spiked analyte according to the following formula:

$$RPD = \frac{\left| R_1 - R_2 \right|}{(R_1 + R_2)/ 2} \times 100$$

Where:
$R_1 = C_s - C_u$
$R_2 = C_s - C_u$
C_s = Measured total concentration of the spiked sample aliquot
C_u = Measured concentration of the unspiked sample aliquot

5.0 MANUAL INTEGRATION

Manual integration is an integral part of the chromatographic analysis; it should be used judiciously to correct for any improper integration by the computer program. Manual integration should only be done by an experienced analyst, under clearly established guidelines, and fully documented. Manual integration should not be invasive and used excessively. Efforts should be made to optimize chromatographic conditions so that manual integration can be minimized. Automated integration should be utilized as much as possible to ensure data of known consistency. Manual integration should not be used to make minor adjustments to automated integration. All manual integration must be well documented and included in the case narrative; a "before" and "after" hard copy of the manual integration must be provided. **Manual integration should be reviewed by an Independent 3rd Party Validator. The validator must review and validate Manual Integration within and beyond the 10% level of raw data that is being validated. An independent 3rd party is defined in Appendix A of this LCG.** At a minimum, the following steps should be included.

The guidelines set forth by Region V USEPA on Manual Integrations are understood. However the majority of the manual integrations are associated only with the 1st and 2nd standards due to the fact that MRLs are being lowered because of risk assessment levels. There are some instances however in which co-elutions occur and manual integration may be necessary. These instances will be documented in the case narrative.

Manual integration should be performed when the following situations occur:

- Missing peak (see Example A)
- Incorrect integration due to:
    - Poor resolution (see Example B)
    - Baseline problems (see Example C)
- Incorrect retention time (see Example D)

Manual integration should be performed using the following techniques (see Peak Evaluation Technique Figure):

- Valley to valley event
- Drop to baseline event
- Peak skimming event

If unconventional manual integration is required due to matrix interference, supervisor approval is required. Manual integration must be consistent with the integration performed in the initial calibration.

5.1 Review and Documentation Process for Manual Integration

Manually integrated chromatograms for GC, IC & HPLC analyses are reviewed as two-dimensional graphs and proper integration is evidently displayed in the final quantitation chromatogram. Hence, the final quantitation chromatogram is used to document proper integration with no further explanation. Examples A through D show proper manual integration.

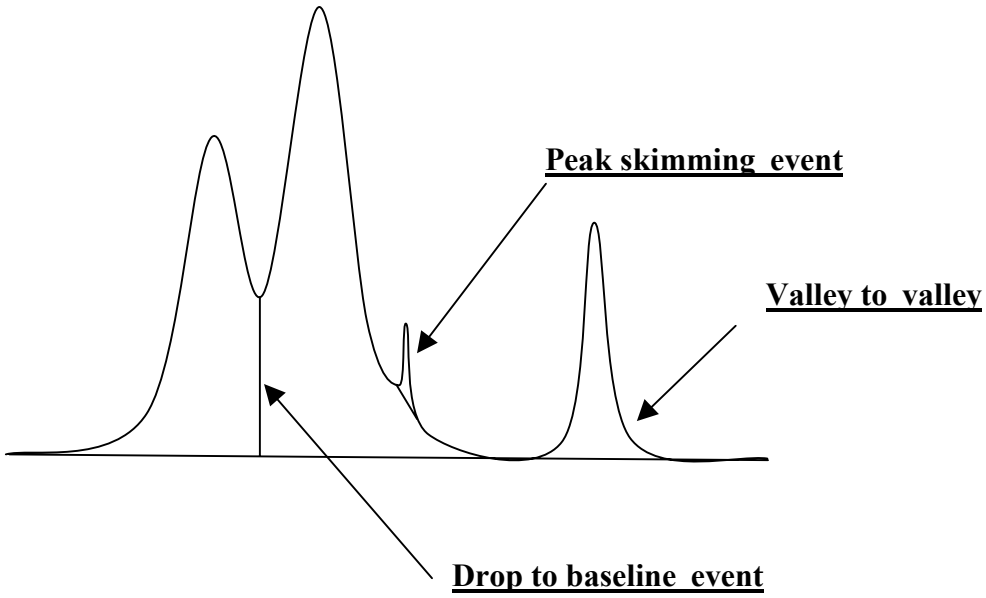
For GC/MS methods manual integration involves the extracted ion profile, which is not discernable in the total ion chromatogram. Therefore to properly review the chromatogram, a comparison of the original quant-ion chromatogram and the manually integrated quant-ion chromatogram is necessary. Both chromatograms shall be retained to document that this process has been performed and the corrected integration shall be explained and acknowledged by the initial of the analyst and the date when it was performed.

The secondary reviewer shall make sure that all of the above are properly executed as part of the data review process.

5.2 Inappropriate Manual Integration

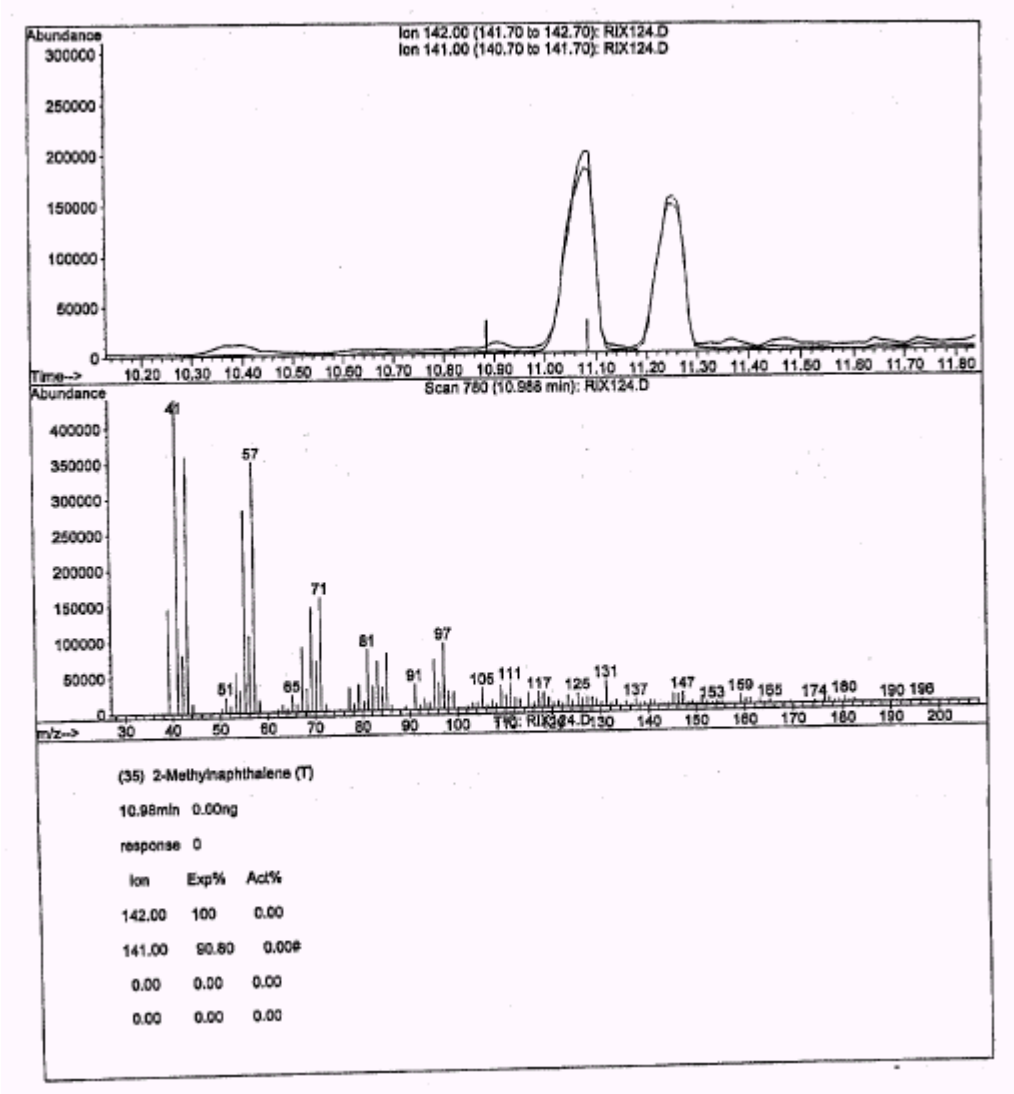
As it is stated in the policy statement for EPA Region 5 Federal Facility Response Section, "Manual integration must not be performed solely for the purpose of meeting quality control criteria. Eliminating part of the subject peak area, or including peaks not belonging to the subject peak are inappropriate manipulation of the analytical data. Examples of improper manual integration are peak shaving, or peak enhancement to make failed calibrations, surrogates, or internal standards appear to meet QC criteria." Examples E through P

Peak Evaluation Technique Figure

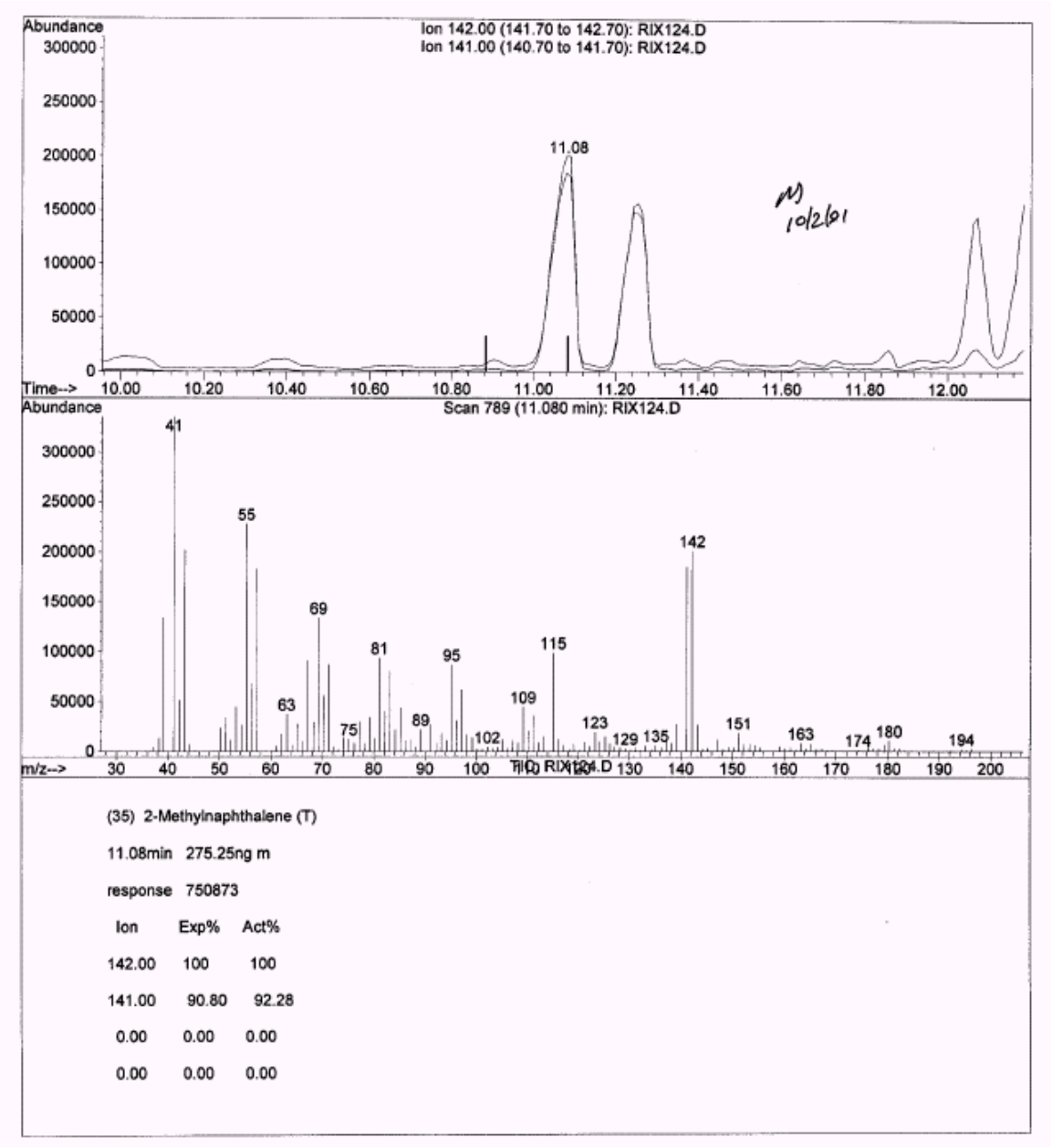


Example A: Missing or Wrong Peak Selected. The 2-Methylnaphthalene (8270 analysis) peak was missed in the initial integration.

Example A Before

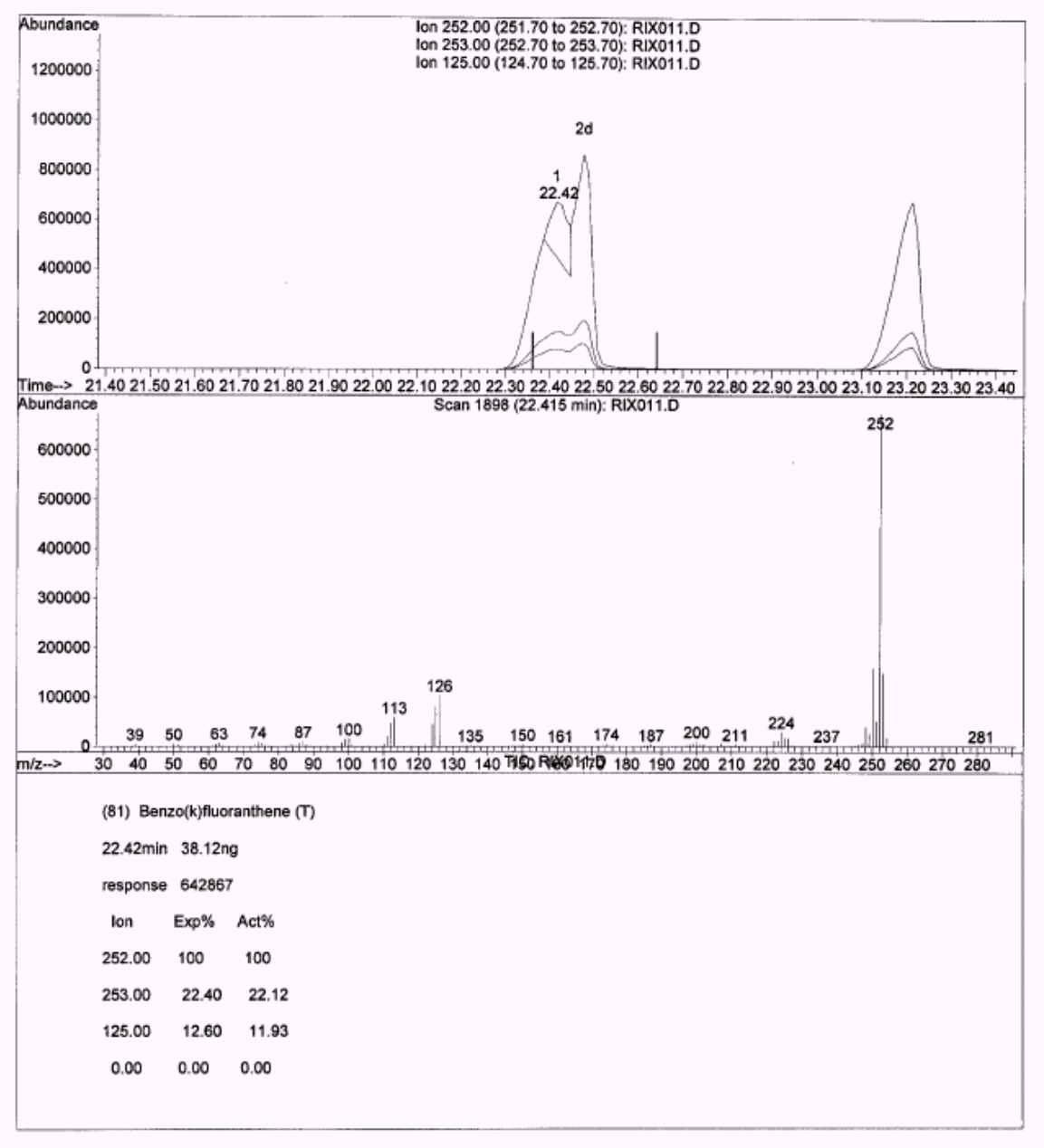


Example A After

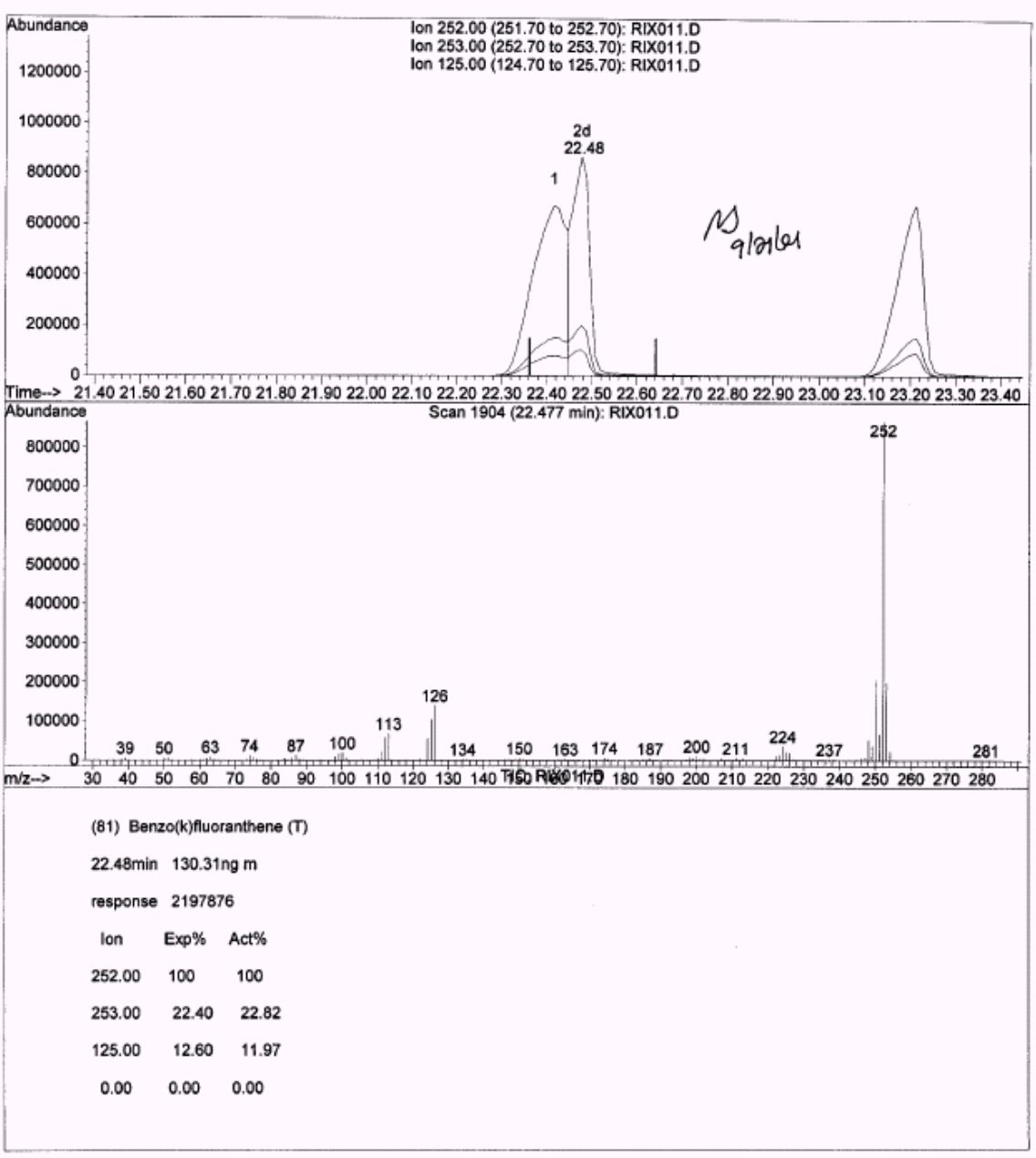


Example B: Incorrect integration due to poor resolution Integration of benzo(k)fluoranthene (8270 analysis) did not properly select the peak and did not split the peaks and drop the baseline on the initial integration.

Example B Before



Example B After



(81) Benzo(k)fluoranthene (T)

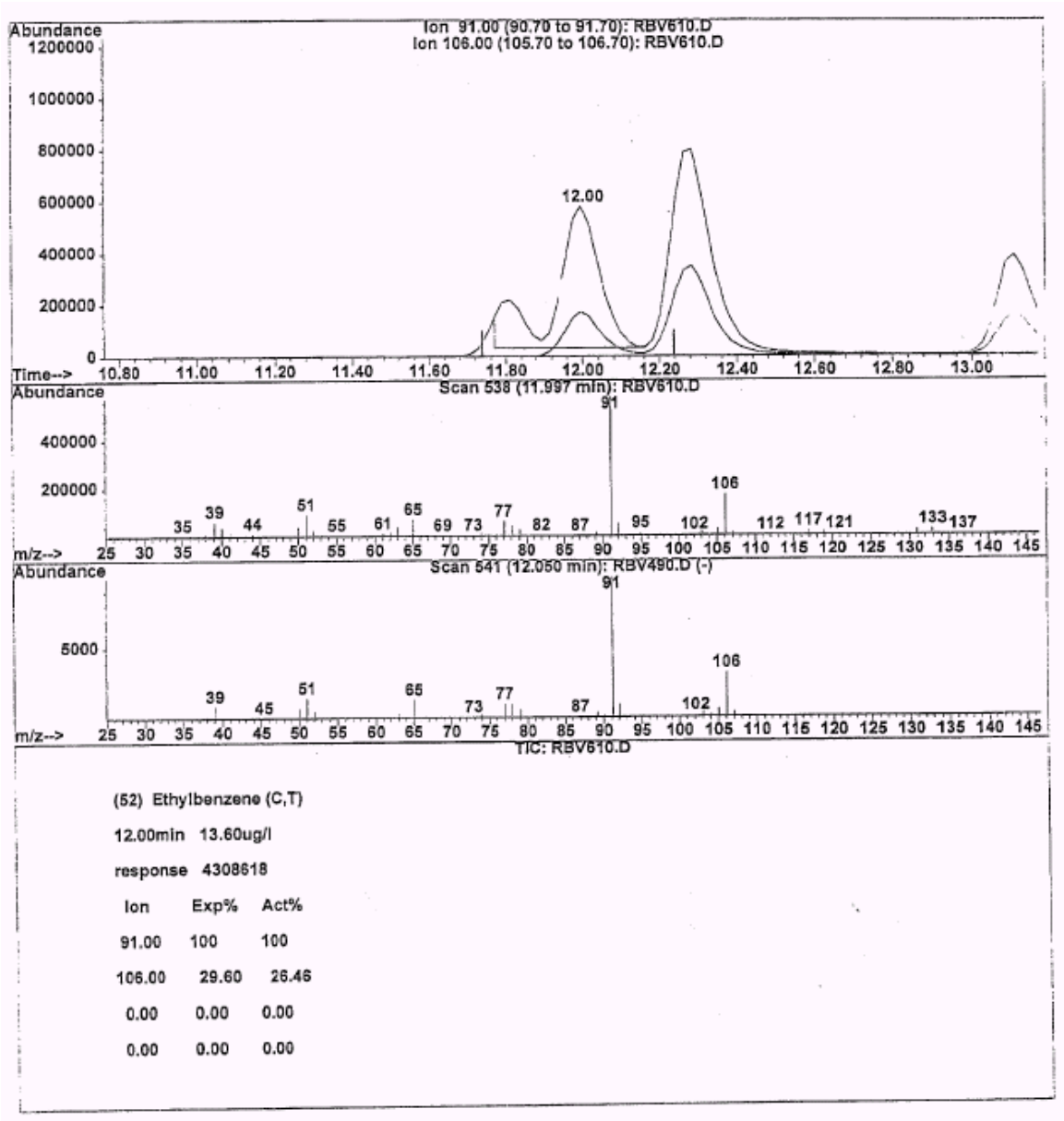
22.48min 130.31ng m

response 2197876

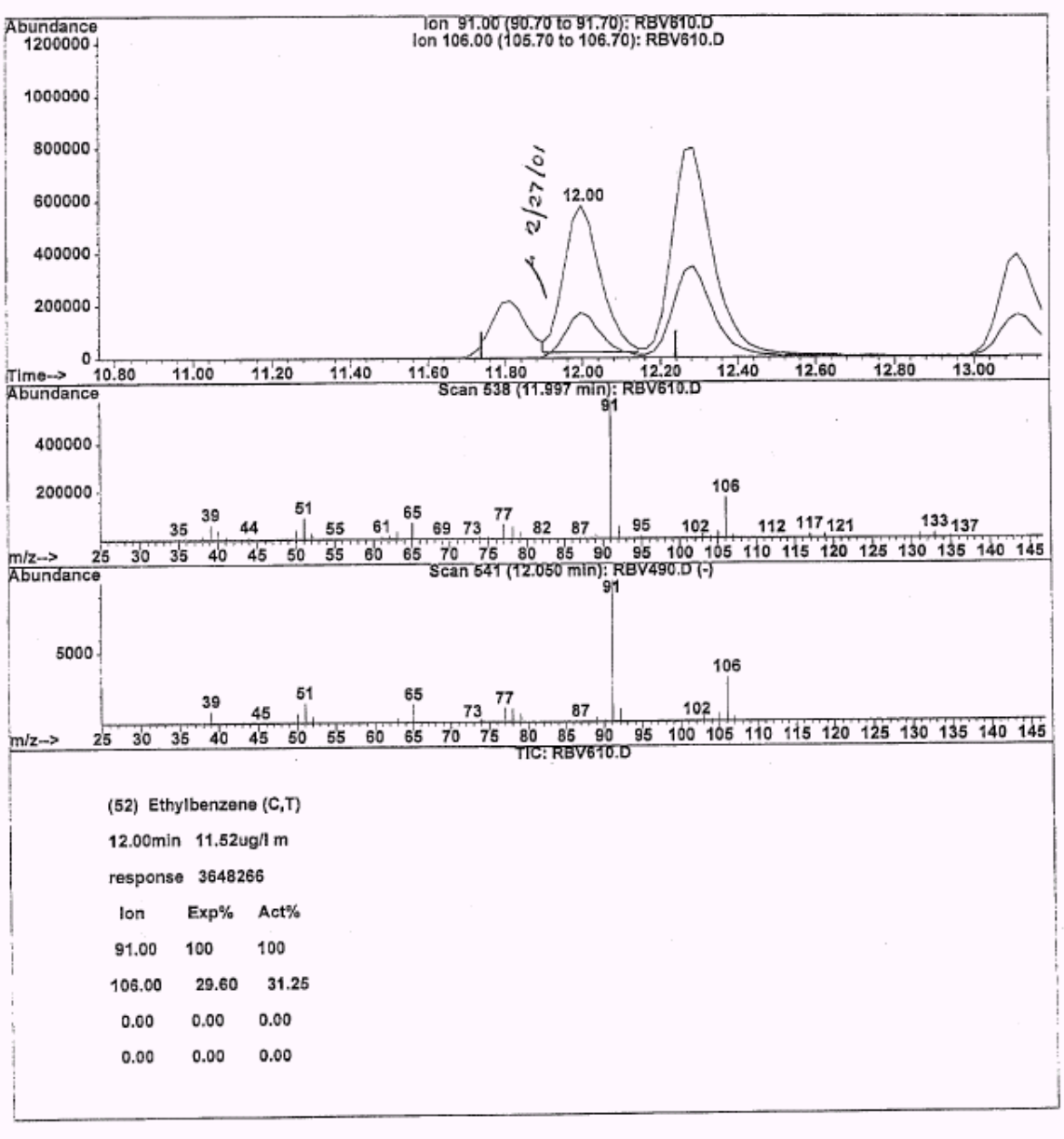
Ion	Exp%	Act%
252.00	100	100
253.00	22.40	22.82
125.00	12.60	11.97
0.00	0.00	0.00

Example C: Baseline integration problem Integration of ethylbenzene (8260 analysis) initially drew the baseline to include area from the peak at 11.80 minutes.

Example C Before

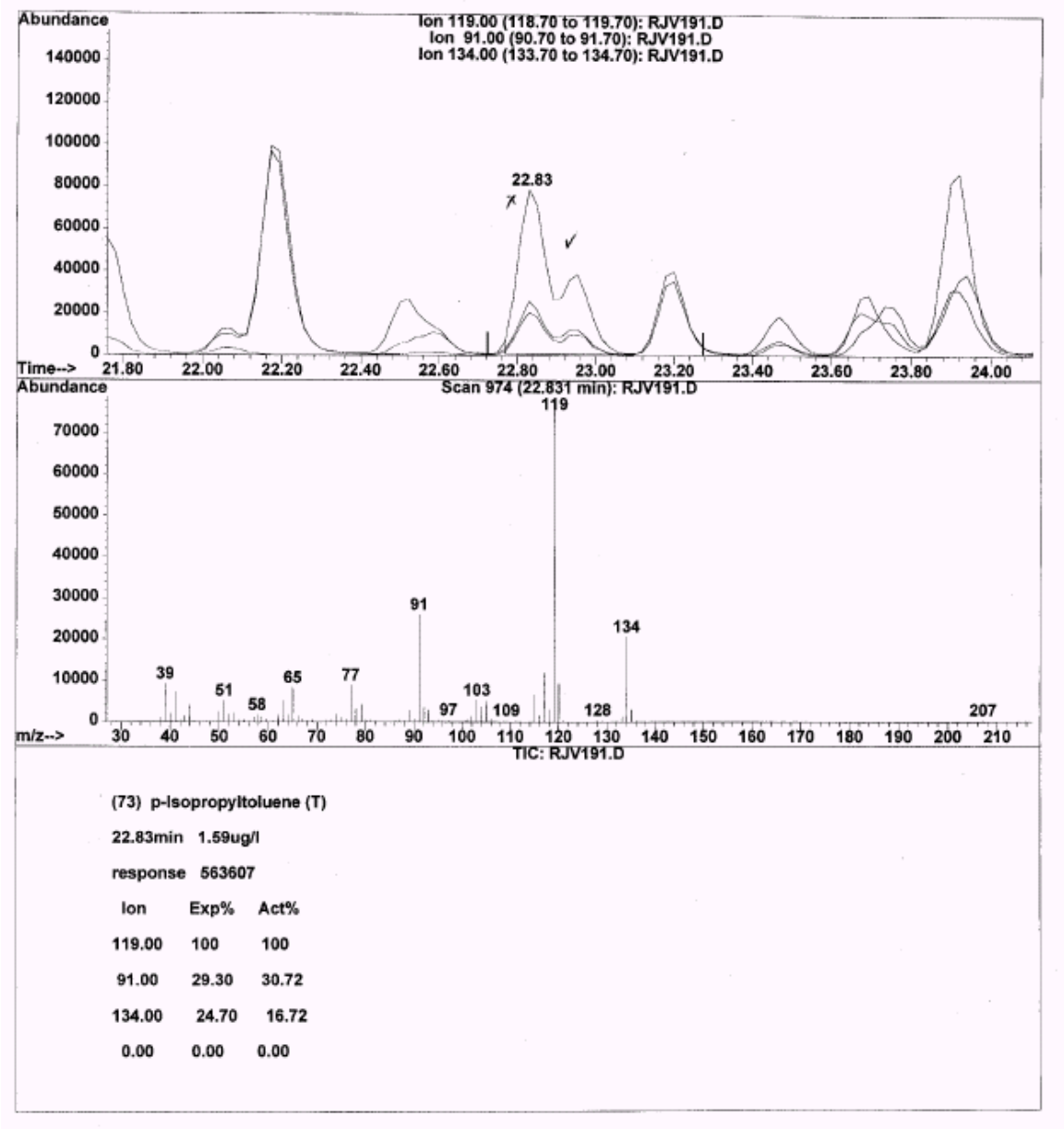


Example C After

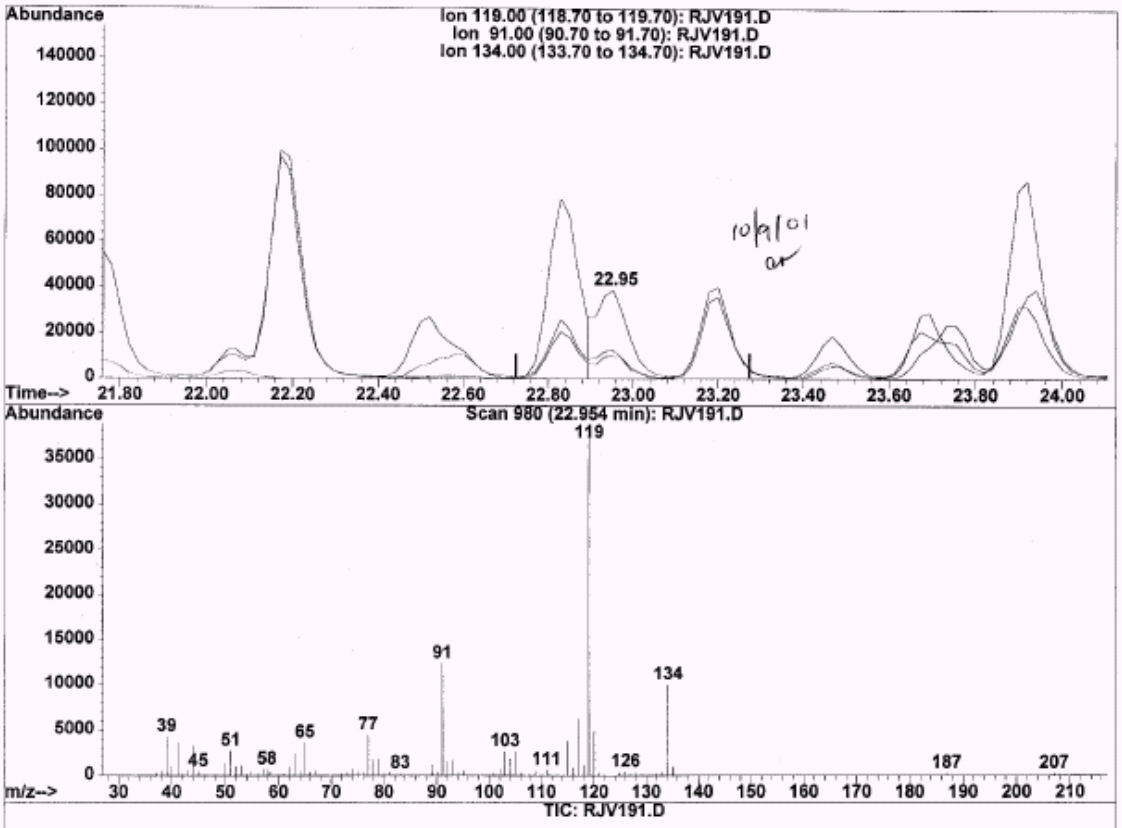


Example D: Incorrect retention time. Integration of p-Isopropyltoluene (8260 analysis) initially selected the incorrect isomer, and did not split the peaks.

Example D Before



Example D After



(73) p-Isopropyltoluene (T)

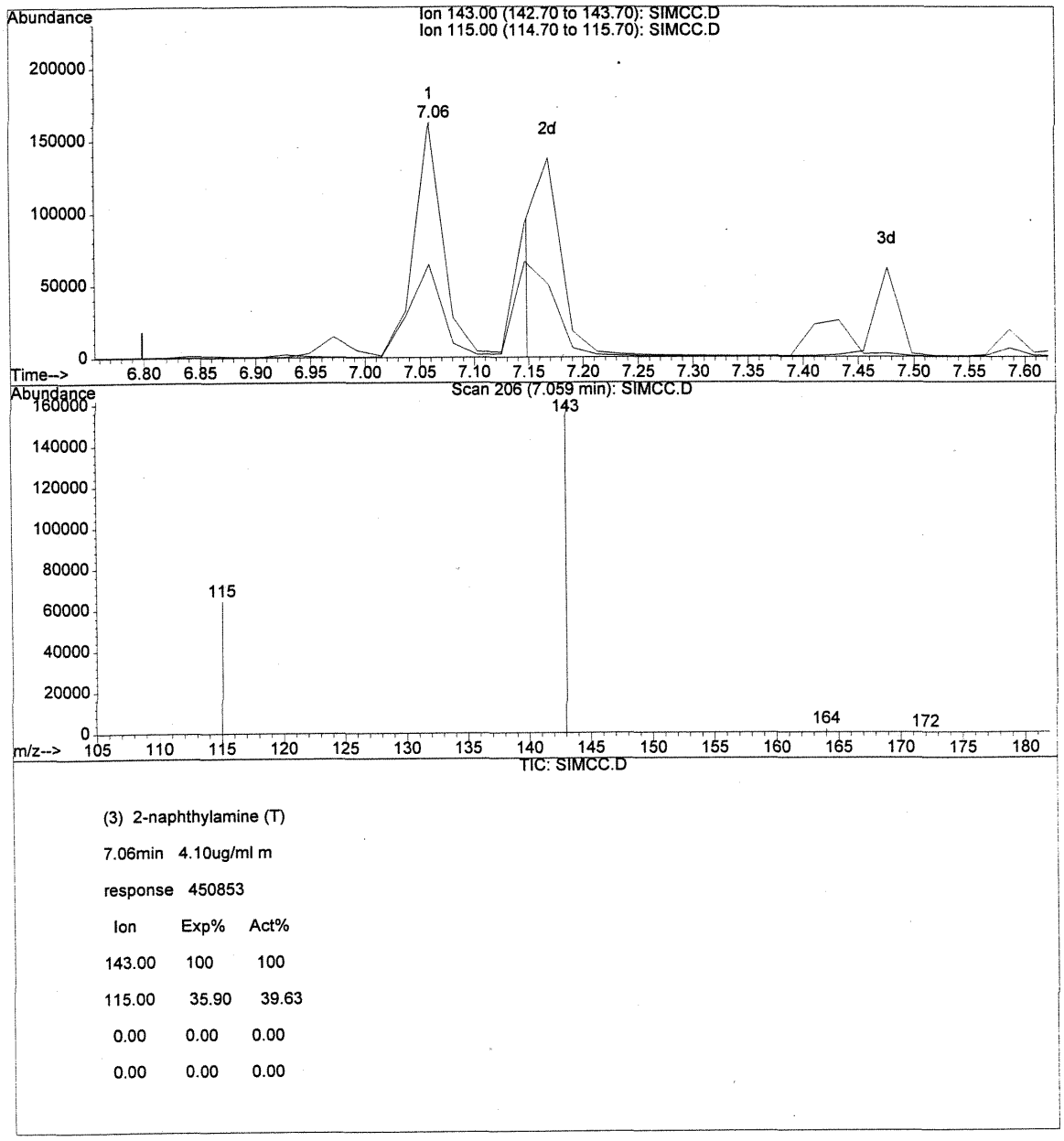
22.95min 0.57ug/l m

response 202740

Ion	Exp%	Act%
119.00	100	100
91.00	29.30	85.39#
134.00	24.70	46.49
0.00	0.00	0.00

Example E

Sample 5 ppm



(3) 2-naphthylamine (T)

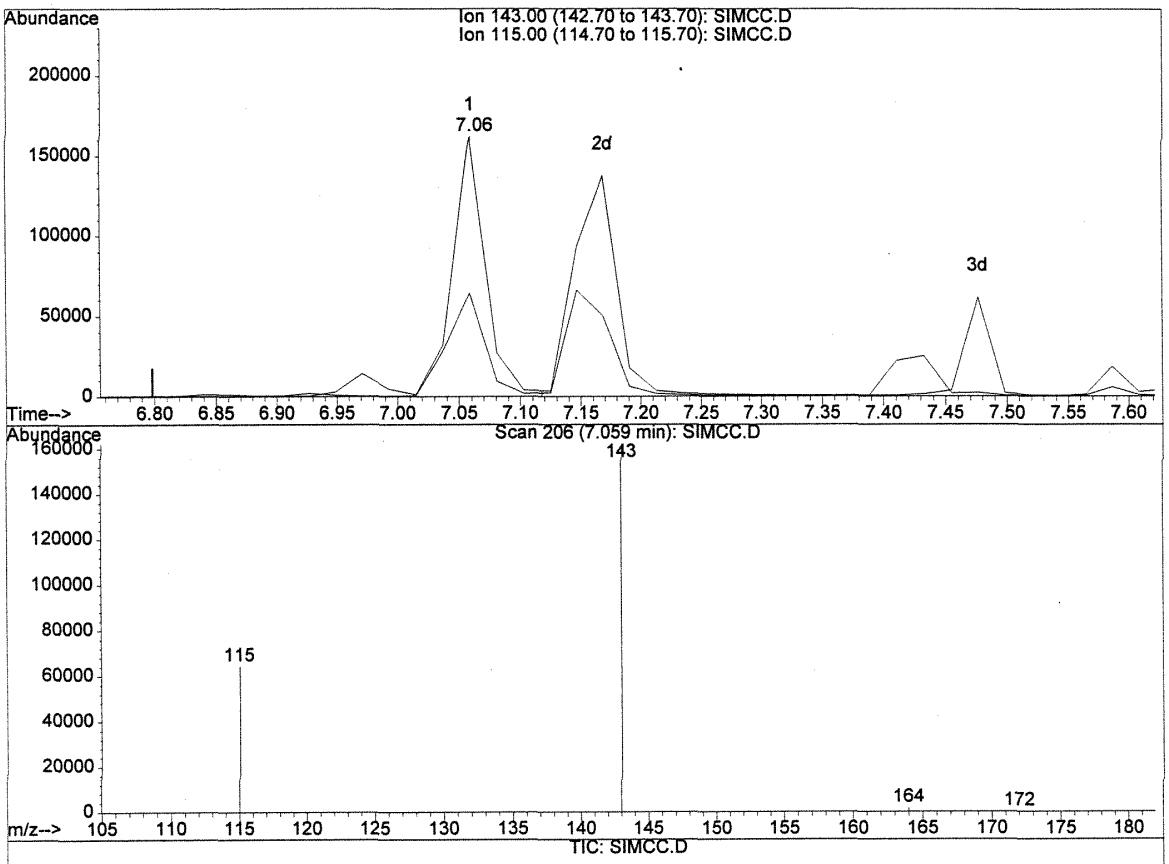
7.06min 4.10ug/ml m

response 450853

Ion	Exp%	Act%
143.00	100	100
115.00	35.90	39.63
0.00	0.00	0.00
0.00	0.00	0.00

Example F

Sample 5 ppm



(3) 2-naphthylamine (T)

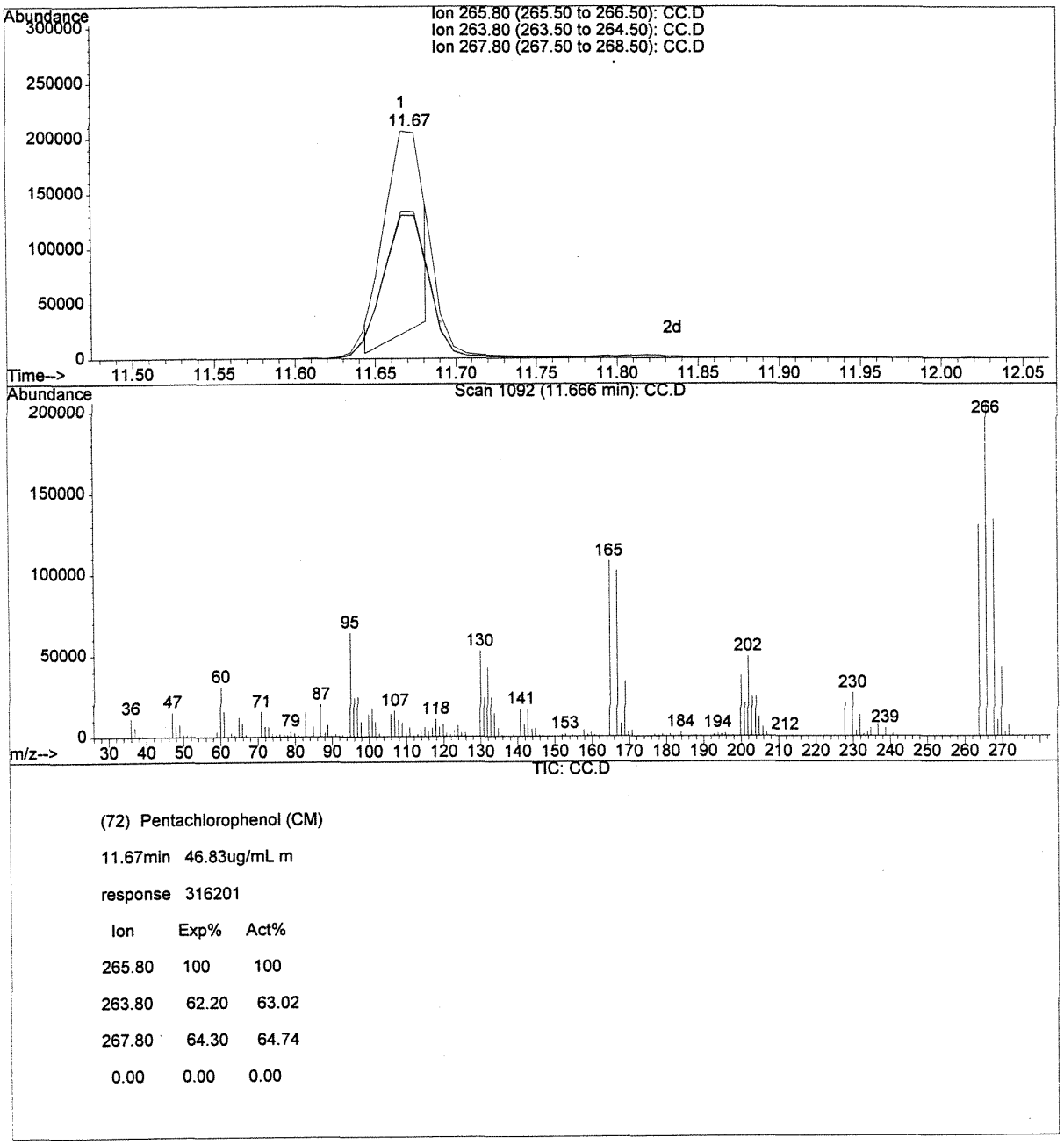
7.06min 27.03ug/ml m

response 2974793

Ion	Exp%	Act%
143.00	100	100
115.00	35.90	6.01#
0.00	0.00	0.00
0.00	0.00	0.00

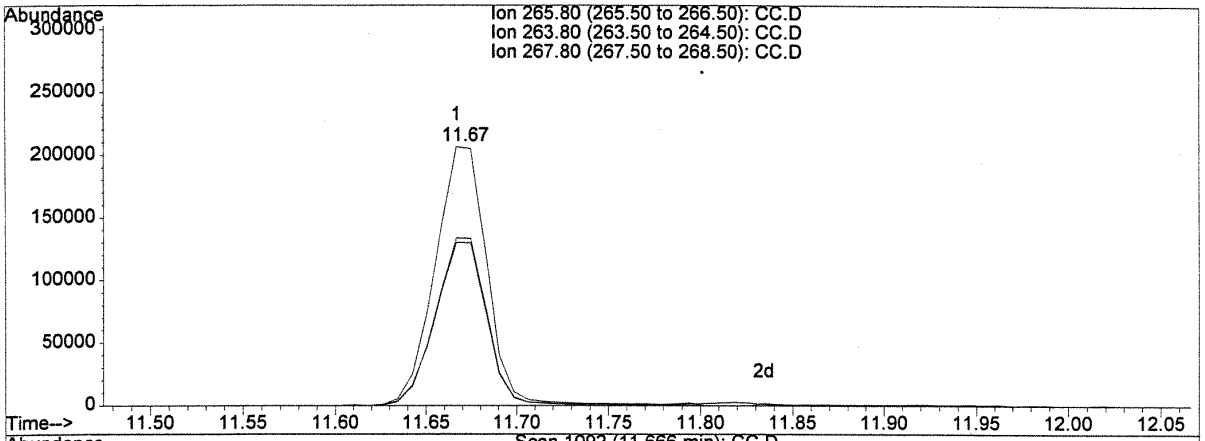
Example G

Sample 40 ppm



Example H

Sample: 40 ppm



(72) Pentachlorophenol (CM)

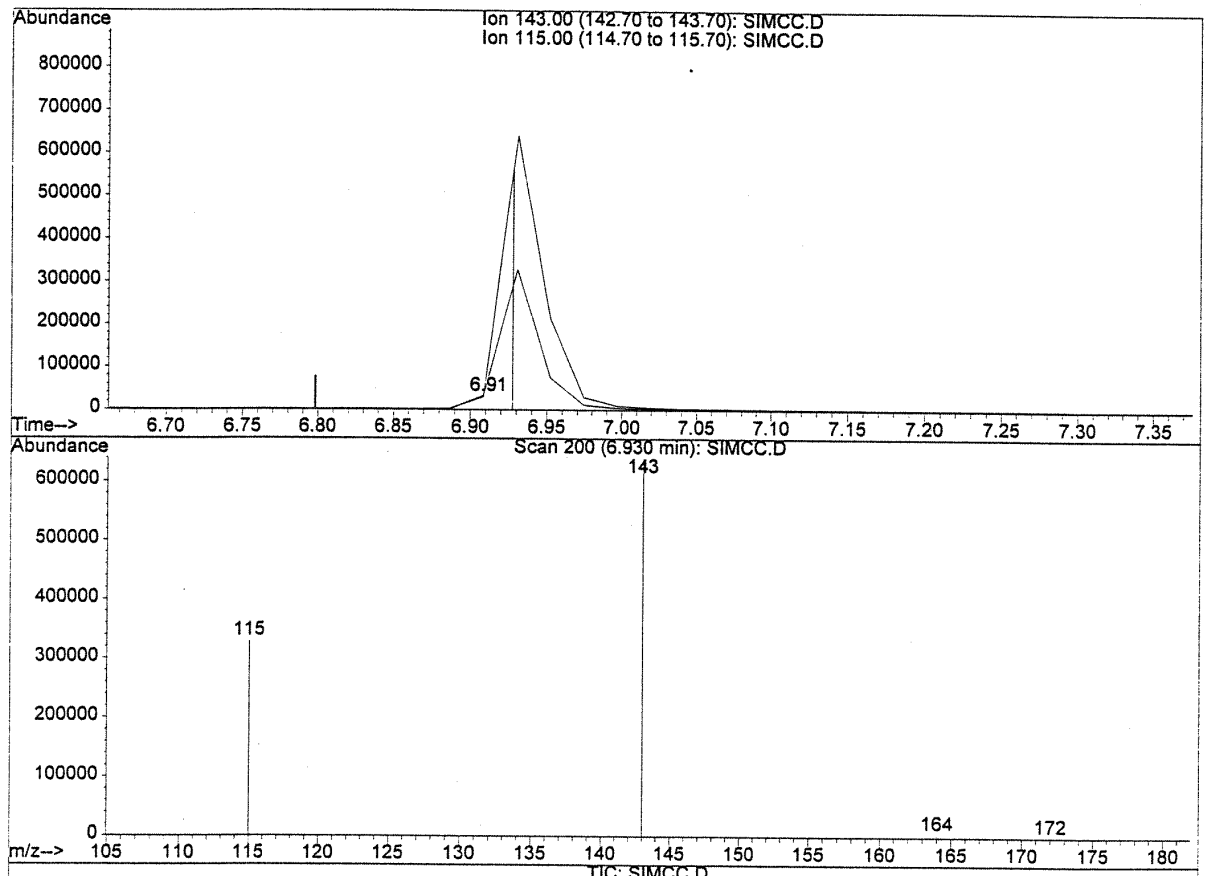
11.67min 610.42ug/mL m

response 4121992

Ion	Exp%	Act%
265.80	100	100
263.80	62.20	63.02
267.80	64.30	64.74
0.00	0.00	0.00

Example I

Sample 5 ppm



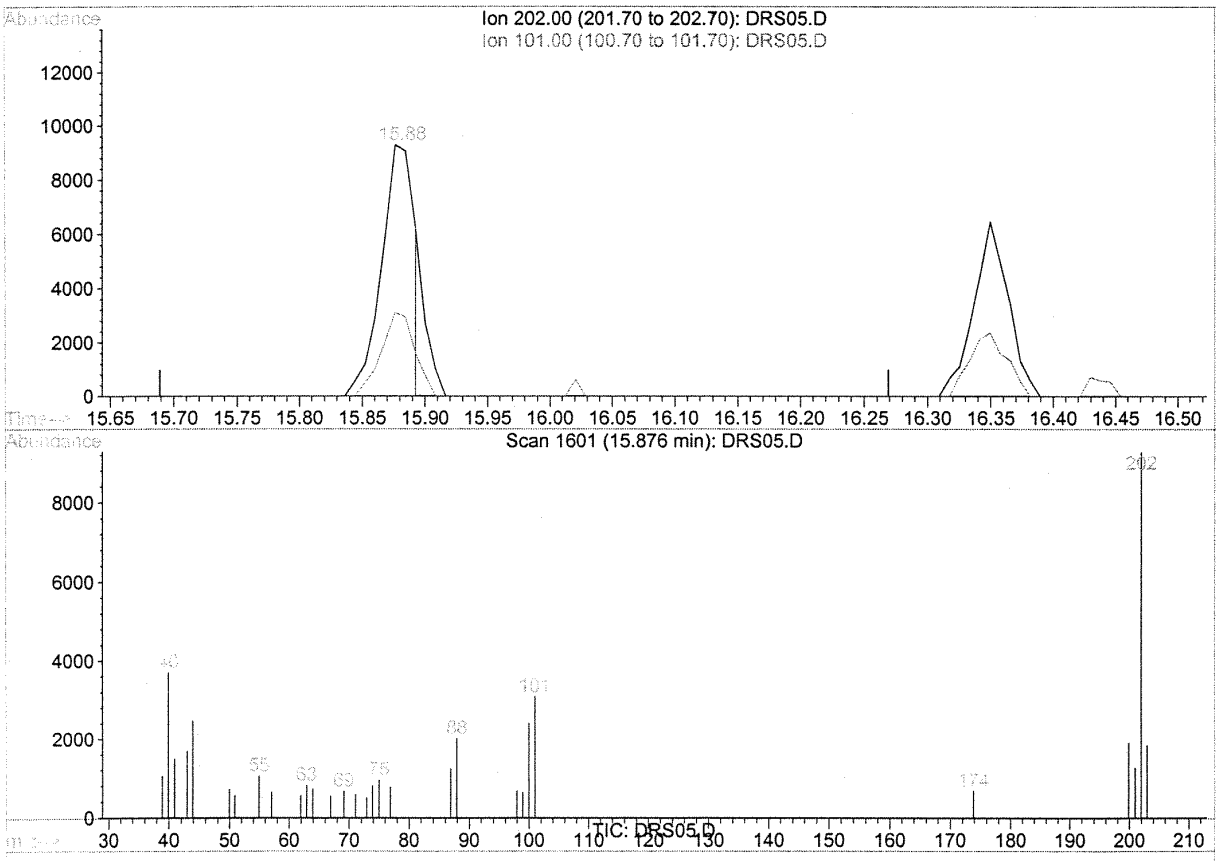
(3) 2-naphthylamine (T)

6.93min 5.60ug/ml m

response 884711

Ion	Exp%	Act%
143.00	100	100
115.00	35.90	51.32#
0.00	0.00	0.00
0.00	0.00	0.00

Example J



(76) Fluoranthene (C)

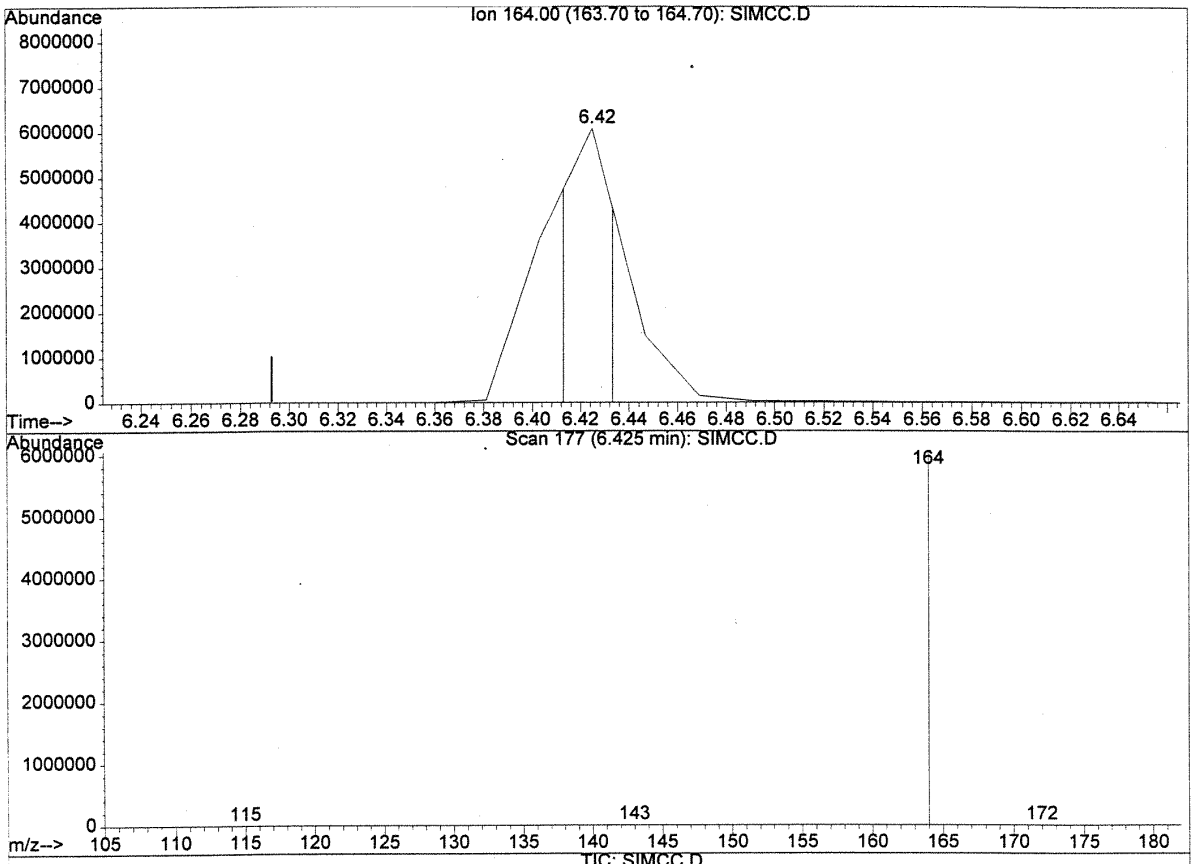
15.88min 0.37ug/mL

response 16974

Ion	Exp%	Act%
202.00	100	100
101.00	28.20	33.28
0.00	0.00	0.00
0.00	0.00	0.00

Example K

Sample 5 ppm



(1) Acenaphthene-d10 (I)

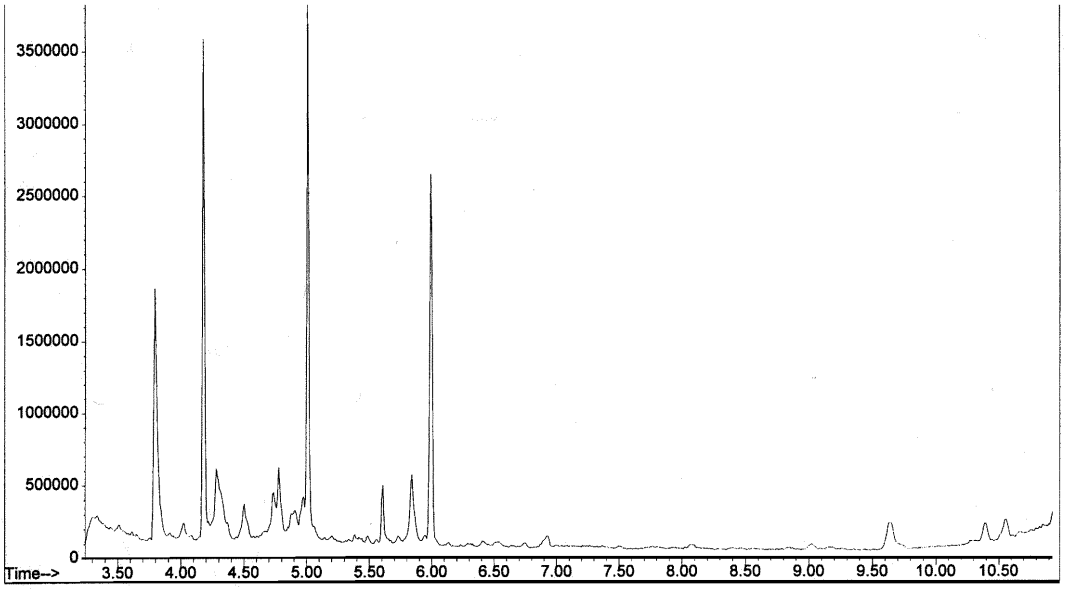
6.42min 40.00ug/ml m

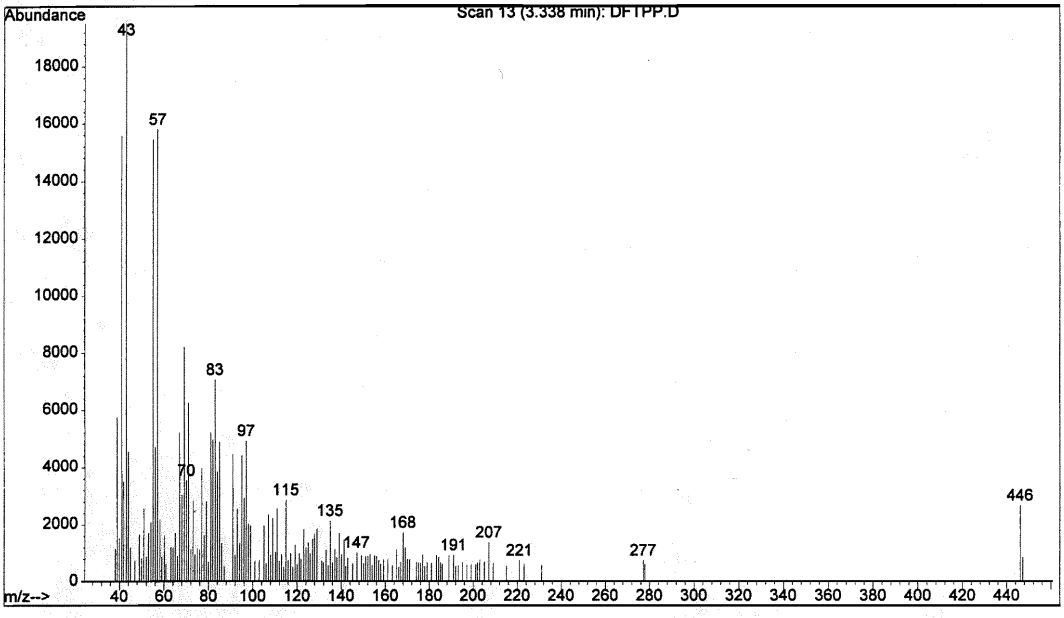
response 8008039

Ion	Exp%	Act%
164.00	100	100
0.00	0.00	0.00
0.00	0.00	0.00
0.00	0.00	0.00

Example L

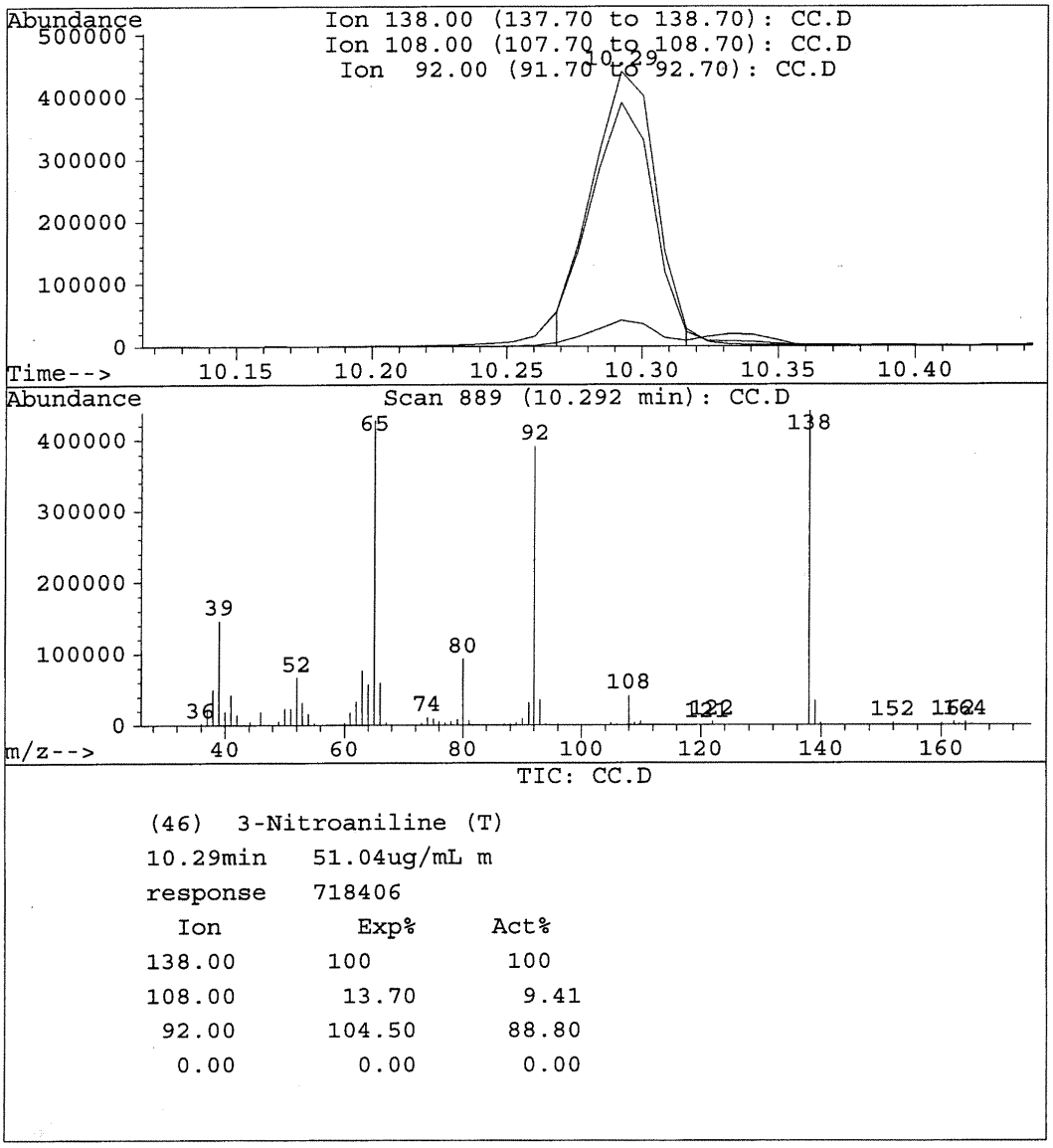
This scan shows an extremely dirty DFTPP chromatogram. There is a significant amount of column bleed and/or carry-over contamination which may lead to the addition of these interferences to the DFTPP peak evaluation.





Example M

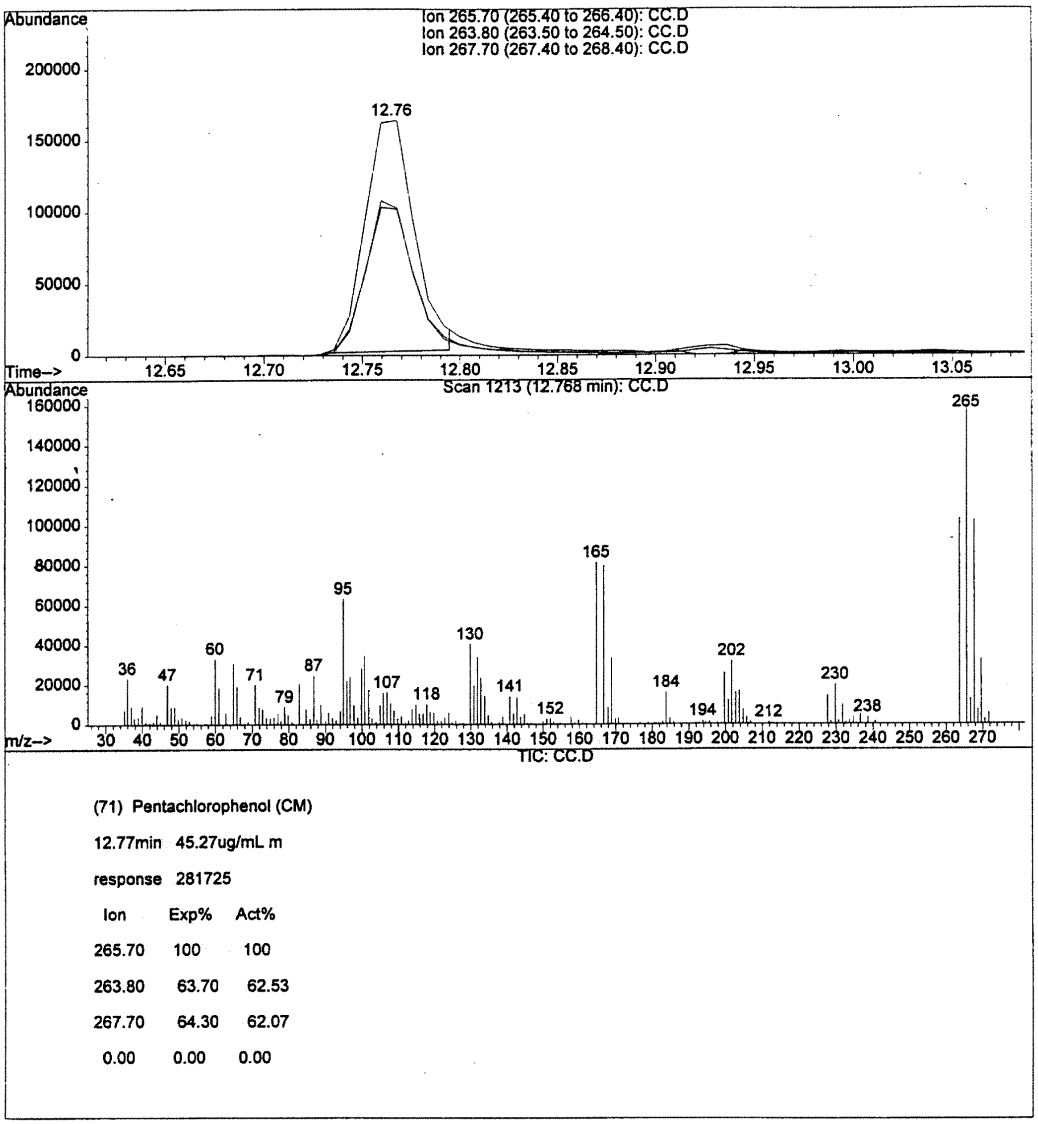
This CCV shows the manual integration of the 3-nitroaniline peak from a retention time of 10.268 to 10.316. This does not include portions of the peak area at the beginning and end of the peak.



Example N

This shows the manual integration of the calibration check compound (CCC) pentachlorophenol, as well as the audit trail for the CCV standard. According to SW846 method 8270B, the CCCs are used to check the validity of the initial calibration. If the percent difference (%D) is greater than 20% then corrective action must be taken prior to the analysis of the samples. The CCCs within the CCV standard are prepared at a concentration of 40 ppm. This allows for an acceptance window of 32 ppm – 48 ppm for this compound.

Review of the chromatogram shows an elevated baseline and the cutting off of portions of the beginning or end of the peaks. The peak shaving results in a decrease in the amount of area integrated and the resulting value for the compound.



Example N Continued

The audit trail shows the computer-generated integration resulted in a value of 51.9517 ppm. This is above the acceptance range. Three manual integrations were performed with the final result of 45.2746 ppm meeting the acceptance criteria.

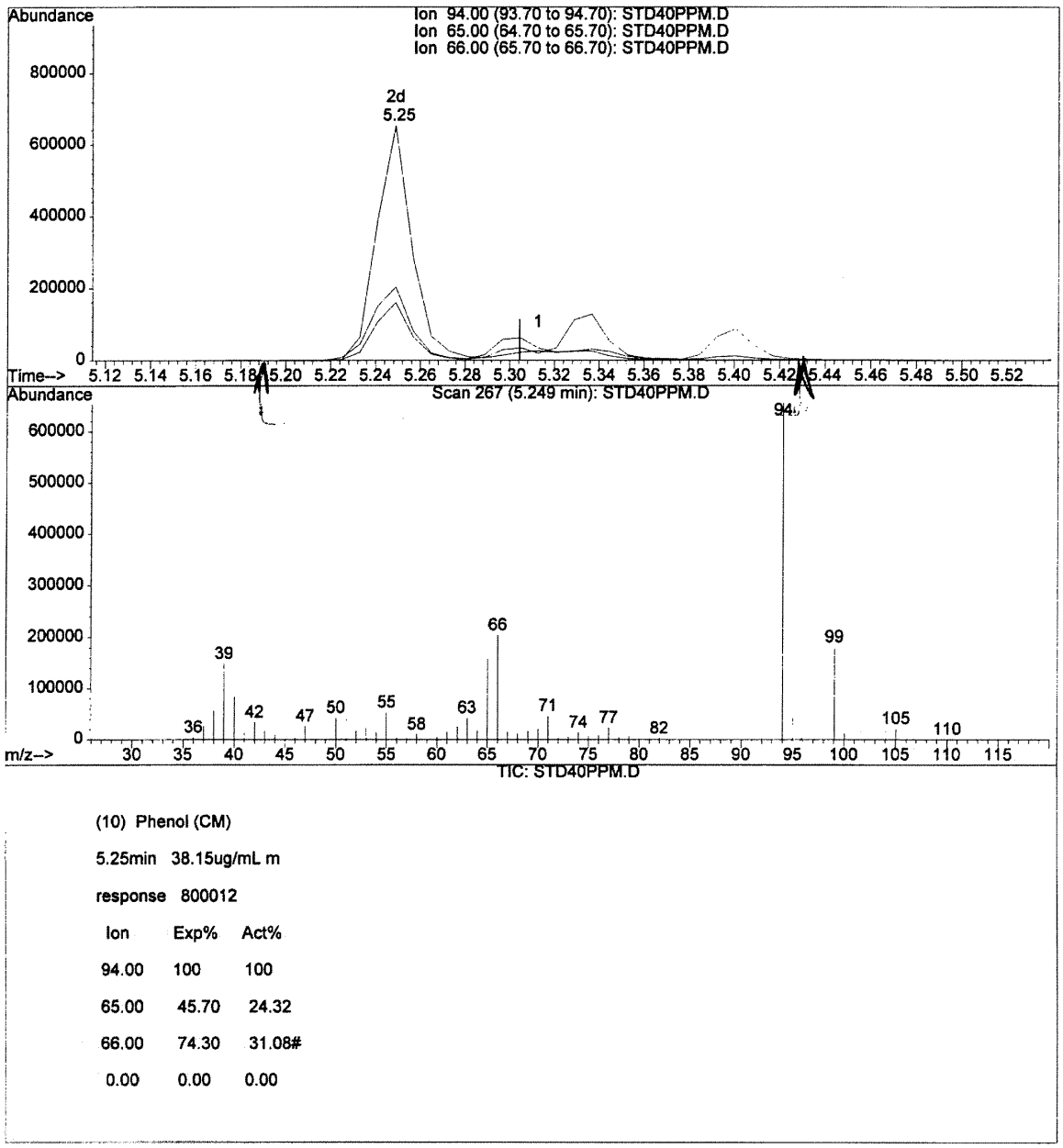
```
 Message  : Changed peak amount for Pentachlorophenol from 51.9517ug/m
L to 48.2194ug/mL
 Severity : 1


 Modified :
 Event    : Manual Integration
 Message  : Changed peak amount for Pentachlorophenol from 48.2194ug/m
L to 44.7427ug/mL
 Severity : 1


 Modified :
 Event    : Manual Integration
 Message  : Changed peak amount for Pentachlorophenol from 44.7427ug/m
L to 45.2746ug/mL
 Severity : 1
```

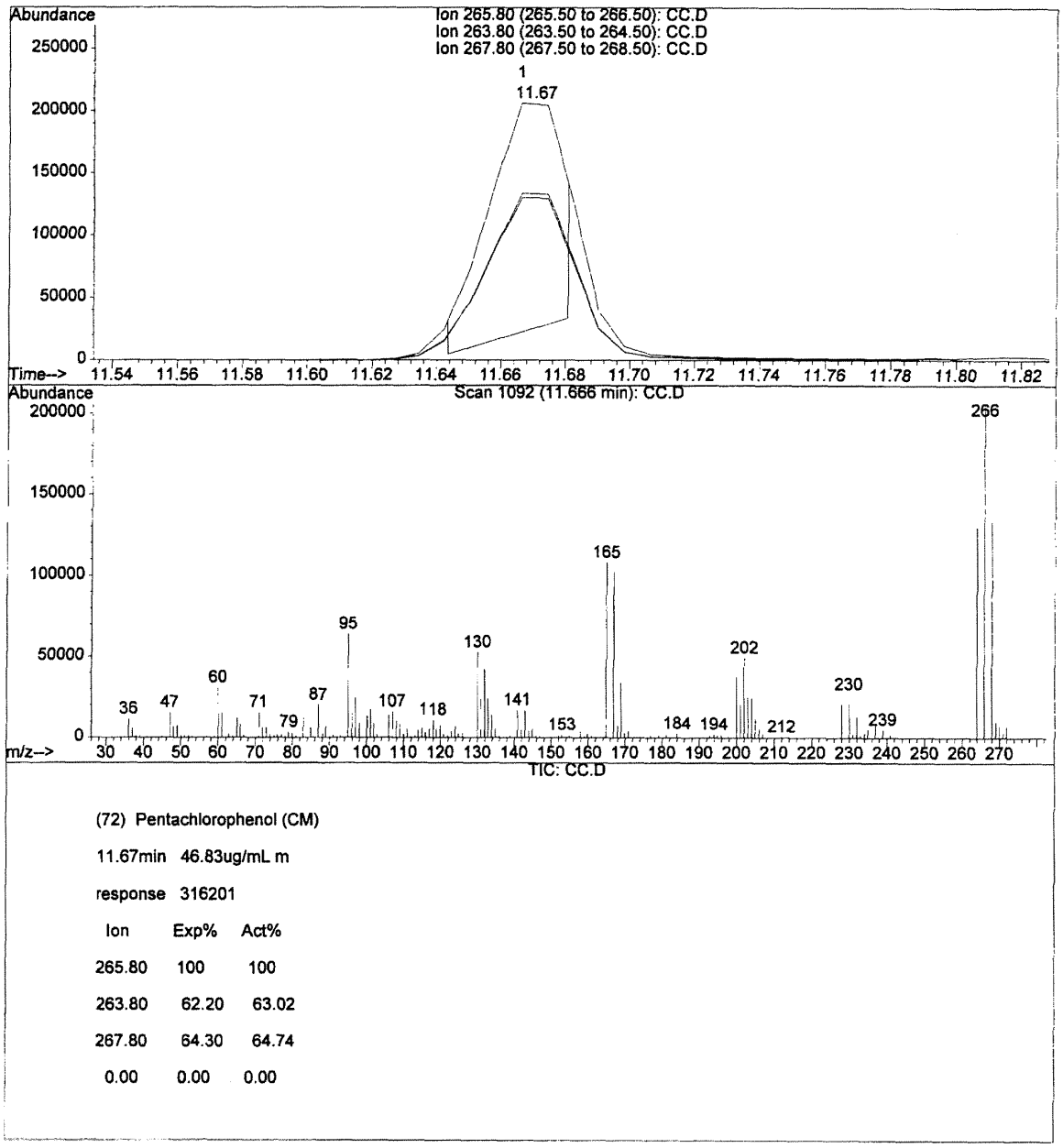
Example O

This shows the data for a 40 ppm standard for phenol that was part of an initial calibration. The peak for phenol was identified at a retention time of 5.25 minutes. The actual integration was performed from 5.19 minutes to 5.43 minutes. The window used included not only the phenol peak, but several additional non-phenol peaks. Thus enhancing the area for the phenol peak and producing a response that was too large.



Example P

This shows the data for a 40 ppm standard for pentachlorophenol that was part of a CCV. The peak was identified at a retention time of 11.67 minutes and the integration was performed from 11.642 minutes to 11.682 minutes. The nonlinear baseline was raised above the actual chromatographic baseline. A large portion of the peak area was excluded (shaved). This shaved area reduced the area for the pentachlorophenol peak, thus producing a lowered response.



6.0 DILUTIONS

Dilutions performed during the analytical process that raise the project reporting limits may render all results unusable. Dilutions, on the other hand, may be necessary, as the laboratory has limited resources to deal with the problem. Labs are limited by having no control over the nature of neither the sample matrix nor the method procedures. Also, dilutions may be required to maintain method compliance. It is important that the laboratories balance as best as possible the project requirements vs. these other factors to use the most appropriate dilution.

In cases where high contamination is suspected prior to analysis, dilutions are acceptable provided that the high concentration is confirmed. For example, a groundwater monitoring well may have a historically high level of a chlorinated solvent and a routine 10-fold dilution may be utilized and no "straight-run" would be required. However, if the dilution shows the target compounds as not detected or detected at the low end of the calibration curve, the sample must be re-analyzed at a lesser dilution.

In cases where high contamination is not known until after the first analysis, dilution would be necessary for target compounds that have exceeded the calibration range. The laboratory should dilute as little as possible so that the target analyte of the highest concentration is above the low end of the calibration curve.

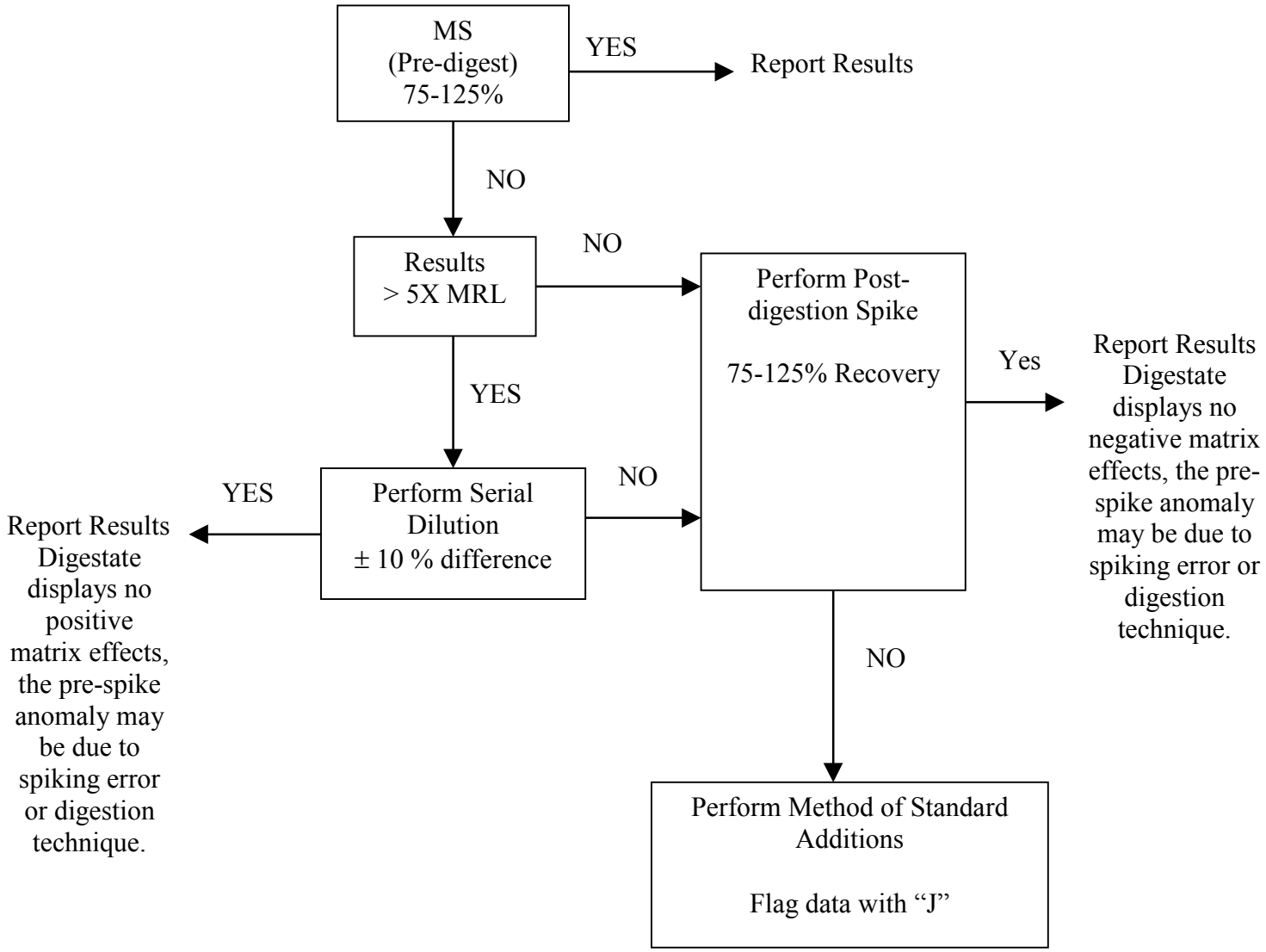
For some other cases, non-target analytes may interfere with the analysis such as a sample contaminated with a petroleum hydrocarbon mixture. When appropriate, clean-up methods are to be used to minimize dilutions. The following procedures are suggestions to deal with the problem. Examine the color and consistency of the supernatant in extraction mixtures after the pre-designated amount of solvent is added to samples (e.g. 60 ml of methylene chloride in 30 g of soil for 8270C extraction.) If the supernatants appear to be opaque, and/or viscous, call the client to discuss how to proceed, and recommend an appropriate clean-up method. After a clean-up, the options are to either use a smaller amount of sample or extract in a greater final volume. Clean-up methods, when used strategically should help the analytical process. The common clean-up methods include:

- GPC
- Silica Gel
- Sulfur
- Acid

Also, the use of GCMS in the Selective Ion Mode (SIM) can be an effective technique to minimize dilutions for specific target analytes. Laboratories are encouraged to discuss alternatives to dilutions as appropriate.

7.0 METHOD OF STANDARD ADDITION/ SERIAL DILUTIONS

The method of standard addition must consist of three analytical spikes and a straight analysis.



8.0 DATA QUALIFIERS

Data qualifier flags are used in an effort to best describe the quality of each piece of data to the data user. These flags are letter codes appended to the numerical data (or in some instances used alone). A series of standard remarks is used to give a more detailed explanation of the data. The following data qualifiers along with the USEPA definitions, modified for this district, will be used during the data validation process.

U - The analyte was analyzed for but not detected. The value preceding the U is the Method Reporting Limit (MRL).

J - The identification of the analyte is acceptable, but the quality assurance criteria indicate that the quantitative values may be outside the normal expected range of precision (i.e. the quantitative value is considered estimated).

R - Data are considered to be rejected and shall not be used. This flag denotes the failure of quality control criteria such that it cannot be determined if the analyte is present or absent from the sample. Re-sampling and analysis are necessary to confirm or deny the presence of the analyte.

UJ - This flag is a combination of the U and J qualifiers which indicates that the analyte is not present. The reported value is considered to be an estimated MRL.

N - There is presumptive evidence that the analyte is present, but it has not been confirmed. The analyte is tentatively identified. There is an indication that the reported analyte is present, however, all quality control requirements necessary for confirmation were not met.

M - Only use the M flag as an indicator of manual integration. The M flag is NOT to be used to qualify the matrix spike.

B - The B flag is to be used for both organic and inorganic analyses when the analyte is found in the associated blank as well as the in the sample. Caution: If using CLP software, override the CLP “B” designation.

E - This flag identifies compounds/analytes whose concentrations exceed the calibration range for that specific analysis.

X - The X flag indicates when more than one qualifier is applied. However, the use of the X flag is not encouraged by the USACE Louisville District.

I - A matrix effect was present

T- The analyte has been "tentatively identified" and the associated numerical value represents its approximate concentration (using GC/MS)

P, C, D, and A Flags are NOT to be used unless prior authorization is given by the USACE Louisville Senior Chemist.

SECTION I
CHEMICAL ANALYSIS CRITERIA

The following procedure has been performed at the Louisville District for Region V. The method reporting limits established can be found in Appendix C in the column with the header "RL".

1 November 2000

MEMORANDUM FOR RECORD

SUBJECT: Method Reporting Limit

1. This memorandum summarizes the various detection limits and outlines a procedure for establishing the reporting limit for projects.

2. MDL: Method Detection Limits are measured by multiplying the student t with the standard deviation (SD) of seven replicates i.e. the MDL is the minimum concentration of a substance that can be measured/detected above zero at 99% confidence in a given matrix. MDLs are determined according to 40 CFR, Part 136. MDL check sample must be performed and be detected at two times the MDL.

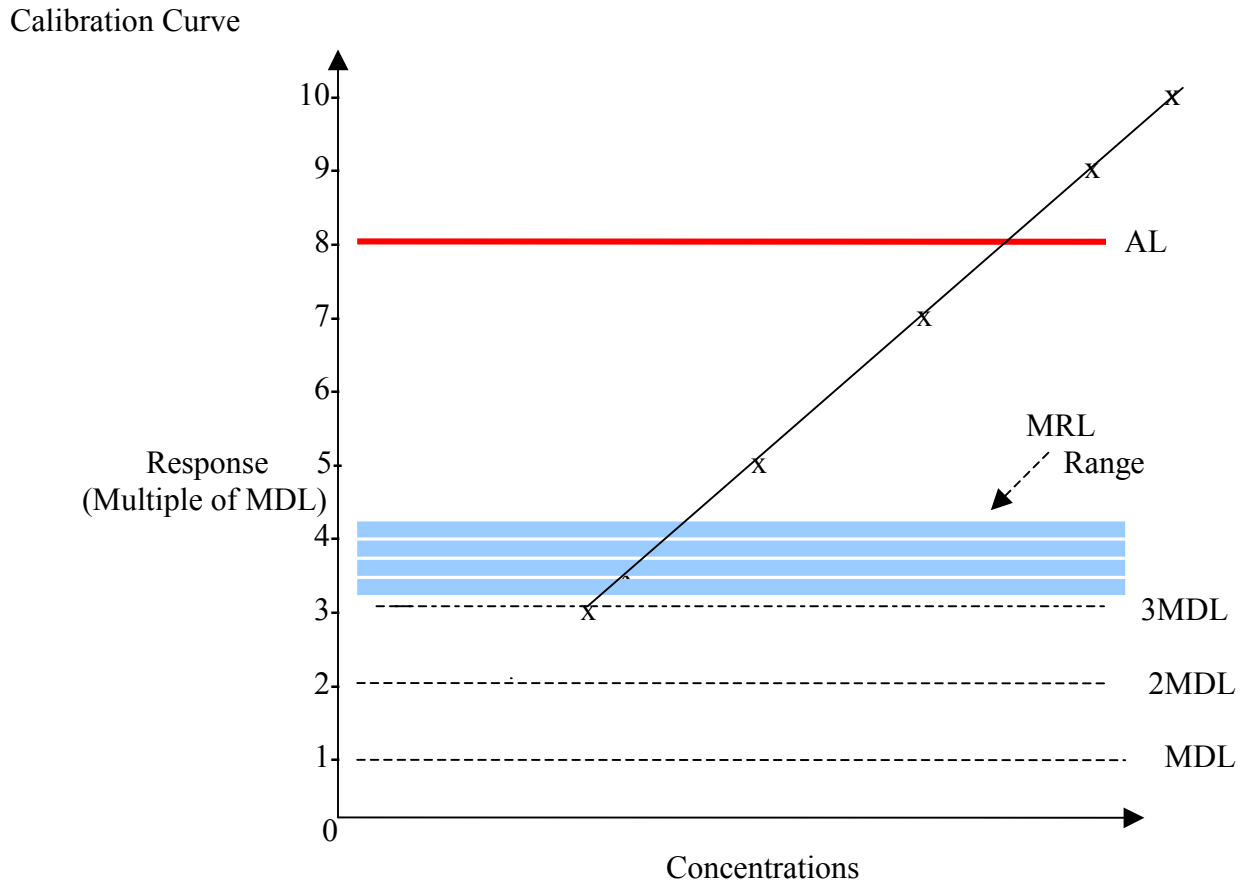
3. MQL: Method Quantitation Limits are established at 3-10 times the MDL levels. The MQLs that were established at such high levels due to the inherited error (± 100%) associated with the results calculated at the MDL. The error associated with the MQL should be comparable to the CCV acceptance limits (± 15%), the statistical error is notably reduced from that of the MDL. Consequently the MQL must be set at the **lowest concentration standard** used for the initial calibration curve. Also, the lowest standard or low-level calibration verification standard must be at least **three times** the MDL. Based on these criteria, analytes detected below the MQL must be flagged with "J" for estimated quantities.

4. MRL: Method Reporting Limits are threshold values above which results are reported as positive quantities, and below which results are reported as non-detect (<) and flagged with "U", or as estimated and flagged with "J". MRL must not be established at levels below the MDL check sample level. MRL value is dependent upon project specifics as discussed in item 6.

5. Relation of MRLs to Action Levels (Als): establishing MRL must be based upon data user, data needs, maximum size of the errors that the user is willing to accept, and the capability of the instrument/method. MRL varies according to project goals, and as a general rule the USACE recommends **the MRLs be established at approximately one-half the project action level.** Therefore, MRL may be established within a range from 3MDL to one-half of the action level. The MRL must have a point on the calibration curve.

6. As an example for explosive analyses, MRL must be established between the MQL (3MDL) and the AL. Accordingly, nitroaromatic compounds detected below the MRL must be reported as non-detect (<), and levels identified below the MRL must not be reported.

7. Compilation of items 2-6 are illustrated on the graph below:



8. For further discussion, please contact the undersigned at 502-315-6324.

SAMIR A. MANSY, Ph.D.
Senior Chemist

SUMMARY OF METHOD QUALITY OBJECTIVES

- Contaminants of concern pertain to VOC and SVOC analyses. Any analyte that may be present at a site should be considered a contaminant of concern. The contaminants of concern must meet the primary evaluation of data as presented in the MQO Tables 1 and 2. No sporadic failure is allowed for the contaminants of concern. Other methods such as metals, PCBs, pesticides, and herbicides, all compounds/analytes on the published target analyte lists are considered contaminants of concern when there is a regulatory risk value associated

- Results for both PCB and Pesticide analysis must be reported from both columns.

- Do not conduct MSD or LCSD for metal analysis.

- Pre-digested, and post-digested metal spikes are required as presented in Section 7.0 Method of Standard Addition/Serial Dilutions.

- A maximum RSD only applies to a few compounds when the mean RSD is met and these compounds should not be among the list of contaminants of concern.

- For metals anlaysis: the r value is utilized due to the fact that the calibration curve consists of at least three points and a blank (depending on the method ie: ICP, GFAA, CVAA and Cyanide).

Table 1
Summary of Method Quality Objectives for Method 8260 - Volatile Organic Compounds

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
BFB Tuning	Ion abundance relative to m/z 95 base peak	Every 12 hours	Method criteria	Correct problem and rerun	No flagging criteria – MUST RETUNE before analysis of samples. Reject all data.
Initial Calibration	Minimum five point curve.		Instrument Evaluation: SPCC: minimum RF values per method requirements CCCs: verify RSD ≤ 30% And All analytes: r ≥ 0.99, RSD ≤ 15%, r^2 ≥ 0.99, mean RSD ≤ 15% with a maximum RSD ≤ 20%, for upto 5% for any non-contaminant of concern)	Correct problem and rerun. There should be no failures for an ICAL– correct and repeat is the corrective action otherwise it will be rejected.	Qualify with R when less than five std. were used. When RSD exceeds 15% or correlation is less than 0.99 -J/R. Manual integration is NOT acceptable for initial calibrations, except for the low level calibration concentrations (1st and 2nd point of the calibration curve). Refer to guideline in Section 5.0.
MDL Level Verification	MDL check (2XMDL)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence, every 12 hours, or Shorter to bracket USACE samples	70-130% Sporadic Marginal Failure 60-140%	Correct problem and repeat. Or run MDL check to verify the non-detect	For results greater than the 130%, apply J to all positives. For a result <70% and >60%, apply J to all positives and UJ to all non-detects. For recovery <60%, apply J to all positives and R to all non-detects. If MDL check was run at end and acceptable do not reject data.

Notes: Sporadic Marginal Failure accumulation is allowed up to 5% of the target list for non-contaminants of concern before corrective action must be taken.

Table 1 (continued)
Summary of Method Quality Objectives for Method 8260 -Volatile Organic Compounds

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	80-120% Sporadic Marginal Failure 60-140%	Correct problem and repeat.	For results greater than the 120%, apply J to all positives. For a result <80% and >60%, apply J to all positives and UJ to all non-detects. For recovery <60%, apply J to all positives and R to all non-detects.
CCV	Mid-level verification	At the beginning of every twelve hours	Instrument Evaluation: SPCC: minimum RF values per method requirements CCC: verify D ≤ 30% And D or mean D ≤ 20% with a max D for each target analyte ≤ 30% for upto 5% for any non-contaminants of concern. Internal Standards: Retention time ±30 seconds from retention time of the mid-point std. of the ICAL. EICP area within –50% to +100% of the ICAL mid-point std.	Correct the problem, reanalyze CCV, if problem continues repeat initial calibration	If mean %D > 20% and D > 30% then R. If mean %D ≤ 20% and D > 30% then J. For each target analyte, when D>20% with a negative bias -J/R. When D>30% with a positive bias –J positives only.
Internal Standards	Analytically spiked non-target compounds used to calculate the concentration of target compounds present	Every sample	Retention time ±30 seconds from retention time of the mid-point std. in the CCC for the sequence. EICP area within –50% to +100% of the CCC mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	For area counts between 10% and 50% or >150%, apply J/UJ. For areas <30%, reject all associated data.

Notes: Sporadic Marginal Failure accumulation is allowed up to 5% of the target list for non-contaminants of concern before corrective action must be taken.

Table 1 (continued)
Summary of Method Quality Objectives for Method 8260 -Volatile Organic Compounds

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MB	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL (up to 5% may exceed)	Reprep. And repeat for >5% exceedance	Apply B to all associated positives when less than 5X blank concentration and 10X for common lab contaminants (acetone, chloroform, and methylene chloride)
LCS	Interference-free matrix containing all target analytes	1 per sample batch	See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery LL and <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	Only if [spike] > 4X[matrix] 70 – 130% Sporadic Marginal Failure 60-140%		No action is taken based on MS recovery alone, use professional judgment.
MSD	Use MD or MS	1 per sample batch	Only apply to results > MRL RPD ≤ 30% Sporadic Marginal Failure ≤ 40%		No action is taken based on duplicate results alone, use professional judgment.
Surrogates		Per sample	50%-150%	Rerun samples and/or re-extract when <10%.	When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R.

Notes: Sporadic Marginal Failure accumulation is allowed up to 5% of the target list for non-contaminants of concern before corrective action must be taken.

Table 2
Summary of Method Quality Objectives for Method 8270 - Semivolatile Organic Compounds

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
DFTPP Tuning	Ion abundance relative to m/z 198 base peak	Every 12 hours	Method Criteria	Correct Problem and rerun	No flagging criteria – MUST RETUNE before analysis of samples Reject all data.
Initial Calibration	Minimum five point curve.		Instrument Evaluation: SPCC: minimum RF values per method requirements CCCs: RSD ≤ 30% AND All analytes: r ≥ 0.99, RSD ≤ 15%, $r^2 \geq 0.990$, or mean RSD ≤ 15% with a maximum RSD ≤ 20% for up to 5% of the non-contaminants of concern for the target list	Correct problem and rerun. There should be no failures for an ICAL – correct and repeat is the corrective action otherwise it will be rejected.	Qualify with R when less than five std. were used. When RSD exceeds 15% or correlation is less than 0.99-J/R. Manual integration is NOT acceptable for initial calibrations, except for the low level calibration concentrations (1st and 2nd point of the calibration curve). Refer to guideline in Section 5.0.
MDL Level Verification	MDL check (2XMDL no extraction)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence, every 12 hours, or Shorter to bracket USACE samples	70-130% Sporadic Marginal Failure 50-150%	Correct problem and repeat. OR run MDL check to verify the non-detect	For results greater than the 130%, apply J to all positives. For a result <70% and >50%, apply J/UJ. For recovery <50%, apply J/R. If MDL check was run at end and acceptable do not reject data.

Notes: Sporadic Marginal Failure accumulation is allowed up to 5% of the target list for non-contaminants of concern before corrective action must be taken.

Table 2 (continued)
Summary of Method Quality Objectives for Method 8270 - Semivolatile Organic Compounds

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	70-130% Sporadic Marginal Failure 50-150%	Correct problem and repeat.	For results greater than the 130%, apply J to all positives. For a result <70% and >50%, apply J/UJ. For recovery <50%, apply J/R.
CCV	Mid-level verification	Every twelve hours	Instrument Evaluation: SPCC: minimum RF values per method requirements CCC: verify D ≤ 20% And D or mean D ≤ 20% with a max D for each target analyte ≤ 30% for upto 5% for any non-contaminants of concern. Internal Standards: Retention time ±30 seconds from retention time of the mid-point std. of the ICAL. EICP area within –50% to +100% of the ICAL mid-point std.	Correct the problem, reanalyze CCV, if problem continues repeat initial calibration	If mean %D > 20% and D > 30% then R. If mean %D ≤ 20% and D > 30% then J. For each target analyte, when D>20% with a negative bias -J/R. when D>20% with a positive bias –J positives only.
Internal Standards	Analytically spiked non-target compounds used to calculate the concentration of target compounds present	Every sample	Retention time ±30 seconds from retention time of the mid-point std. in the CCC for the sequence. EICP area within –50% to +100% of the CCC mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	For area counts between 30% and 50% or >150%, apply J/UJ. For areas <30%, reject all associated data.

Notes: Sporadic Marginal Failure accumulation is allowed up to 5% of the target list for non-contaminants of concern before corrective action must be taken.

Table 2 (continued)
Summary of Method Quality Objectives for Method 8270 - Semivolatile Organic Compounds

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MB	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL (up to 5% may exceed)	Reprep and repeat for >5% exceedance	Apply B to all associated positives when less than 5X blank concentration and 10X for common lab contaminants (phthalates)
LCS	Interference-free matrix containing all target analytes	1 per sample batch	See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL flag J/UJ. For a recovery <LL and <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	Only if [spike] > 4X[matrix] 45-135% Sporadic Marginal Failure 20-150%		No action is taken based on MS recovery alone, use professional judgment.
MSD	Use MD or MS	1 per sample batch	Only apply to results > MRL Water: RPD ≤ 40% Soil: RPD ≤ 50% SMF: ≤ 50%		No action is taken based on duplicate results alone, use professional judgment.
Surrogates		Per sample	50-150%	Rerun samples and/or re-extract when <10%.	When recovery is >UL, flag positives with J. For recovery between 10% and the LL, flag J/UJ. For surrogate recovery <10%, flag J/R.

Notes: Sporadic Marginal Failure accumulation is allowed up to 5% of the target list for non-contaminants of concern before corrective action must be taken.

Table 3
Summary of Method Quality Objectives for Method 8082 - PCBs

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration (Aroclors 1016 & 1260)	Minimum five point curve.		$r \geq 0.99$, RSD $\leq 20\%$, $r^2 \geq 0.990$, mean RSD $\leq 20\%$	Correct problem and rerun.	Qualify with R when less than five std. Were used. When RSD exceeds 20% or correlation is less than 0.99 -J/R. Manual integration is NOT acceptable for initial calibrations, except for the low level calibration concentrations (1st and 2nd point of the calibration curve). Refer to guideline in Section 5.0.
MDL Level Verification	MDL check (2XMDL no extraction)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence, every 12 hours, or Shorter to bracket USACE samples	70-130%	Correct problem and repeat. . Or run MDL check to verify the non-detect	For results >130%, apply J only. For a result marginally less than the LL (65-70%), apply J/UJ. For recovery more than marginally out (<65%), apply J/R. If MDL check was run at end and acceptable do not reject data.
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	85-115%	Correct problem and repeat.	For results >115%, apply J only. For a result marginally less than the LL (80-85%), apply J/UJ. For recovery more than marginally out (<80%), apply J/R.
CCV	Mid-level verification	Every twelve hours	D or mean D $\leq 15\%$ with a maximum %D<20% for upto 5% of the calibrated peaks.	Correct problem and rerun.	For each target analyte, when the mean D>20% with a negative bias -J/R. when D>15% with a positive bias for any of the calibrated peaks for each arochlor–J positives only.

Table 3 (continued)
Summary of Method Quality Objectives for Method 8082 - PCBs

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MB	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL (up to 5% of the total list of analytes may exceed)	Reprep and repeat for >5% exceedance	Apply B to all associated positives when less than 5X blank concentration
LCS	Interference-free matrix containing all target analytes	1 per sample batch	See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery <LL and <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	To consider [spike] > 4X[matrix] 50-150%		No action is taken based on MS recovery alone, use professional judgment.
MSD	Use MD or MS	1 per sample batch	Only apply to results > MRL Water: RPD ≤ 50% Soil: Lab QC Limits		No action is taken based on duplicate results alone, use professional judgment.
Surrogates			50-150%	Rerun samples and re-extract when <10%.	When recovery is >150% or between 10% and 50%, flag positives with J. For non-detect associated results where recovery is between 10% and 50%, flag UJ. For an analyte recovery <10%, flag R.
Target Analyte Confirmation	All positive results greater than the MRL.	Whenever a positive is detected on one column and confirmed on 2nd column.	RPD ≤ 40		Qualify with J when RPD>40 for positive results.

Table 4
Summary of Method Quality Objectives for Method 8081 - Pesticides

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
DDT/Endrin % Breakdown	DDT & Endrin Breakdown	Every twelve hours	Degradation ≤ 15%	Maintenance and rerun.	Flag results for DDT or endrin and breakdown components with J/R.
Initial Calibration	Minimum five point curve.		r ≥ 0.99, RSD ≤ 20%, r^2 ≥ 0.990, or mean RSD ≤ 20% for upto 5% of the non-contaminants of concern.	Correct problem and rerun.	Qualify with R when less than five std. were used. When RSD exceeds 20% or correlation is less than 0.99 -J/R. Manual integration is NOT acceptable for initial calibrations, except for the low level calibration concentrations (1st and 2nd point of the calibration curve). Refer to guideline in Section 5.0.
MDL Level Verification	MDL check (2XMDL no extraction)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence, every 12 hours, or Shorter to bracket USACE samples	70-130%	Correct problem and repeat. . Or run MDL check to verify the non-detect	For results >130%, apply J only. For a result marginally less than the LL (65-70%), apply J/UJ. For recovery more than marginally out (<65%), apply J/R. If MDL check was run at end and acceptable do not reject data.
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	85-115%	Correct problem and repeat.	For results >115%, apply J only. For a result marginally less than the LL (80-85%), apply J/UJ. For recovery more than marginally out (<80%), apply J/R.
CCV	Mid-level verification	Every twelve hours	D ≤ 15%, mean D ≤15% with a max D ≤20% for upto 5% of the non-contaminants of concern.	Correct problem and rerun.	For each target analyte, when D>15% with a negative bias -J/R. when D>15% with a positive bias –J positives only.

Table 4 (continued)
Summary of Method Quality Objectives for Method 8081- Pesticides

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MB	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL (up to 5% of the total list of analytes may exceed)	Reprep and repeat for >5% exceedance	Apply B to all associated positives when less than 5X blank concentration
LCS	Interference-free matrix containing all target analytes	1 per sample batch	See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL flag J/UJ. For a recovery <LL and <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	To consider [spike] > 4X[matrix]:50-150%		No action is taken based on MS recovery alone, use professional judgment.
MSD	Use MD or MS	1 per sample batch	Only apply to results > MRL Water: RPD ≤ 50% Soil: Lab QC Limits		No action is taken based on duplicate results alone, use professional judgment.
Surrogates			50-150%	Rerun samples or re-extract when <10%.	When recovery is >150% or between 10% and the 50%, flag positives with J. For non-detect associated results where recovery is between 10% and the 50%, flag UJ. For an analyte recovery <10%, flag R.
Target Analyte Confirmation	All positive results greater than the MRL.	Whenever a positive is detected on one column and confirmed on 2nd column.	RPD ≤ 40		Qualify with J when RPD>40 for positives results.

Table 5
Summary of Method Quality Objectives for Method 8330 -Explosives

Quality Control Element	Description of Element	Frequency Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration (IC)	Minimum five point Curve		$r \geq 0.99$, RSD $\leq 20\%$, $r^2 \geq 0.990$, or mean RSD $\leq 20\%$	Correct problem and rerun.	Qualify with R when less than five std. Were used. When RSD exceeds 20% or correlation is less than 0.99 -J/R. Manual integration is NOT acceptable for initial calibrations, except for the low level calibration concentrations (1st and 2nd point of the calibration curve). Refer to guideline in Section 5.0.
MDL Level Verification	MDL check (2XMDL no extraction)	Once per quarter per instrument	Analytes detected	Conduct Sensitivity Test	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level Check standard at MRL	At the beginning and end of every daily sequence, every 12 hours, or Shorter to bracket USACE samples	QC/MRL: D ≤ 30%	Correct problem and repeat. . Or run MDL check to verify the non-detect	For results >130%, apply J only. For a result marginally less than the LL (65-70%), apply J/UJ. For recovery more than marginally out (<65%), apply J/R. If MDL check was run at end and acceptable do not reject data.
ICV Initial calibration Verification	Mid-level (2nd source) Verification	After initial Calibration	Recovery = 85 - 115%	Correct problem, repeat IC or ICV	For results >115%, apply J only. For a result marginally less than the LL (80-85%), apply J/UJ. For recovery <65% apply J/R.
CCV	Mid-level verification	Daily	D ≤ 15%, mean D ≤15% with a max D ≤20% for upto 5% of the non-contaminants of concern.	Correct problem and rerun.	For each target analyte, when D>15% with a negative bias -J/R. when D>15% with a positive bias –J positives only.

Table 5 (continued)
Summary of Method Quality Objectives for Method 8330 - Explosives

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MB	Reagent Blank to assess contamination	1 per sample batch	Target Analytes: Analytes < 1/2MRL	Re-prep, Repeat.	Apply B to all associated positives when less than 5X blank concentration
LCS	Interference-free matrix containing all target analytes		See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery <LL and <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to extraction	1 per sample batch	To consider [spike] > 4X[matrix]:50-150%		No action is taken based on MS recovery alone, use professional judgment.
MSD/MD	Sample matrix spiked with all target analytes prior to extraction	1 per sample batch	Only apply to results > MRL Water: RPD ≤ 50% Soil: Lab QC Limits		No action is taken based on duplicate results alone, use professional judgment.
Surrogates			50-150%	Rerun samples or re-extract when <10%.	When recovery is >150% or between 10% and the 50%, flag positives with J. For non-detect associated results where recovery is between 10% and the 50%, flag UJ. For an analyte recovery <10%, flag R.
Target Analyte Confirmation	All positive results greater than MRL	Whenever a positive is detected on one column and confirmed on 2nd column.	RPD ≤ 40		Qualify with J when RPD>40 for positive results.

Table 6
Summary of Method Quality Objectives for Method 8310 -Polyaromatic Hydrocarbons

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	Minimum five point curve.		R ≥ 0.99, RSD ≤ 20%, r^2 ≥ 0.990, mean RSD ≤20%	Correct problem and rerun.	Qualify with R when less than five std. were used. When RSD exceeds 20% or correlation is less than 0.99 -J/R. Manual integration is NOT acceptable for initial calibrations, except for the low level calibration concentrations (1st and 2nd point of the calibration curve). Refer to guideline in Section 5.0.
MDL Level Verification	MDL check (2XMDL no extraction)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence, every 12 hours, or Shorter to bracket USACE samples	70-130%	Correct problem and repeat. . Or run MDL check to verify the non-detect	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (65-70%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<65%), apply J to all positives and R to all non-detects. If MDL check was run at end and acceptable do not reject data
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	85-115%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (80-85%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<80%), apply J to all positives and R to all non-detects.
CCV	Mid-level verification	Every twelve hours and at the end of sequence	D ≤ 15% , mean D ≤ 15% with a max D ≤ 20% for upto 5% of non-contaminants of concern.	Correct problem and rerun.	For each target analyte, when D>15% with a negative bias -J/R. When D>15% with a positive bias –J positives only.

Table 6 (continued)
Summary of Method Quality Objectives for Method 8310 - Polyaromatic Hydrocarbons

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MB	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL (up to 5% of total list of anlaytes may exceed)	Reprep and repeat for >5% exceedance	Apply B to all associated positives when less than 5X blank concentration
LCS	Interference-free matrix containing all target analytes	1 per sample batch	See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL flag J/UJ. For a recovery<LL and <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to extraction	1 per sample batch	To consider [spike] > 4X[matrix]:50-150%		No action is taken based on MS recovery alone, use professional judgment.
MSD	Use MD or MS	1 per sample batch	Only apply to results > MRL Water: RPD ≤ 50% Soil: Lab QC Limits		No action is taken based on duplicate results alone, use professional judgment.
Surrogates			50-150%	Rerun samples or re-extract when <10%.	When recovery is >150% or between 10% and the 50%, flag positives with J. For non-detect associated results where recovery is between 10% and the 50%, flag UJ. For an analyte recovery <10%, flag R.
Target Analyte Confirmation	All positive results greater than MRL	Whenever a positive is detected on one detector and confirmed on 2nd detector.	RPD ≤ 40		Qualify with J when RPD>40 for positive results.

Table 7
Summary of Method Quality Objectives for Method 6010 - ICP Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	3-stds and a blank	Daily	R ≥ 0.995	Correct problem and repeat.	Apply R to all associated results when not performed daily. When correlation is less than 0.995 -J/R.
Instrument Precision	RSD 3 integration's (exposures)	Each calibration and calibration verification standard (ICV/CCV)	RSD<5%	Correct problem and repeat.	Apply R to all associated results.
MDL Level Verification	MDL check (2XMDL no digestion)	Once per quarter per instrument used	Analytes detected	Correct MDL and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL [Common Elements can be between the MRL and 2X MRL level (Fe, Al, Mg and Ca)]	At the beginning and end of every daily sequence or shorter to bracket USACE samples	70-130%	Correct problem and repeat. Or run MDL check to verify the non-detect	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (65-70%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (65%), apply J to all positives and R to all non-detects. If MDL check was run at end and acceptable do not reject data
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	90-110%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (85-90%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<85%), apply J to all positives and R to all non-detects.
Initial Calibration Blank(ICB)	Interference-free matrix to assess analysis contamination	After initial calibration verification	Analytes <1/2 MRL (up to 5% may exceed)	Correct problem and repeat.	Apply U to all associated positives when less than 5X blank concentration

Table 7 (continued)
Summary of Method Quality Objectives for Method 6010 - ICP Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Interelement Check Standard (ICS)	ICS-A interferents only ICS-AB interferents and target analytes	Beginning of analytical sequence	ICSA: No detection of other elements: analytes <1/2 MRL (up to 5% may exceed) ICSAB: 80-120% for target analytes	Correct problem and repeat.	Only for samples with concentrations approximate to [ICS]. For recovery >120%, apply J to positives. For recovery in between 50-79%, apply J to positives and UJ to non-detects. For recovery <50%, apply R. For detections of other elements, use professional judgment.
Continuing Calibration Blank (CCB)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes <1/2 MRL (up to 5% may exceed)	Correct problem and repeat.	Apply U to all associated positives when less than 5X blank concentration
Continuing Calibration Verification(CCV)	Mid-level verification	Every 10 samples and at end of analytical sequence	90-110%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (85-90%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<85%), apply J to all positives and R to all non-detects.
Method Blank (MB)	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL (up to 5% may exceed)	Redigest and repeat.	Apply B to all associated positives when less than 5X blank concentration
Laboratory Control Sample (LCS)	Interference-free matrix containing all target analytes	1 per sample batch	See limits in Appendix C	Correct problem and repeat.	When recovery is >UL flag positives with J. For recovery <LL flag J/UJ. For a recovery <LL and <60%, flag J/R.

Table 7 (continued)
Summary of Method Quality Objectives for Method 6010, ICP Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Matrix Spike (MS) (pre-digested)	Sample matrix spiked with all of target analytes prior to digestion	1 per sample batch	To consider [spike] > 4X[matrix] Recovery=75-125%	See Section 7	For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. Associated with an analyte recovery <30%, flag non-detects R and positives J.
Matrix Duplicate (MD)	A USACE sample digested in duplicate	1 per sample batch	RPD ≤ 20		Flag all positives for the analytes out with a J in associated samples.
Post Digestion Spike	Sample digestate spiked with all of target analytes	As needed to confirm matrix effect	Recovery=75-125%	See Section 7	For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag R.
Serial Dilution (SD)	5X dilution analyzed to assess matrix effect	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results within 10%	See Section 7	Flag associated positives with J.

Table 8
Summary of Method Quality Objectives – GFAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	3-stds and blank	Daily	R ≥ 0.995	Correct problem and repeat.	Apply R to all associated results when less than three stds. were used or not performed daily. When correlation is less than 0.995 -J/R.
Instrument Precision	RPD 2 injections	All standards, and ICV/CCV	RPD ≤ 10	Correct problem and repeat.	Apply J to all associated positives.
MDL Level Verification	MDL check (2XMDL no digestion)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence or shorter to bracket USACE samples	70-130%	Correct problem and repeat. Or run MDL check to verify the non-detect	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (65-70%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<65%), apply J to all positives and R to all non-detects. If MDL check was run at end and acceptable do not reject data
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	80-120%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (75-80%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<75%), apply J to all positives and R to all non-detects.
Initial Calibration Blank(ICB)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes <1/2 MRL	Correct problem and repeat	Apply U to all associated positives when less than 5X blank concentration

Table 8 (continued)
Summary of Method Quality Objectives – GFAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Continuing Calibration Blank (CCB)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes <1/2 MRL (up to 5% may exceed)	Correct problem and repeat.	Apply U to all associated positives when less than 5X blank concentration
Continuing Calibration Verification(CCV)	Mid-level verification	Every 10 samples and at end of analytical sequence	80-120%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (75-80%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<75%), apply J to all positives and R to all non-detects.
Method Blank (MB)	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL	Reprep and repeat.	Apply B to all associated positives when less than 5X blank concentration
Laboratory Control Sample (LCS)	Interference-free matrix containing all target analytes	1 per sample batch	Recovery 80-120%	Redigest and repeat.	For analyte recoveries >120%, apply J to positives. For analyte recoveries between 50-79%, apply J to positives and UJ to non-detects. For analyte recoveries <50%, apply R.
Matrix Spike (MS) (pre-digested)	Sample matrix spiked with all of target analytes prior to digestion	1 per sample batch	To consider [spike] > 4X[matrix] Recovery=75-125%	See Section 7	For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. Associated with an analyte recovery <30%, flag non-detects R and positives J.

Table 8 (continued)
Summary of Method Quality Objectives – GFAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Matrix Duplicate (MD)	Use MD or MS	1 per sample batch	RPD ≤ 20		Flag all positives for the analytes out with a J in associated samples.
Post Digestion Spike	Sample digestate spiked with all of target analytes	As needed to confirm matrix effect	Recovery=75-125%	See Section 7	For positive associated results where recovery is >125% or between 50-74%, flag with J. For non-detect associated results where recovery is between 50-74%, flag UJ. All results associated with an analyte recovery <30%, flag R.
Serial Dilution (SD)	5X dilution analyzed to assess matrix effect	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results within 10%	See Section 7	Flag associated positives with J.

Table 9
Summary of Method Quality Objectives - CVAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	5-stds and blank- CVAA	Daily	R ≥ 0.995	Correct problem and repeat.	Apply R to all associated results when less than five stds. were used or not performed daily. When correlation is less than 0.995 -J/R.
Instrument Precision	RPD 2 injections	All standards, and ICV/CCV	RPD ≤ 10	Correct problem and repeat.	Apply J/UJ to all associated results
MDL Level Verification	MDL check (2XMDL no digestion)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence or shorter to bracket USACE samples	70-130%	Correct problem and repeat. . Or run MDL check to verify the non-detect	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (65-70%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<65%), apply J to all positives and R to all non-detects. If MDL check was run at end and acceptable do not reject data
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	80-120%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (75-80%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<75%), apply J to all positives and R to all non-detects.
Initial Calibration Blank(ICB)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes <1/2 MRL	Correct problem and repeat	Apply U to all associated positives when less than 5X blank concentration
Continuing Calibration Blank (CCB)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes <1/2 MRL	Correct problem and repeat.	Apply U to all associated positives when less than 5X blank concentration

Table 9 (continued)
Summary of Method Quality Objectives - CVAA Metals

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Continuing Calibration Verification(CCV)	Mid-level verification	Every 10 samples and at end of analytical sequence	80-120%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (75-80%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<75%), apply J to all positives and R to all non-detects.
Method Blank (MB)	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL	Reprep and repeat.	Apply B to all associated positives when less than 5X blank concentration
Laboratory Control Sample (LCS)	Interference-free matrix containing all target analytes	1 per sample batch	Recovery 80-120%	Redigest and repeat.	For analyte recoveries >120%, apply J to positives. For analyte recoveries between 50-79%, apply J to positives and UJ to non-detects. For analyte recoveries <50%, apply R.
Matrix Spike (MS) (pre-digest)	Sample matrix spiked with all/subset of target analytes prior to digestion	1 per sample batch	To consider [spike] > 4X[matrix]: Recovery=75-125%	See Section 7	For positive associated results where recovery is >120% or between 30-74%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag non-detects R and positives J.
Matrix Duplicate (MD)	Use MD or MS	1 per sample batch	RPD ≤ 20%	See Section 7	Flag all positives for the analytes out with a J in associated samples.
Post Digestion Spike	Sample digestate spiked with all of target analytes	As needed to confirm matrix effect	Recovery=75-125%	See Section 7	For positive associated results where recovery is >125% or between 50-74%, flag with J. For non-detect associated results where recovery is between 50-74%, flag UJ. All results associated with an analyte recovery <30%, flag R.
Serial Dilution (SD)	5X dilution analyzed to assess matrix effect	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results within 10%	See Section 7	Flag associated positives with J.

Table 10
Summary of Method Quality Objectives - Cyanide

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	6-stds and blank- CN	As necessary when CCV is not met.	R ≥ 0.995	Correct problem and repeat.	Apply R to all associated results when less than six stds. were used or not performed daily. When correlation is less than 0.995 -J/R.
Standard Distillation	Distillation of high and low standard.	Daily	± 10% agreement between distilled and undistilled standards	Correct problem and repeat	Apply "R" to all data
Instrument Precision	RPD	All standards, and ICV/CCV	RPD ≤ 10	Correct problem and repeat.	Apply J to all associated positives.
MDL Level Verification	MDL check (2XMDL no digestion)	Once per quarter per instrument used	Analytes detected	Correct problem and repeat.	Apply "R" to all non-detects.
MRL Level Verification	QC/MRL: Low-level check standard at MRL	At the beginning and end of every daily sequence or shorter to bracket USACE samples	70-130%	Correct problem and repeat. . Or run MDL check to verify the non-detect	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (65-70%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<65%), apply J to all positives and R to all non-detects. If MDL check was run at end and acceptable do not reject data
Initial Calibration Verification (ICV)	Mid-level (2nd source) verification	After initial calibration	85-115%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (80-85%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<80%), apply J to all positives and R to all non-detects.
Initial Calibration Blank(ICB)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes <1/2 MRL	Correct problem and repeat	Apply U to all associated positives when less than 5X blank concentration

Table 10 (continued)
Summary of Method Quality Objectives - Cyanide

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Continuing Calibration Verification(CCV)	Mid-level verification	Every 10 samples and at end of analytical sequence	80-120%	Correct problem and repeat.	For results greater than the UL, apply J to all positives. For a result marginally less than the LL (75-80%), apply J to all positives and UJ to all non-detects. For recovery more than marginally out (<75%), apply J to all positives and R to all non-detects.
Method Blank (MB)	Reagent Blank to assess method contamination	1 per sample batch	Analytes <1/2 MRL	Reprep and repeat.	Apply B to all associated positives when less than 5X blank concentration
Laboratory Control Sample (LCS)	Interference-free matrix containing all target analytes	1 per sample batch	Recovery 80-120%	Redigest and repeat.	For analyte recoveries >120%, apply J to positives. For analyte recoveries between 50-79%, apply J to positives and UJ to non-detects. For analyte recoveries <50%, apply R.
Matrix Spike (MS)	Sample matrix spiked	1 per every 10 samples	To consider [spike] > 4X[matrix]: Recovery=75-125%	Perform Post-digestion Spike	For positive associated results where recovery is >120% or between 30-74%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag non-detects R and positives J.
Matrix Duplicate (MD)	Use MD or MS	1 per sample batch	RPD ≤ 20%	See Section 7	Flag all positives for the analytes out with a J in associated samples.
Post Digestion Spike	Sample digestate spiked with all of target analytes	As needed to confirm matrix effect	Recovery=75-125%	See Section 7	For positive associated results where recovery is >125% or between 50-74%, flag with J. For non-detect associated results where recovery is between 50-74%, flag UJ. All results associated with an analyte recovery <30%, flag R.
Serial Dilution (SD)	5X dilution analyzed to assess matrix effect	As needed to assess new and unusual matrices	Agreement between undiluted and diluted results within 10%	See Section 7	Flag associated positives with J.

SECTION II
DATA REPORTING

Data Reporting

Analytical data packages submitted by laboratories should contain sufficient data in order to allow the data reviewer to assess the accuracy, precision, representatives, comparability, and sensitivity. The analytical data packages must be presented in a well-organized manner, with pages sequentially numbered and include at a minimum the following:

1. Cover sheet
2. Table of contents
3. Case narrative
4. Analytical results
5. QC/QA Summary
6. Corrective Actions Documentation
7. Chain of Custody/Cooler Receipt Forms

1. **Cover Page:**
   Name of the Laboratory, and full address
   Project Name & Site location
   Date Report Issued
   Contract Number
   Signature/Date of Laboratory Director, Quality Assurance Officer, and Project Manager
   Statement indicating the authenticity of the data

2. **Table of Content:**
   An index or table of content should be included to allow easy retrieval of information for sample analyses.

3. **Case Narrative:**
   A detailed description of any problems with analysis should be discussed. Low recoveries should not automatically be considered due to matrix effect. Corrective actions and resolution should be addresses in the case narrative. Sample preparation and/or analysis out of the holding time should be noted. Discussion should include in the following if applicable: Volatile Organic, Semivolatile Organic, PCB, Pesticides, Explosive residues, Inorganic parameters, and metals. Samples that were received and were not analyzed should be indicated.

4. **Analytical Results:**
   It is preferable to report the analytical data in CLP format. The following information is needed:
   Project Name
   Field Sample ID as indicated on the Chain of Custody
   Laboratory sample ID that correspond to the field sample ID
   Method Numbers for sample preparation
   Method Number for sample analysis
   Sample date collected
   Sample date prepared

Sample date analyzed
Dilution Factor
Sample weight for soil/sediment
Sample volume for water samples
MDL levels based on dilution factor
MRL
Matrix type
Percent solid
Surrogate Recoveries
Chromatographs

All this information, with the exception of chromatographs, must be entered on the analytical data sheets.

The data packages should be organized in the following order: Volatile Organics, Semivolatile Organics, Dioxin/Furans, PCBs, Pesticides, Explosives, Poly-aromatic Hydrocarbons, Herbicides, Cyanide, metals and other Inorganic Parameter sections.

5. **QC/QA Summary:**
This summary should include results of method blanks, LCS, and recovery of the surrogate spikes for the QC/QA samples. All levels of spikes, accuracy (%) and precision (RPD) must be provided. Pre-digested and post-digested spikes for metals must be clearly identified. Also, the acceptance criteria for the recoveries and for the RPD must be documented in the data packages. A summary table for all sample numbers, and their surrogate recoveries may be provided. Furthermore, a table of QC/MRL (%D), and CCV (%D) must be provided in the QC/QA section.

6. **Corrective Actions:**
All corrective actions that were initiated during the project and their resolution must be included in the data packages.

7. **Chain-of-Custody/Cooler Receipt Forms:**
Original chain of custody and the cooler receipt forms, that are applicable to the analyzed samples, must be included in the data packages.

SECTION III
DATA ASSESSMENT

The text of data assessment was entered from US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997. Steps one through five were introduced in chapter 3 in order to link the text to the Flow Chart steps presented on page 11.

Chapter 3 DATA ASSESSMENT

3-1. Data Assessment. Any time chemical data are generated; their quality must be assessed prior to use. The type and degree of assessment required depends upon the project DQOS. Several different levels of data assessment exist, including data verification, data review, data evaluation, and data validation.

STEP-1, -2:

a. Data Verification. Data verification is the most basic assessment of data. Data verification is a process for evaluating the completeness, correctness, consistency, and compliance of a data package against a standard or contract. In this context, "completeness" means all required hard- copy and electronic deliverables are present. Data verification should be performed by the government or independent entity for QA laboratory deliverables, and by the laboratory contract holder for primary laboratory deliverables.

STEP-3:

b. Data Review. Data review is the next step in the data assessment hierarchy. Data review is the process of data assessment performed to produce the CQAR. Data review includes an assessment of summary QC data provided by the laboratory. CQAR preparation is described in detail in Chapter 4. Data review may include examination of primary and QA laboratory data and the internal QC and QA sample results to ascertain the effects on the primary laboratory's data.

STEP-4:

c. Data Evaluation. Data evaluation is the process of data assessment done by district project chemists to produce a CDQAR. Data evaluation is performed to determine whether the data meet project-specific DQOs and contract requirements. CDQAR preparation is described in Chapter 5. To prepare a CDQAR, the district project chemist relies upon the DQO summary from the SAP, the CQAR, field oversight findings, laboratory audits, PE sample results, and any other data quality indicators available.

STEP-5:

d. Data Validation. Data validation may be required for certain projects. Validation is a process of data assessment in accordance with EPA regional or national functional guidelines, or project-specific guidelines. Data validation includes assessment of the whole raw data package from the laboratory.

e. Special Requirements. Often, the requirements for data assessment will depend upon the project phase. In particular, data for use in a risk assessment will have specific quality requirements. There are several excellent references on this topic, including Chapter 3 of EM 200-1-4, ["Risk Assessment Handbook: Human Health Evaluation"]; and "Guidance for Data Usability in Risk Assessments (Parts A and B) [Office of Emergency and Remedial Response, EPA Directive 9285.7-09A, 19921.

3-2. Required Level of Data Assessment. The degree of data assessment will be different for screening level data than for definitive data. Screening level data are typically characterized by less stringent QC/QA procedures. Assessment of screening level data consists of checking whatever QC/QA indicators are available, and confirming the results with definitive analyses, usually at a 10% frequency.

3-3. Assessment of Definitive Data. Definitive data are characterized by rigorous QA/QC procedures. The following set of general procedures should be applied to the extent possible for all definitive data sets.

A. **Data Verification**. Definitive data assessment begins at the primary and QA laboratories. General processes for data quality management at the laboratory are described in EM 200- 1 -1 as well as EM 200-1-2. Once the data have met the laboratory's standards, data verification is performed to determine if the data package is correct and complete.

B. **Data Review**. See the attached Table 3-1 for more details on the specifics of data review. Data review documents possible effects on the data that result from various QC failures. It does not determine data usability, nor does it include assignment of data qualifier flags.

1. The initial inspection of the data screens for errors and inconsistencies. The chemist checks the chain of custody forms, sample-handling procedures, analyses requested, sample description and ID, and cooler receipt forms. The chemist then verifies that the data were checked by the laboratory manager or QA officer. Sample holding times and preservation are checked and noted.

2. The next phase of data quality review is an examination of the actual data. By examining data from laboratory matrix duplicates, blind duplicates, TBs, EBs, laboratory MBs, LCS, MS samples, matrix spike duplicate (MSD) samples, surrogate recoveries, and field samples, the chemist can determine whether the data are of acceptable quality.

a. Both laboratory control samples (LCS) and matrix duplicates are examined during data review. The precision of the data is quantified by the RPD between two results obtained for the same sample. The samples may be either internal laboratory QC samples (i.e., LCS) or field samples. A high RPD in an LCS is an indication of overall method failure, and may result in the rejection of an entire data set. Laboratory matrix duplicates and MSDs are also assessed by their RPD values. High RPD values for matrix duplicates indicate a lack of reproducibility, and such data may be qualified or rejected. Any such results should be noted in the assessment of data quality.

b. Data from blank samples are examined to determine if sample contamination occurred either during or after the sample collection. Equipment or rinsate blanks consist of reagent water passed through or over sampling equipment following sample collection and sample equipment decontamination. Contaminated EBs indicates inadequate decontamination between samples, and the strong likelihood of cross-contamination between samples. MBs are blank samples prepared in the laboratory and analyzed along with project samples. If analytes are detected in a MB, it is a strong indication of laboratory contamination. This would raise the possibility that project sample aliquots

were contaminated in the laboratory as well. TBs are samples of pure water that accompany the project samples from the field to the laboratory. TBs accompany each shipment of water samples to be analyzed for volatile organic compounds. Analysis of the TBs indicates whether sample contamination occurred during shipment and/or storage.

c. Surrogate recoveries are scrutinized to ensure they fall within an acceptable range. Adequate surrogate recoveries in QC samples (blanks and LCS) indicate that sample extraction procedures were effective, and that overall instrument procedures were acceptable. Surrogate recoveries in field samples are a measure of possible matrix effects and can indicate complete digestion or extraction of a sample. Surrogate recoveries outside control limits may result in qualified or rejected data.

d. A LCS is an aliquot of a clean matrix (i.e., clean water or sand) which contains a known quantity of an analyte. Good recoveries from an LCS indicate that the analytical method is in control and that the laboratory is capable of generating acceptable data. The evaluation of possible matrix effects and accuracy of the data are monitored by analysis of MS/MSD samples.

A MS sample is prepared by adding a known quantity of an analyte to a field sample. The MSD is prepared in an identical manner. MS/MSD should be analyzed at least once per every twenty samples, or once per preparation batch, whichever is greater. Recovery of the MS indicates the absence of a matrix effect and is another measure of data accuracy. Comparison of the MS/MSD results provides an indication of data precision. All MS/MSD data should be examined. Low or high spike recoveries are evidence of matrix effects and poor accuracy; a high RPD for duplicates is evidence of low precision; all such results should be reported in the data review.

e. A blind duplicate QC sample is submitted to the primary laboratory, which analyzes the majority of the samples. Analysis of the QC duplicate sample provides a measure of sample homogeneity and intra-laboratory variations. An additional replicate sample is provided to an independent QA laboratory, to provide a further test of sample homogeneity and a test of inter-laboratory accuracy. QC and QA samples effectively provide triplicate analysis of a subset of the total project samples. The three results for each set are carefully compared and tabulated. Data comparison criteria for evaluation of data comparability are described in Chapter 4. If two of three data sets agree, each laboratory's internal QC/QA data should be reassessed to determine which set of data is the most accurate. Data from related analyses may be inspected to determine which set of data is more accurate.

C. **Data Evaluation**. Data evaluation follows data review. During data evaluation, the district project chemist uses the results of the data review as summarized in the CQAR to determine the usability of the data. The CQAR documents the potential effects of QC/QA failures on the data, and the district project chemist assesses their impact on attainment of DQOs and contract compliance.

D. **Data Qualifiers**. Data assessment will result in documentation of the quality and usability of the data. Data qualifiers, called flags, will be applied as appropriate to alert the data user of deficiencies in the data. Data qualifiers are applied by the district project chemist, taking into account the project-specific DQOS. The qualifiers may be different depending on the type of data evaluation performed. Data validation by EPA functional guidelines procedures may employ different flags than project-specific validation data qualifiers. Despite the data assessment flags used, the qualifiers serve the same purpose. The flags are used to delimit the usability of the data, generally because of QC failures.

Table 3-1
Data Evaluation (Note 1)

QC Element Sample Type, Analysis Condition, or Characteristic	Type of Failure	Possible Causes (Note 2)	Major PARCCS Parameters Affected (Note 3)	Possible Effect on Data (Documented in CQAR)	Worst Case Data Evaluation (Documented in CQAR) (Note 4)
Chain of Custody	Chain broken or not kept	***Missing signatures***; missing seals; missing dates/times.	Completeness	Incomplete data	Data not legally defensible.
Sample labeling	Sample labels unreadable, missing or not attached to containers	***Failure to protect from moisture***; failure to use appropriate marker or labels; improper SOP	Representative ness Completeness	Incomplete data False positives False negatives	Invalidates all sample results
Sample Labeling	Samples mislabeled	***Sampler error***; improper SOP	Representative ness	Incomplete data False positives False negatives	Invalidates all sample results
Sample Containers	Plastic containers for organic analytes	***Samplers unaware of requirement;*** improper SOP; failure to read SAP; SAP incorrect; insufficient containers	Representative ness Accuracy Completeness	False positives False negatives High or low bias Phthalate interference	Invalidates all sample results
Sample Containers	Glass container for boron, silica, & fluoride	***Samplers unaware of requirement***; improper SOP; failure to read SAP; SAP incorrect; insufficient containers	Representative ness Accuracy Completeness	False positives High bias	Invalidates all sample results
Headspace	Bubbles in water VOC vial >6mm; visible headspace in soil VOC container	***Poor sampling technique***; caps not sealed tight; septum caps not used; dirt between cap and rim; soil not packed tight; improper SOP	Representative ness Accuracy Completeness	False negatives Low bias	Invalidates all sample results. Sample results>DL considered as minimum values only.

Table 3-1
Data Evaluation (Note 1)

QC Element Sample Type, Analysis Condition, or Characteristic	Type of Failure	Possible Causes (Note 2)	Major PARCCS Parameters Affected (Note 3)	Possible Effect on Data (Documented in CQAR)	Worst Case Data Evaluation (Documented in CQAR) (Note 4)
Preservation	No preservatives or wrong pH	***No preservatives added*** or improper amount of preservative added.	Representativeness Accuracy Completeness	False negatives Low bias	Invalidates all sample results. Affects legal defensibility of data. Sample results > DL considered as minimum values only
Preservation	Wrong preservatives	***Improper SOP;*** failure to read SAP; SAP incorrect; correct preservative unavailable	Representativeness Accuracy Completeness	Incomplete data False positives False negatives	Invalidates or qualifies some or all sample results. Affects legal defensibility of data.
Preservation	Too warm(>6 C; (Note 6))	***Insufficient ice***; shipping container inadequately insulated; samples not pre-chilled prior to shipping; transit time too long.	Representativeness Accuracy Completeness	False negatives Low bias	Invalidates all sample results. Affects legal defensibility of data. Sample results > DL considered as minimum values only.
Preservation	Too cold (<2 C; (Note 6))	***Shipping container inadequately insulated***; use of dry ice	Representativeness Accuracy Completeness	False negatives Low bias	Invalidates all sample r Affects legal defensibility of data. Sample results > DL considered as minimum values only results.
Sample filtration	Samples not filtered or preserved in field for dissolved metals	***Samplers avoided time consuming step;*** samplers unaware of requirement; improper SOP; failure to read SAP; SAP incorrect; filtration apparatus not available.	Representativeness Accuracy Completeness	False positives False negatives High bias Low bias	Invalidates all sample results for dissolved metals.
Laboratory status	Laboratory not validated by HTRW-CX	Validation request not made by A/E, PM, or TM; laboratory not validated for one or more parameters; laboratory validation lapsed.	All may be affected	Various	Invalidates all or part of data set.

Table 3-1
Data Evaluation (Note 1)

QC Element Sample Type, Analysis Condition, or Characteristic	Type of Failure	Possible Causes (Note 2)	Major PARCCS Parameters Affected (Note 3)	Possible Effect on Data (Documented in CQAR)	Worst Case Data Evaluation (Documented in CQAR) (Note 4)
Holding times	Holding times exceeded	***Excessive analysis time; tardy ship date;*** inappropriate shipping method.	Representative ness Accuracy Completeness	False negatives Low bias (Note 7)	Invalidates all sample results. Sample results > DL considered as minimum value
Analysis method	Wrong method	***Incorrect COC;*** laboratory /analyst unaware of requirement; failure to read SAP; SAP incorrect.	Representative ness Comparability Completeness Accuracy Sensitivity	False negatives Low or high bias Low or high sensitivity	Invalidates or qualifies some or all sample results
Detection limit (DL)	DL too high	***Insufficient measures to combat interferences (i.e., cleanup, background correction);*** insufficient sample; high dilution factor; wrong or inappropriate method.	Comparability Completeness Sensitivity	False negatives Low sensitivity	Invalidates sample results <DL
Method blank (MB)	Method blank absent (Note 8)	Improper SOP; lost during analysis.	Representative ness Accuracy Completeness	False positives	Invalidates all sample results >DL; sample results <DL are valid.
Method blank (MB)	Contaminatio n > DL	***Contaminated reagents, gases, glassware; ambient contamination;*** poor laboratory technique.	Representative ness Accuracy Completeness	False positives High bias	Invalidates all sample results where MB contamination is > 5% of sample concentration.
Equipment blank (EB) (rinsate blank)	Contaminatio n > DL	***Improper decontamination of field sampling equipment;*** contaminated rinsate water, containers, or preservatives.	Representative ness Accuracy Completeness	False positives High bias	Invalidates all sample results where EB contamination is > 5% of sample concentration
Trip blank (TB) (travel blank) Applies to volatile-type analyses only (VOCs, BTEX, & GRO)	Trip blank absent	***Improper SOP;*** broken during shipment; lost during analysis.	Representative ness Accuracy Completeness	False positives	Invalidates all sample results >DL; sample results <DL are valid.

Table 3-1
Data Evaluation (Note 1)

QC Element Sample Type, Analysis Condition, or Characteristic	Type of Failure	Possible Causes (Note 2)	Major PARCCS Parameters Affected (Note 3)	Possible Effect on Data (Documented in CQAR)	Worst Case Data Evaluation (Documented in CQAR) (Note 4)
Trip blank (TB) (travel blank) Applies to volatile-type analyses only (VOCs, BTEX, & GRO)	Contamination > DL	***Cross-contamination during shipment or storage;*** contaminated reagent water, glassware, or preservatives.	Representativeness Accuracy Completeness	False positives High bias	Invalidates all sample results where TB contamination is > 5% of sample concentration
LCS	LCS absent (Note 9)	***Improper SOP***	Accuracy Completeness Comparability	False positives False negatives Poor precision (high or low bias)	Invalidates all sample results
LCS and/or LCSD (also blank spike (BS) and/or blank spike duplicate (BSD))	Low recoveries	***Method failure***; improper spiking; degraded spiking solution; failed spiking device.	Accuracy Completeness Comparability	False negatives Low bias	Invalidates all sample results
LCS and/or LCSD (also BS and/or BSD)	High recoveries	***Method failure;*** improper spiking; degraded spiking solution; failed spiking device; contaminated reagents, gases, glassware, etc.	Accuracy Completeness Comparability	High bias Possible false positives	Invalidates all sample results
LCSs	High RPDs	***Method failure;*** improper spiking; failed spiking device; contaminated reagents, gases, glassware, etc.	Representativeness Precision Completeness Comparability	Poor precision (high variability)	Invalidates all sample results
Surrogates in MB, LCS, and LCSD (or BS and/or BSD)	Low recoveries	***Method failure;*** improper spiking; degraded spiking solution; failed spiking device.	Accuracy Completeness	False negatives Low bias	Invalidates all sample results
Surrogates in MB, LCS, and LCSD (or BS and BSD)	High recoveries	***Method failure;*** improper spiking; degraded spiking solution; failed spiking device; contaminated reagents, gases, glassware, etc.	Accuracy Completeness	High bias Possible false positives	Invalidates all sample results
Surrogates in samples	Low recoveries	***Matrix effects;*** inappropriate method; method failure; improper spiking; degraded spiking solution; failed spiking device.	Accuracy Completeness	False negatives Low bias	Qualifies all sample results (i.e., possible matrix effects); rejection of individual sample results.

Table 3-1
Data Evaluation (Note 1)

QC Element Sample Type, Analysis Condition, or Characteristic	Type of Failure	Possible Causes (Note 2)	Major PARCCS Parameters Affected (Note 3)	Possible Effect on Data (Documented in CQAR)	Worst Case Data Evaluation (Documented in CQAR) (Note 4)
Surrogates in samples	High recoveries	***Matrix effects;*** inappropriate method; method failure; improper spiking; degraded spiking solution; failed spiking device; contaminated reagents, gases, glassware, etc.	Accuracy Completeness	High bias False positives	Qualifies all sample results (i.e., possible matrix effects); rejection of individual sample results.
MS and/or MSD	MS and/or MSD missing	***Insufficient sample;*** improper SOP; lost during analysis.	Representative ness Accuracy Precision	False negatives Low bias High bias	Qualifies all sample results (i.e., no measure of matrix effects)
MS and/or MSD	Low recoveries (Note10)	***Matrix effects;*** inappropriate method; method failure; inadequate cleanup; inadequate background correction; failure to use method of standard additions; improper spiking; degraded spiking solution; failed spiking device.	Accuracy	False negatives Low bias	Qualifies all sample results (i.e., possible matrix effects)
MS and/or MSD	High recoveries (Note 10)	***Matrix effects;*** inappropriate method; method failure; inadequate cleanup; inadequate background correction; failure to use method of standard additions; improper spiking; degraded spiking solution; failed spiking device; contaminated reagents, gases, glassware, etc.	Accuracy	False negatives Low bias	Qualifies all sample results (i.e., possible matrix effects).
MS / MSD		***Sample inhomogeneity;*** inadequate sample mixing in laboratory; samples misidentified; method failure; improper spiking; failed spiking device; contaminated reagents, gases, glassware, etc.	Representative s Precision	High bias False positives	Qualifies all sample results > DL (i.e., possibly highly variable results).

Table 3-1
Data Evaluation (Note 1)

QC Element Sample Type, Analysis Condition, or Characteristic	Type of Failure	Possible Causes (Note 2)	Major PARCCS Parameters Affected (Note 3)	Possible Effect on Data (Documented in CQAR)	Worst Case Data Evaluation (Documented in CQAR) (Note 4)
Dilution factors	MS and/or MSD missing	***High concentration of interferences or analytes;*** inappropriate method	Accuracy Completeness Comparability	False negatives Low bias High bias	Invalidates samples with high DLs. May qualify sample results as "estimated".
Field QC sample	Low recoveries (Note10)	***Sample inhomogeneity;*** insufficient mixing in field; samples not split but collocated insufficient mixing laboratory	Representatives Precision	False negatives Low bias	Qualifies all sample results > DL (i.e., possibly highly variable results). Sample results < DL is valid.
Field QA sample (Note 12)	QA sample results do not agree with project and/or QC sample results	Improper SOP (QA and primary laboratories used different analytical methods), inadequate cleanup; inadequate background correction; laboratory contamination; preservative problem; sample misidentification; method failure; etc.; sample inhomogeneity (no agreement with both project and QC sample results).	Accuracy	False negatives Low bias	Qualifies all sample results (i.e., possible matrix effects).

Notes
(1) This table can be applied to both QA laboratory and primary laboratory sample results. Entries in the Possible causes, PARCCS Parameters Affected, Effect on Data, and Possible Data evaluation columns assume only one type of failure occurring at any one time. The cumulative or synergistic effects of more than one failure type occurring simultaneously make data evaluation more complex. Data evaluation involving multiple failure types is beyond the scope of this table.
(2) Most common cause in **bold**, *italic* and <u>underline</u> type.
(3) PARCCS parameters most affected are listed; one could almost argue that Representatives, Completeness, and Comparability are affected by all of these failures, but only the most obvious are listed. Any failure that results in invalid data affects Completeness.
(4) All data evaluations are subject to discretion of district project chemist taking into account project DQOs and other factors.
(5) Refrigeration not required for trace metals (excluding mercury), bromide, chloride, fluoride, hexavalent chromium, gross alpha, gross beta, and total radium.
(6) Applies to silica in water. Also may apply to fresh and marine water sediments.
(7) Exceeding holding times on some analyses can produce false positives (i.e., carbonates, dissolved oxygen, etc.) and high bias (i.e., pH, carbonates, dissolved oxygen, etc.). High bias and false positives can also occur when degradation products of contaminants are also themselves analytes, i.e., when 4,4'-DDT is present and holding times are exceeded, high bias and false positives for the degradation products 4,4'-DDD and 4,4'-DDD can occur.

(8) Method blanks are not appropriate for all analyses, i.e., pH, conductivity, %solids, etc.
(9) Laboratory control samples (LCSs) are not appropriate for all analyses, i.e., pH, % solids, total suspended solids (TSS), etc.
(10) Note that when native sample concentrations are significantly greater than the effective spike concentration that the conclusion of a matrix effect is only tentative. As a general rule of thumb, the native sample concentration should be no more than four times higher than the effective matrix spike concentration for the matrix effect to be considered probably present.
(11) Conventional sampling protocols for some analyte classes (i.e., VOCs, BTEX, and GRO) prohibit sample mixing and splitting because it results in the loss of major fractions of the analytes. Field and QC samples for these analytes are more appropriately collected and collocated sample pairs.
(12) Use of field QA sample data to evaluate project sample data assumes that field QA sample data is supported by a complete set of in-control laboratory quality control data.

SECTION IV
CHEMICAL QUALITY ASSURANCE REPORT

(CQAR)

The text of CQAR was entered from US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997.

DEPARTMENT OF THE ARMY
U.S. ARMY ENGINEER DISTRICT, LOUISVILLE
CORPS OF ENGINEERS
P.O. BOX 59
LOUISVILLE, KENTUCKY 40201-0059

REPLY TO
ATTENTION OF

26 April 2002

CELRL-ED-EB (200-1C)

MEMORANDUM FOR RECORD

SUBJECT: Minimum Requirements for Waiving QA-Split Sample Analysis

Reference: ER1110-1-263 dated 30 April 1998

1. This memorandum summarizes the various criteria for mandatory QA split sample analysis and the waiving of such requirements for certain analytes or projects.

2. Duplicate samples are collected at a frequency of ≥10% of the primary samples; splits off of the duplicate samples (QA-Split Sample) are collected at a frequency of 100% of the duplicate samples, and that is at levels of ≥10% of the primary samples. Also, Splits off of the primary samples (QA-Split Sample) may be collected at a frequency of ≥10% of the primary samples i.e. QA-Split samples may be off primary samples that do not have duplicate samples. QA-Split samples must be collected when the data will be used for risk assessment.

3. QA-Split samples may be collected at ≥10% of the duplicate/primary samples per sampling event, and per sample matrix (water & soil). Sediment samples are considered of soil matrix, and both surface and groundwater samples are considered of water matrix.

4. The purpose of collecting QA-split samples is different from collecting duplicate samples. Duplicate samples as well as the primary samples are analyzed in the same laboratory (Primary Laboratory) for determining the precision of the analyst and analytical methods employed in extraction, digestion, and/or instrumentation. Assurance of levels of the analytes/compounds detected in primary samples are determined via the duplicate sample analysis for risk assessment purposes, but assurance of the identification of these analytes and/or compounds may not be achieved via the duplicate analysis under the same circumstances in the same laboratory. However, analysis of QA-Split

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SUBJECT: Minimum Requirements for Waiving Split Sample Analysis

samples in a different laboratory (QA-Laboratory) may verify the identification of the detected analytes/compounds in the primary and duplicate samples. Therefore, the purpose of the QA-split sample analysis is to assure the identity of the analytes/compounds. Verification of the presence of analyte/compounds as positive, false positive, negative and/or false negative is a very important step in the defensibility and integrity of the analytical data analyzed in the primary laboratory.

5. The collection and analysis of QA split samples are mandatory in the Louisville District for HTRW projects. Associated primary sample results will be compared with those obtained from the QA split samples. The results of the comparisons will be documented in a Chemical Quality Assurance Report (CQAR). Discrepancies are not expected to be observed between the primary/duplicate and QA-split samples for water matrix. However, in soil samples, it is expected that non-homogeneity of the sample matrix would play a role in the minor and/or major discrepancies observed during the comparison of the results. Guideline of the minor and major discrepancies are defined in Table 4-1 of EM 200-1-6, 10 Oct 97. The chemist who prepares the CQAR should further investigate the major discrepancies. This may include data validation, and possibly a laboratory tape audit. Results of the investigation must be documented in the CQAR. In addition to the CQAR, data validation of the primary samples is always required.

6. Exceptions: stakeholders must be in concurrence with waiving QA-Split sample analysis to the above requirements as listed below:

   a. Petroleum Sites: BTEX, TPH, DRO, and GRO samples (see item #8).

   b. Lead base paint (LBP), and surface wipe samples.

   c. Air Monitoring Samples.

   d. BIOTA Samples.

   e. Field screening samples.

   f. Bio-Remediation: the purpose of this process is to decrease contaminants level via microbiological process; so verification of the decrease in the levels can be monitored via duplicate analysis (see item #8).

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SUBJECT: Minimum Requirements for Waiving Split Sample Analysis

7. Monitoring Wells/Long Term Monitoring Program: after establishing the quality and acceptability of the analytical data received from the primary laboratory via the QA split sample result comparisons (CQAR), QA split sample collection may be reduced to once annually for the remaining period of the Long Term Monitoring Program. Stakeholder must be in concurrence with such action of dropping the frequency of QA-Split sample collection. Furthermore, QA-Split samples may be re-instated to its original frequency whenever there is a change in the laboratory that performs the primary sample analysis (see item #8 for next phase).

8. Items #6(a), 6(f), and 7 requires QA-Split sample analysis for confirmatory samples before risk-based site closure.

9. Waiving of QA split sample requirements must be documented by the USACE project chemist and approved by the USACE Senior Chemist.

SAMIR A. MANSY, Ph.D.
Senior Chemist-Subject Matter Expert

Chapter 4 CHEMICAL QUALITY ASSURANCE REPORTS

4-1. Purpose. The purpose of the CQAR is to provide the data user with a timely review of chemical data quality. This is achieved through the inspection and analysis of QA samples, and through an examination of the corresponding project sample data. The exact format of the document is not as important as its content. The CQAR author should feel free to arrange the document in whatever format he/she is comfortable with as long as the essential information is conveyed in a succinct and timely fashion. The following format is suggested as a guide only. Whatever format is chosen should encompass at a minimum the same content that is specified below.

4-2. Cover Memorandum. The purpose of this attachment to the CQAR is to route the CQAR to its primary audience (the PM or TM). The standard memorandum format usually is adequate, which would identify the office symbol of the originating organization; the date of the transmittal; the facility name and project feature; major findings; and a point-of-contact (POC) and telephone number. The cover memorandum should be signed by the QA director whenever possible. Where local requirements for routing signed documents through the chain of command would delay delivery of the CQAR to the client, it is recommended that an unsigned advanced copy be sent to the client while the formal signed copy proceeds through channels. The cover memorandum should always refer the reader to the text for details (i.e., to find out explicitly which sample results were affected), and should always advise the reader to have the district project chemist evaluate the data usability using the project DQOS.

4-3. Cover Page. The cover page should identify the title of the document, the report status (i.e., draft, interin-4 final), its origin (i.e., the name of the firm producing it), the project facility name (i.e., Fort Green), project feature involved (i.e., Lagoon Area), the date of preparation, and the name and signature of the responsible party.

4-4. Report Contents. The CQAR should contain the items listed below, although not necessarily in the format nor in the order presented. The format should present the information in an organized fashion, which the reader can easily comprehend. The information below assumes that QA samples were collected and analyzed as part of the project QA effort.

a. Project Information. This Section should contain any pertinent reference information to aid the reader in assessing the relevance of this report. This could include such things as funding documents (i.e., MIPR Nos.), related report numbers and dates for the primary and QA laboratory data, sample receipt dates, and previous CQARS. The name, office symbol, and telephone number of a POC is also helpful.

b. Executive Summary.

(1) A summary description of the QA/QC effort expended on this data should be presented. Suggest citing the number, matrices, and types of samples tested (i.e., 10 soils, I TB, 2 EBs, I QC soil, I QA soil), as well as the tests performed. Example statements might be, "Five soil samples were collected and analyzed in triplicate (project, QC, and QA); one for lead, one for mercury, and five for PCBs. A complete assessment of the data quality could not be made because there were no QC or QA samples collected for explosives and pesticides." The identities of the laboratories performing the various project tests should be cited. Any tables and/or attachments provided in the report which are not specifically referred to in later text should be referenced here (i.e., all tables and attachments should be referenced somewhere in the text). Suggest referring to the Sample List, Analytical Methods List, and Data Comparison Tables here if this information is not provided elsewhere.

(2) The content and format of this Section is mostly left up to the author, keeping in mind that the intent is to succinctly convey the overall results of the QA effort to the reader. Any major findings should be summarized here. State the possible effects upon the project sample data based upon: 1) a review of QA sample inspection results; 2) a comparison of QA sample data with project sample data; 3) a comparison of QC sample data with project sample data; 4) a review of primary and CMQAL QC data; and 5) a review of field QC data (i.e., TB and EB results). Use the Data Evaluation Table in Chapter 3 for guidance in making this data review. State when a data review revealed no potential effects upon the project data. Also state when a complete data review could not be performed, i.e., "A complete data review could not be performed because there were no QC or QA samples collected for pesticides." Potential effects on project data which might require immediate response or corrective action by the reader (i.e., resampling) should be highlighted in some fashion (i.e., bold print or underlined). Avoid, however, the use of strong adjectives to describe data with potential effects. The determination of data quality and usefulness lies solely with the district project chemist. The district project chemist is usually a district chemist, but may also be a HTRW-CX chemist or an CMQAL chemist when a district project chemist is not available. Do not use adjectives such as "invalid", "unacceptable", "suspicious", or "unreliable". The use of these or similar terms in the CQAR may be interpreted as contradictory by a regulator in the situation where a district project chemist determines that the data may be useful for project purposes, or may meet project DQOs in spite of weaknesses in laboratory or field QC measurements. For analogous reasons, avoid applying such terms as 6 "valid", "acceptable" "reliable", etc. in describing data. The CQAR instead only should comment concerning the potential effects upon sensitivity (false negatives), precision (variability), accuracy (bias, false negatives, and false positives), representativeness, completeness (loss of data), and comparability (specified methods). Use statements such as,
"The volatiles data may have an apparent negative bias because of improper preservation." or, "The zinc values may contain false positives because of MB contamination." or, "The explosives results were not corroborated by the method-required second column confirmation and may contain false positives." or, "The low LCS recoveries for all semivolatile analytes may have caused some false negatives and probably a negative bias to detected analyte results. Any

Positive semivolatile results should be considered as minimum values only." or, "The disagreement between the field, QC, and QA sample results for metals may indicate sample inhomogeneity and a non-representative sample." or, "The PCB results may be subject to false negatives because of elevated sample detection limits." or, "The project data may not be legally

defensible since the chains of custody were never signed in the field". Some indication of what portion of the data was affected should be given, i.e., "EB contamination may indicate false positives and/or high bias in five of the nine sample results for mercury."

c. Sample List. List all QC, QA, and corresponding project samples with descriptive information including matrices, sample dates, field IDs, and laboratory IDs. A comprehensive list of all project samples is not required. Only those project samples which are part of a QC/QA/project sample set will be listed. This may not be necessary if there is only one set of QC/QA samples, or if there is relatively little data for each sample (i.e., if the only analysis performed was for lead). However, where there is a large amount of data on multiple samples, a sample list is highly recommended to aid the reader in grasping what data is available to examine.

d. Analytical Methods List. This information can be presented in tabular form and at a minimum should specify the analytical method numbers and preferably (if known) the preparation method numbers as well. Note that this information may alternatively be provided in the data comparison tables.

e. Review of QA Sample Data. One of the purposes of this Section is to assure the reader of the quality of the QA sample results, since the QA sample results will be the benchmark against which the project sample results will be judged. A second purpose is to evaluate the sample handling of the QA samples, since that has implications on how the project samples may have been handled.

(1) Review of QA Laboratory Quality Control Data. At a minimum, the following laboratory QC data should be reviewed: holding times, methods utilized, the results for MBs, LCS, MS/MSDs, matrix duplicates, and surrogates (see also Paragraph 4-4.g(l) below). This may be accomplished through tables summarizing laboratory QC data, or through descriptive statements such as, "The data package from XYD laboratory was complete with all required QC information. All MBs were free from contamination. All analyses were performed using specified methods within proper holding times. The majority of the duplicates, RPDs, laboratory control, surrogate, and MS recoveries were within laboratory control limits with the following exceptions..." Any excursions beyond laboratory control limits could then be listed. Since the QA data should be of high quality to begin with (implying that excursions should be few), it may be more efficient to just list the deviations from acceptable limits, rather than to tabulate all of the QC data in some kind of statistical format. The actual evaluation criteria could be the laboratories own control limits, or could be set by the project DQOs. Project DQO evaluation criteria sometimes may include USACE validation guidelines, or EPA national or regional functional guidelines, depending upon regulator requirements. See the Data Evaluation Table in Chapter 3 for general guidelines on evaluating data.

(2) Review of QA Sample Handling. Review of sample handling is performed at sample log-in and includes looking for correct sample containers, sampling procedures, sample preservation (i.e.,temperature, pH, etc.), packaging, labeling, and chain of custody procedures. Deficiencies noticed on QA samples at the QA laboratory imply that the project samples possessed similar deficiencies upon arrival at the primary laboratory. The QA laboratory should notify the district project chemist or TM of any serious deficiencies upon arrival. The project POC should be

apprised of the implications of the deficiencies when notified, and asked for a decision on whether to proceed with the analyses. If the samples are analyzed in spite of the deficiencies, then the possible effects upon the QA and project sample data should be discussed in this Section, highlighting any potential negative effects upon the data.

f. Data Comparison Tables. These tables compare the project, QC, and QA sample results in a matrix-type presentation. The header information should include project, sample, and analysis information, including facility name, project feature, sample date, field and laboratory ID numbers, sample description, method numbers, dates analyzed, dilution factors percent moisture, and concentration units. The primary and QA laboratories should be identified here as weld. The body of the table should list any detected analytes, estimated detection limits (DLs) and quantitation limits (QLs) for detected analytes and a range of DLs and QLs for non-detected analytes from both the primary and QA laboratories; results from the project, QC, and QA samples, including the number of tentatively identified compounds (TICS) and the sum of the TIC concentrations; and an indication of agreement or disagreement in the data. A separate page detailing the agreement criteria and explaining any qualifiers used in the tables (i.e., <, J, U, B, etc.) should be attached. Sensitivity (i.e., DLs and RLs) should be evaluated only to verify that project-specific DQOs were satisfied. The agreement criteria shall be as shown in Table 4-1.

Table 4-1
Criteria for Comparing Field
QC and QA Sample Data
(See Text)

Matrix	Parameter	Disagreement	Major Disagreement
All	All	>5x difference when one result is < DL	>10x difference when one result is < DL
All	All	>3x difference when one result is < RL	>5x difference when one result is < RL
Water	All except TPH	>2x difference	> 3x difference
Soil	All except metals, VOCS, BTEX, and TPH	>4x difference	>5x difference
Soil	Metals	>2x difference	>3x difference
Water and Soil	TPH	Arbitrary (suggest >3x difference)	Arbitrary (suggest >5x Soil difference)
Soil	VOCs and BTEX	Arbitrary (suggest >5x difference)	Arbitrary (suggest >10x difference)

Reference: CRREL Special Report No. 96-9, "Comparison Criteria for Environmental Chemical Analyses of Split Samples Sent to Different Laboratories - Corps of Engineers Archived Data", Grant, C.G., Jenkins, T.F., and Mudambi, A.R., USACE Cold Regions & Environmental Research Laboratory, Hanover NH, May 1996.

The above criteria shall be applied when comparing field and QC sample pair data, as well as when comparing project and QA sample pair data. With the exceptions of volatile organic compounds (VOCS) in soil; and benzene, toluene, ethyl benzene, and xylenes (BTEX) in soil; and of total petroleum hydrocarbons (TPH) in either water or soil, the above criteria shall be used for all CQAR data comparisons. There is no definitive data for establishing comparison criteria for TPH (in water or soils) because of the wide variety of method modifications used by laboratories in the SW-846 8015M method ("M" is for "Modified"). The same is true for VOC and BTEX in soils because of the large error introduced during the conventional sample handling process. Result pairs are considered to disagree whether they are in the "Disagreement" or "Major Disagreement" category.

g. Review of Project Sample Data. This is the Section the reader will refer to when seeking more details after reading the cover memorandum or the Executive Summary.

(1) Review of Primary Laboratory Quality Control Data. At a minimum, the laboratory QC data for the project and QC samples which correspond to the QA samples shall be reviewed. Some districts may arrange with the CMQAL to review the QC data for all of the project samples, although that is not required content for a CQAR. The laboratory QC data for project sample results should be examined in a manner similar to that used for the QA sample data (paragraph 4- 4.e(l), above, and Table 3-1 in Chapter 3. Observed weaknesses in laboratory QC data may undermine the credibility of project sample data, even before comparison with the QA sample results. Missing QC data is always a deficiency, and will automatically injure data credibility by presenting the data in an unsupported manner. Samples prepared or analyzed outside of holding time may promote false negatives and give a negative bias to the associated data. Data sets without the required frequency of laboratory QC samples may have undefined data quality, although some explanation may be required. For example, sample results from a data set without a MB may be subject to false positives, but any samples in that same data set with undetectable levels of analyte would be unaffected (assuming LCSILCSD recoveries were acceptable). Serious matrix effects may cause the data to fail project DQOS, making it unusable for project purposes. High RPDs in the project sample/matrix duplicate and MSIMSD pairs indicate inhomogeneity in the sample matrix, which would imply high variability (i.e., low precision) in the project sample results. Some samples defy homogenization attempts; i.e., sludges, clayey sods or sediments, multiphasic samples, and samples with macroscopic particles of analytes such as explosives and metals. High sample inhomogeneity can result in a determination that the samples were non-representative, making the associated analytical data unusable for project purposes. Determine if the primary laboratory possessed a current HTRW-CX validation when the analyses occurred, and if the project DQOs required that the project laboratories be validated. Data generated by an invalidated laboratory can adversely affect sensitivity, as well as all of the PARCCS parameters (precision, accuracy, representativeness, comparability, completeness, and sensitivity), making evaluation of its quality difficult. The above techniques also may be applied to QA sample data. Provide as much discussion as necessary to fully explain the implications of out-of-control laboratory QC data upon the project sample results.

(2) Review of Field Quality Control Data. Any detectable analyte concentrations in the EB and/or TB should be commented on and their implications explained. There may be field notes provided separately or as part of the chains of custody that may yield clues concerning out-of-

control QC data. Provide as much discussion as necessary to fully explain the implications of out- of-control field QC data upon the project sample results.

(3) Comparison with QA Sample Data. The availability of QA sample data provides more information for the data evaluator to further qualify the project sample data. QA sample data can reveal defective project sample data even when laboratory QC data are all in control. On the Other hand, the confirming analysis of QA samples by an independent QA laboratory can provide evidence supporting the usability of project data that may otherwise have been questioned because of out-of-control laboratory QC data. QA sample data that does not agree with either the project sample or QC sample data should be discussed in detail in this Section. When a data disagreement is observed, every attempt should be made to explain or to reconcile the disagreement. Verify at the outset that the data being compared all originated from splits (or co- located replicates in the case of volatiles) of the same sample. Do this by comparing sample descriptions, laboratory and field ID numbers, and the results from other analytes. Where feasible, both laboratories should be asked to check their results. Although there is the presumption that QA sample data in general is of higher quality, that may not always be the case. Where there is a disagreement involving the QA sample data, both data sets should be evaluated to ascertain if either has any weaknesses in its supporting laboratory QC data (i.e., missing or out- of-control data). If the QA laboratory QC data is an present and in control, then the QA sample data is to be considered the "more correct", regardless of the status of the primary laboratory QC data. If the primary laboratory QC data is deficient, but the QA data agrees with the project sample results, then the QA data can be used to confirm the project data. These discussions all assume a single analyte perspective, i.e., an out-of-control analyte will not affect the evaluation of another analyte that is in control, even if analyzed by the same laboratory. There is always the possibility that differences between the QA and project data could be due to sample inhomogeneity, and the temptation might exist to assign data discrepancies to this effect. The data evaluator is cautioned to use this explanation only as a last resort, or when supporting information is available, i.e., when all three sample results (i.e., project, QC, and QA) disagree, or when data from other parameters is also highly variable.

h. Sample Handling Documentation. This Section should contain copies of all documentation related to sample handling and the QA inspection process, i.e., chains of custody, cooler receipt forms, notices of deficiency, and documentation of any other communications (written or oral) with USACE or contractor POCs on correction of sample handling deficiencies.

i. Project Specific Concern's. This Section should address all communications between the district, field offices, prime contractor, and the CMQAL. Examples of this may be a request from the district for lower detection Emits, quick turnaround analysis, or other requests or comments of an unusual nature (i.e., outside the boundaries of the pre-established project DQOS). This Section should also address anything that may have improved the chemistry aspects of the project (i.e., use of a USACE-validated laboratory, more appropriate methods, more QC and QA samples, faster turnaround of QA sample results, more field oversight, etc.).

SECTION V
DATA VALIDATION REPORT

Standard Operating Procedure Data Validation

This report serves as the Standard Operating Procedure (SOP) for executing data validation. It is divided into two attachments; Attachment A contains the technical methodologies to be used for data validation, and Attachment B contains the data validation checklists.

Attachment A:
Concepts provided in this document are based on Method Quality Objectives-SW846. The criteria for data validation are based on SW-846 methodology and the USACE Shell. In more subjective areas, only general guidance is offered due to the complexities and uniqueness of the issue related to specific samples and methods used for analysis.

Attachment B:
The checklists for each method are provided in Attachment B as follows:

Volatile Organic Data Validation Checklist
Semivolatile Organic, Base/Neutral/Acid (BNA Data Validation Checklist
PCB Data Validation Checklist
Pesticides Data Validation Checklist
Nitroaromatics & Nitroamines Data Validation Checklist
Polynuclear Aromatic Hydrocarbon Data Validation Checklist
Metal Data Validation Checklist
Special Parameters

The reviewer/validator will submit a Validation Report that summarizes the findings to inform the data user on the limitation of the data, discussing areas of concerns, and issues that might affect the quality of the data. The validator will review raw data submitted by the laboratory, QC results, Chain of Custody forms, Cooler Receipt forms, and laboratory narratives. The validator may use the attached Checklists, or modification of them in documenting the findings, however, the validator must use the chemistry guideline presented in the attached checklists, and the MQO tables included in this document under Chemical Analysis Criteria, (Section I). Laboratories might have QC limits that are more stringent than the criteria included in the MQO tables, however, the validator must validate the data based on the MQO tables, and not on the laboratory QC limits. So, data should not be considered estimated "J" if they are out of the laboratory but within the established QC limits that are presented in the MQO tables, and the validator would override the laboratory's qualifier "J" and consider the data unqualified. Also, the validator will use the **10X, or 5X rule** for qualifying positive hits in the samples that appear in the method blank (see qualification process in Section 1). The **10X rule** is employed when the contaminants are the common laboratory contaminants such as methylene chloride and acetone, for volatile organic compounds and phthalate compounds for semivolatile organic compounds. If the levels in the samples were within 10 times the levels in the method blanks, then the positive hits in the sample must be qualified with "B", otherwise the positive hits should be considered analytes

detected in the sample. The **5X rule** is for non-common laboratory contaminants, if analytes detected in the samples are within 5 times the levels detected in the method blanks, then the analytes in the samples must be qualified with "B", otherwise, the analytes in the samples should not be qualified. All samples affected will be qualified as necessary, and all filled checklists forms must be included in the report. When a deviation from a procedure is observed, the Validator must contact the laboratory to obtain additional information in order to reach a decision. The Validator might prepare the report in the format outlined on the next page. The report will contain an overall assessment, and specifics of any relevant issues.

Proposed Contents of Data Validation Reports

Cover
Title Page: With signature blocks for US Army Corps principal member and senior reviewers
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Glossary
Acronyms

Background
- Description of Prior Activities
- Description of Prior Data

Description of Work Performed
- Approach/Methodology
  - Data validation
  - Data Completeness
- Summary of Qualifications
- Impact on data Quality

Data Review
- Description of Data Completeness Review
- Data Summary Tables
  - Summary of Laboratory Completeness
- Summary of Field Quality Control Sample Completeness
- Deficiencies in Data and any Flagging Codes
- Source of Deficiencies
- Impact on Quality of Data
- Data Review Comments
- Analysis Specific Comments

Control Charts
- Control Charts for groups of 20 or more samples
  - LCS, surrogates (as applicable), MRL

Finding on Overall Quality of Data (%Usability)

Attachments:
1. Environmental data Assurance Guideline (MQO-Tables)
2. Individual data lot checklists/Filled out and signed by the reviewer
3. Data Completeness Excel Spreadsheet
4. Control Charts for surrogate recoveries, MS/MSD, and RPDs
5. Chain of Custody and Cooler Receipt Forms
6. Analytical Results with qualifiers for compounds/analyte based on reviewer's findings

The following is an example of a data validation report. All QC criteria listed in MQO tables must be addressed in the DVR in Sec 2.0 - Quality Control Results.

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2.1.2 Initial Calibration - GC/MS .....
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2.5 TARGET ANALYTE LIST (TAL) METALS DATA VALIDATION.....
2.5.1 Initial Calibration - ICP and CVAA .....
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2.5.3 Laboratory Control Sample Analyses.....

2.5.4 Duplicate Sample and Matrix Spike Analyses.....
2.5.5 Contract Required Detection Limit (CRDL) Standard Analyses - Metals .....
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ATTACHMENT C ANALYTICAL RESULTS AND QUALITY CONTROL SUMMARY TABLES

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TABLE 6 QUALIFIED SAMPLE RESULTS DUE TO OUTLYING SURROGATE RECOVERIES
TABLE 7 QUALIFIED SAMPLE RESULTS DUE TO OUTLYING LABORATORY CONTROL SAMPLE (LCS) AND LCS DUPLICATE (LCSD) RECOVERIES
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ACRONYMS

1.0 INTRODUCTION

The introduction should contain the following information or similar information at a minimum:

This Data Validation Report (DVR) details the assessment and validation for analytical data collected and generated during field activities at *insert site and location.* The laboratories subcontracted for the chemical analysis of the soil and water samples were *insert name of laboratory and location of laboratory.* All of the laboratories are United States Army Corps of Engineers (USACE) validated to perform hazardous waste analysis.

Between *insert date of sampling and description of sampling event.* A summary table, in Attachment A, provides the laboratory performing the analyses, field sample identification, laboratory sample identification, sample date, sample time, and the analyses requested for each sample discussed in this DVR. Copies of sample chain-of-custody (COC) documents and cooler receipt forms for samples discussed in this DVR are included in Attachment B. Analytical results of the samples are provided in tabular format in Attachment C, Table 1a for soil results and Table 1b for water results. The analyses performed included the following:

- *STATE THE ANALYSIS AND METHOD*

- *Describe to what extent the data validation was conducted and who conducted it.*

Data validation of all sample results was performed by *insert company that did data validation*. A review of 100% of the data, which allows for complete independent data review without reconstruction of analytical data was conducted. Approximately 10% of the data underwent a comprehensive or extensive review which allowed for complete reconstruction of the chemical analyses. The comprehensive review included the recalculation of calibration curves and sample results. The data were validated in accordance with the analytical methods and the documents entitled:

LIST DOCUMENTS PERTAINING TO DATA VALIDATION THAT WERE USED

The results of the data validation are presented in the following subsections.

An overview of the validation findings are presented in table format in Attachment C. Several data validation flags were utilized in the validation process. The definitions of these qualifier flags are as follows:

R Quality control (QC) indicates the data is not usable

J Indicates an estimated value.

I A matrix effect was present.

T The analyte has been "tentatively identified" and the associated numerical value represents its approximate concentration (using GC/MS).

U Indicates the compound or analyte was analyzed for, but not detected at or above the stated limit.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

B The compound was also detected in the method blank

All flags have been incorporated into the data tables presented in this report (Attachment C).

2.0 QUALITY CONTROL RESULTS

This section provides a summary of the field and laboratory QC sample results, which were used to meet the project data quality objectives (DQOs) for the investigation. The following subsections summarize the validation findings in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC) criteria.

In each of the following sections discuss the quality control criteria and how it was applied to the data. Discuss any non-conformances and refer to a summary table of the non-conformances.

2.1 VOLATILE ORGANIC COMPOUNDS DATA VALIDATION

2.1.1 Gas Chromatograph/Mass Spectrometer (GC/MS) Tuning and Mass Calibration

2.1.2 Initial Calibration –GC/MS

2.1.3 Continuing Calibration

2.1.4 Internal Standard (IS) Recoveries

2.1.5 Surrogate Spike Recoveries

2.1.6 Laboratory Control Sample Analyses

2.1.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

2.2 SEMIVOLATILE ORGANIC COMPOUNDS DATA VALIDATION

2.2.1 Gas Chromatograph/Mass Spectrometer Tuning and Mass Calibration

2.2.2 Initial Calibration – GC/MS

2.2.3 Continuing Calibration

2.2.4 Internal Standard (IS) Recoveries

2.2.5 Surrogate Spike Recoveries

2.2.6 Laboratory Control Sample Analyses

2.2.7 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

2.3 POLYCHLORINATED BIPHENYLS (PCBS) DATA VALIDATION

2.3.1 Initial Calibration - Gas Chromatograph/Electron Capture Detector (GC/ECD) Calibration

2.3.2 Continuing Calibration

2.3.3 Surrogate Spike Recoveries

2.3.4 Laboratory Control Sample Analyses

2.3.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

2.4 EXPLOSIVES DATA VALIDATION

2.4.1 Initial Calibration

2.4.2 Continuing Calibration

2.4.3 Surrogate Spike Recoveries

2.4.4 Laboratory Control Sample Analyses

2.4.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

2.4.6 Field Duplicates

2.4.7 Method Blank Analyses

2.5 TARGET ANALYTE LIST (TAL) METALS DATA VALIDATION

2.5.1 Initial Calibration - Inductively Coupled Plasma (ICP) Calibration - Metals And Cold Vapor Atomic Absorption (CVAA) - Mercury

2.5.2 Continuing Calibration

2.5.3 Laboratory Control Sample Analyses

2.5.4 Duplicate Sample and Matrix Spike Analyses

2.5.5 Contract Required Detection Limit (CRDL) Standard Analyses

2.5.6 ICP Serial Dilution

2.6 SULFATE DATA VALIDATION

2.6.1 Initial Calibration

2.6.2 Laboratory Control Sample Analyses

2.6.3 Field Duplicates

2.6.4 Method Blank Analyses

2.7 FIELD DUPLICATES

To assess the analytical and sampling protocol precision, field duplicates were collected and submitted to the laboratory. The following field duplicates were submitted to the laboratory for analyses:

Sample Type	
Sample Duplicate	**Primary Sample**

2.8 REPRESENTATIVENESS EVALUATION

Representativeness is a qualitative evaluation of whether the data represent actual environmental conditions. Representativeness was evaluated using holding time criteria, which reflect the length of time after sample collection that a sample or extract remains representative of environmental conditions. Depending on the analysis, either one or two holding times were evaluated. For those analyses that do not include a sample extraction, only one holding time was evaluated: the length of time between sample collection and analysis. For analyses that require sample extraction prior to analysis, two holding times were evaluated: the length of time from sampling until extraction and the length of time from extraction to analysis. Holding times were compared to standard method-specific holding times accepted by the EPA. All holding times that are within acceptance criteria are considered representative. Those holding times outside of EPA acceptance criteria are qualitatively evaluated to determine their effect on sample representativeness.

Representativeness was also evaluated by analysis of laboratory method blanks, trip blanks, and equipment blanks which were used to identify sources of contamination not associated with environmental conditions.

2.8.1 Sample Holding Times

2.8.2 Method Blank Analyses

2.8.3 Trip Blanks – VOCs

2.9 USABILITY AND COMPARABILITY

Usability of data was evaluated by assuring that all the analytical requests were met, samples were received in the proper condition, and all analyses were performed within the appropriate holding times.

3.0 QC SUMMARY

Summarize the results of the data validation and determine the percent completeness for the project.

4.0 REFERENCES

LIST ANY REFERENCES USED DURING THE DATA VALIDATION OR THE PREPARATION OF THIS DOCUMENTS.

ATTACHMENT A
CHAIN OF CUSTODY

EXAMPLE CHAIN OF CUSTODY TRACKER TABLE

Sample Identification	Depth (Ft bgs)	Laboratory	Laboratory ID	Laboratory SDG	Date Sampled	Time Sampled	Matrix	Sample Type	VOCs/8260B	SVOCs/SW 8270C	Comment

ATTACHMENT B

CHAIN OF CUSTODIES AND COOLER RECEIPT FORMS

INSERT COPIES OF CHAIN OF CUSTODIES AND COOLER RECEIPT FORMS IN THIS SECTION

ATTACHMENT C

ANALYTICAL RESULTS AND QUALITY CONTROL SUMMARY TABLES

Table 1
ANALYTICAL RESULTS SUMMARY
ANALYSIS
SITE NAME AND LOCATION
(UNITS)

TABLE 2
QUALIFIED SAMPLE RESULTS FOR SAMPLES ANALYZED OUTSIDE OF GC/MS TUNE WINDOW
SITE NAME
SITE LOCATION

Laboratory SDG	Parameter	Sample ID	Tune Window Exceedance (Minutes)	Sample Result	Qualifier	Units

A TABLE FOR EACH QUALITY CONTROL CRITERIA THAT HAS NOT MEET THE STATED CRITERION SHOULD BE INCLUDED IN ATTACHEMENT C

TABLE 10
PRECISION RESULTS FOR SAMPLE DUPLICATE ANALYSIS
ANALYSIS
SITE
LOCATION
(UNITS)

	Date Sampled	**SDG**	**Analyte or compound**	**Flags**	**Analyte or compound**	**Flags**	**Analyte or compound**	**Flags**	**Analyte or compound**	**Flags**
Sample ID										
Duplicate Sample ID										
RPD										

ATTACHMENT A
DATA VALIDATION GUIDELINES

DATA VALIDATION GUIDELINES

INTRODUCTION

This Section presents guidance in laboratory data evaluation and validation to be used by Louisville District Chemists and/or contractors. This review process will be conducted using initial calibration, initial calibration verification (ICV), continuing calibration verification (CCV), QC results, chain-of-custody forms, and laboratory narratives. When a deviation from the procedure is observed, the laboratory will be requested to provide additional information to enable the reviewer to reach a decision. The data validation report will address each of the following sections as applicable. The details of how data validation will be conducted is presented in the following manner of this document:

1.0 Volatile Organic Compounds, GC/MS
2.0 Semivolatile Organic Compounds, Base-Neutral-Acid (GC/MS)
3.0 PCB Validation Procedure (GC/ECD)
4.0 Pesticide Validation Procedure (GC/ECD)
5.0 Explosive Residues Validation Procedure (HPLC)
6.0 Polynuclear Aromatic Hydrocarbons Procedure(HPLC)
7.0 ICP Metals Procedure
8.0 GFAA Metals Procedure
9.0 CVAA Metals Procedure
10.0 Cyanide Validation Procedure
11.0 Special Parameters Procedure

VALIDATION PROCEDURE

The Validator will review the following requirements as applicable per method:

Holding times
GC/MS Tuning (as applicable)
Surrogate recovery (as applicable)
Matrix spike/Matrix Spike Duplicate (as applicable)
QCMDL
Calibration
- Breakdown Standards (if applicable)
- Initial calibration
- Initial calibration verification (ICV)
- QC/MRL
- Continuing calibration verification

Internal Standards (as applicable)
Blanks
- Method blank
- Trip blank for Volatile Organic Analysis
- Field blank

Laboratory Control Sample

Duplicate sample results
- Laboratory duplicate
- Field duplicate

Sample Results
- Confirmation RPD

Laboratory data assessment (Narrative report)

1.0 VOLATILE ORGANIC ANALYSES

1.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with “J”, meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and non-detect with "R".

1.2 GC/MS TUNING

Criteria: The instrument performance check BFB for volatiles must be performed at the beginning of every 12 hour analytical period during which samples and standards are analyzed. These performance checks must meet the ion abundance requirement criteria recommended in the method.

Action: No flagging criteria – MUST RETUNE before analysis. If lab did not correct the problem and re-tune prior to processing the samples, reject all data.

1.3 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at prior to the sample analysis i.e. producing a linear calibration curve.

Action: The reviewer must verify that the initial calibration standards contain both volatile target compounds and surrogate compounds (system monitoring compounds) at concentration of <MRL, 20, 50, 100, 200 µg/L, although these levels are not mandatory. The relative standard deviation (RSD) for each target analyte must be ≤ 15% with a maximum RSD ≤20%.

The Validator must check the response factor (RF) for the five system performance check compounds (SPCC) The RF must meet the minimum requirements. The mean Response Factor must meet the following: Chloromethane = 0.10, 1,1-Dichloroethane = 0.10, Bromoform = 0.10, Chlorobenzene = 0.3, and 1,1,2,2-Tetrachloroethane = 0.30. When the minimum mean Response Factor is less than that specified for the volatile SPCCs**,** all results will be qualified “R”, meaning rejected values.

The validator must check the system for leaks and/or reactive sites on the column, by evaluating the calibration check compounds (CCC). The RSD must be ≤ 30% for each of the CCC compounds, 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. If RSD > 30%, then qualify data with "R" for reject.

The calibration should be re-run prior to sample analysis if the calibration for compounds is not linear. If any volatile organic compounds have an RSD greater than 15%, apply J to positives and R to non-detects. When $r<0.99$ ($r^2<0.99$) and/or D>15%, apply J to positives and R to non-detects.

1.4 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 80-120%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure. Sporadic marginal failure allows for up to 5% of the target list for non-contaminant of concern to meet 60-140% before corrective action.

Action: For results greater than 120%, apply J to all positives. For a results < 80% and > 60%, apply J to all positives and UJ to all non-detects. For recovery < 60%, apply J to all positives and R to all non-detects.

Criteria: The Validator must check the %D for the QC/MRL (low ICV) and that the recovery is within 70-130%. Sporadic marginal failure allows for up to 5% of the target list for non-contaminants of concern to meet 60-140% before corrective action.

Action: For results greater than 130%, apply J to all positives. For a results < 70% and > 60%, apply J to all positives and UJ to all non-detects. For recovery < 60%, apply J to all positives and R to all non-detects.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed, then a deficiency must be entered in the report and qualify all non-detect with “R”..

1.5 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every twelve hours. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. If the CCV did not meet the RF criteria for the SPCC, and/or the %D for the CCC, then results of all analyzed samples become suspect during that 12 hours period. In addition all of the following must comply: SPCC: minimum RF values per method requirements, CCC: verify D ≤ 20%, and D or mean D ≤ 20% with a max D for each target analyte ≤ 30%.

Action: For D>20% with a negative bias apply J to positives and R to non-detects; and with a positive bias apply J to positives only.

1.6 INTERNAL STANDARDS

Criteria: For continuing calibration, the retention time must be within ± 30 seconds from the retention time of the mid-point standard in the ICAL. The internal standards area must be within –50% to + 100% of the ICAL mid-point standard.

For samples, the retention time must be within ± 30 seconds from the retention time of the mid-point continuing calibration of the sequence. The internal standards area must be within –50% to + 100% of the CCV mid-point standard.

Action: If the continuing calibration internal standard retention times or the internal standards area do not meet the criteria, then qualify data with "R". If the internal standards in the samples fall between 10% and 50% or >150%, qualify all associated positive target compound J and non-detects with "UJ". For results associated with an internal standard <10% of the cooresponding CCV internal standard area count, apply R. Professional judgement should be used to qualify data with shifts in internal standard retention times.

1.7 METHOD BLANK

Criteria: Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X and 10X rule should apply. Apply the 10X rule for common lab contaminants such as acetone and methylene chloride.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 10X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 10 times the amount in any blank for common contaminants, or 5 times the amount for other compounds will be qualified "B", meaning that compound was found in the blank and may be present due to laboratory contamination.

The Validator will evaluate the TIC using similar consideration. The 5 times criteria will apply for sample and associated blank results.

1.8 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 30% and the lower limit (LL), flag J/UJ. For a recovery < 30%, flag J/R.

1.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: The MS/MSD results should not be used alone to qualify data. The reviewer should consider other quality control results in conjunction with professional judgment. Some sample results may be qualified "J", meaning estimated values due-to matrix interference. However, if all parameters were met for the sample analysis such as ICV, CCV, MDL Check, LCS, and surrogate recoveries, but MS/MSD failed the QC limits, then the data must not be qualified. The only data that could be qualified are those of the sample that was utilized for the MS/MSD. The reviewer must be careful in qualifying data based on the MS/MSD results. The reviewer must establish a link between the MS/MSD failure and the sample data for justifying data qualification. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

1.10 SURROGATE RECOVERY

Criteria: Samples are spiked with surrogate compounds prior to preparation. The surrogate recoveries for volatile organic compounds must be within acceptable limits of 50-150%.

Action: When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R.

1.11 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

1.12 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\text{\# usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\text{\# total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

2.0 SEMIVOLATILE ORGANIC ANALYSIS (EPA8270)

2.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with "J", meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and non-detect with "R".

2.2 GC/MS TUNING

Criteria: The instrument performance DFTPP for base-neutral-acid fractions, must be performed at the beginning of every 12 hours analytical period during which samples and standards are analyzed. These performance checks must meet the ion abundance requirement criteria recommended in the method.

Action: No flagging criteria – MUST RETUNE before analysis. All data must be rejected.

2.3 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed prior to sample analyses i.e. producing a linear calibration curve.

Action: The reviewer must verify that the initial calibration standards contain both semi-volatile target compounds and surrogate compounds (system monitoring compounds) at concentration of <MRL, 20, 50, 100, 200 µg/L, although these levels are not mandatory. The relative standard deviation (RSD) for each target analyte must be ≤ 15% with a maximum RSD ≤20%.

The validator must check the response factor (RF) for the five compounds, system performance check compounds (SPCC), the RF must meet the minimum requirement of 0.05 for n-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol.. When the minimum mean Response Factor is less than that specified for the semivolatile SPCCs**,** all results will be qualified "R", meaning rejected values.

The validator must check the system for leaks and/or reactive sites on the column, by evaluating the calibration check compounds (CCC). The RSD must be ≤ 30% for each of the CCC compounds. If RSD > 30%, then qualify data with "R" for reject.

If any semivolatile organic compounds have an RSD greater than 15% and less than 30%, then r must be greater than or equal to 0.99. The calibration should be re-run prior to sample analysis if the calibration for a compound is not linear. If any semi-volatile organic compounds have an RSD greater than 15% apply J to positives and R to non-detects. When r<0.99 (r^2<0.99) and/or D>15%, apply J to positives and R to non-detects.

2.4 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 70-130%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure. Sporadic marginal failure allows for up to 5% of the target list of non-contaminants of concern to meet 50-150% before corrective action is required.

Action: For results greater than 130%, apply J to all positives. For a results < 70% and > 50%, apply J to all positives and UJ to all non-detects. For recovery < 50%, apply J to all positives and R to all non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%. Sporadic marginal failure allows for up to 5% of the target list for non-contaminants of concern to meet 50-150% before corrective action.

Action: For results greater than 130%, apply J to all positives. For a results < 70% and > 50%, apply J to all positives and UJ to all non-detects. For recovery < 50%, apply J to all positives and R to all non-detects.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or analytes were not detected, then a deficiency must be entered in the report, and qualify associated sample data with R..

2.5 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every twelve hours. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. If the CCV did not meet the RF criteria for the SPCC, and/or the "D" for the CCC, then results of all analyzed samples become suspect during that 12 hours period. In addition all of the following must comply: <u>SPCC</u>: minimum RF values per method requirements, <u>CCC</u>: verify D ≤ 20%, and D or mean D ≤ 20% with a max D for each target analyte ≤ 30%.

Action: For D>20% with a negative bias apply J to positives and R to non-detects; and with a positive bias apply J to positives only.

2.6 INTERNAL STANDARDS

Criteria: For continuing calibration, the retention time must be within ± 30 seconds from the retention time of the mid-point standard in the ICAL. The internal standards area must be within –50% to + 100% of the ICAL mid-point standard.

For samples, the retention time must be within ± 30 seconds from the retention time of the mid-point continuing calibration of the sequence. The internal standards area must be within –50% to + 100% of the CCV mid-point standard.

Action: If the continuing calibration internal standard retention times or the internal standards area do not meet the criteria, then qualify data with "R". If the internal standards in the samples fall between 10% and 50% or >150%, qualify all associated positive target compound J and non-detects with "UJ". For results associated with an internal standard <10% of the cooresponding CCV internal standard area count, apply R. Professional judgement should be used to qualify data with shifts in internal standard retention times.

2.7 METHOD BLANK

Criteria: Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X and 10X rule should apply. Apply the 10X rule for common lab contaminants such as phthalates.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 10X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 10 times the amount in any blank for common contaminants, or 5 times the amount for other compounds will be qualified "B", meaning that compound was found in the blank and may be present due to laboratory contamination.

The Validator will evaluate the TIC using similar consideration. The 5 times criteria will apply for sample and associated blank results.

2.8 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 30% and the lower limit (LL), flag J/UJ. For a recovery 30%, flag J/R.

2.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: The MS/MSD results should not be used alone to qualify data. The reviewer should consider other quality control results in conjunction with professional judgment. Some sample results may be qualified "J", meaning estimated values due-to matrix interference. However, if all parameters were met for the sample analysis such as ICV, CCV, MDL Check, LCS, and surrogate recoveries, but MS/MSD failed the QC limits, then the data must not be qualified. The only data that could be qualified are those of the sample that was utilized for the MS/MSD. The reviewer must be careful in qualifying data based on the MS/MSD results. The reviewer must establish a link between the MS/MSD failure and the sample data for justifying data qualification. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

2.10 SURROGATE RECOVERY

Criteria: Samples are spiked with surrogate compounds prior to addition of extracting solvents to samples. The surrogate recoveries for semivolatile organic compounds must be within acceptable limits of 50-150%.

Action: When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R. One surrogate from each fraction is allowed to be below the LL but greater than 10% without qualification.

2.11 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

2.12 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\text{\# usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\text{\# total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

3.0 PCB VALIDATION PROCEDURE

3.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with “J”, meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and non-detect with "R".

3.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run i.e. producing a linear calibration curve.

Action: The reviewer must verify that the initial calibration contained a minimum of five points for Aroclors 1016 and 1260 with a minimum of three peaks per arochlor per concentration. If less than five standards are used, qualify with R. If the mean RSD>20% or individual peaks of the minimum three display r<0.99 (r2<0.99), then apply R to all associated data. Of course only one passing column or detector is required for non-detects.

3.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 85-115%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 115%, apply J to all positives. For a result <85% and >80%, apply J/UJ. For recovery < 80%, apply J/R. Only one passing column or detector is required for non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a result <70% and >65%, apply J/UJ. For recovery < 65%, apply J/R. Only one passing column or detector is required for non-detects.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed and/or analytes were not detected, then a deficiency must be entered in the report, and qualify the non-detects with R.

3.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every twelve hours. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is: D or mean D ≤ 15% with a maximum %D<20% for a specific aroclor peak.

Action: For D>20% with a negative bias apply J to positives and R to non-detects; and with a positive bias apply J to positives only. Of course only one passing column or detector is required for non-detects.

3.5 METHOD BLANK

Criteria: If a Pesticide/PCB is found in the blank but not in the sample, no action is taken. Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X should apply.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 5X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 5 times the amount in any blank will be qualified "B", meaning that compound was found in the blank and may be present due to laboratory contamination.

3.6 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria. The common arochlors 1016 and 1260 are used as spiking constituents when the contaminants are unknown.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 30% and the lower limit (LL), flag J/UJ. For a recovery < 30%, flag J/R.

3.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: The MS/MSD results should not be used alone to qualify data. The reviewer should consider other quality control results in conjunction with professional judgment. Some sample results may be qualified "J", meaning estimated values due-to matrix interference. However, if all parameters were met for the sample analysis such as ICV, CCV, MDL Check, LCS, and surrogate recoveries, but MS/MSD failed the QC limits, then the data must not be qualified. The only data that could be qualified are those of the sample that was utilized for the MS/MSD. The reviewer must be careful in qualifying data based on the MS/MSD results. The reviewer must establish a link between the MS/MSD failure and the sample data for justifying data qualification. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

3.8 SURROGATE RECOVERY

Criteria: Samples are spiked with surrogate compounds prior to addition of extracting solvents to samples. The surrogate recoveries for PCB compounds must be within acceptable limits of 50-150%.

Action: When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R.

3.9 TARGET ANALYTE CONFIRMATION

Criteria: Whenever a positive hit is detected on one column it must be confirmed on a confirmation 2nd column.

Action: The RPD must be ≤ 40%. If the RPD > 40% apply J for positive results.

3.10 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

3.11 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\text{\# usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\text{\# total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

4.0 PESTICIDE VALIDATION PROCEDURE

4.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with “J”, meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

4.2 INSTRUMENT PERFORMANCE: DDT/ENDRIX BREAKDOWN

Criteria: The total percent breakdown of either DDT or endrin may not exceed 15%. The percent breakdown is the amount of decomposition that endrin and 4,4-DDT undergo when analyzed.

The retention time of DBC in each analysis must be compared to the retention time of DBC in the standard mix. The calculation of retention time windows will be calculated as three times the standard deviation of the retention time of three injections of a CCV over 72 hours, as in SW-846 8000B, section 7.6.

Action: If **DDT breakdown** is greater than 15%, beginning with the samples following the last in control standard: Flag all quantitative results for DDT as estimated (J). If DDT was not detected, but DDD and DDE were detected, then flag the quantitation limit for DDT as unreliable (R). Flag results for DDD and/or DDE as presumptively present at an estimated quantity (J).

If **Endrin breakdown** is greater than 15%: Flag all quantitative results for endrin as estimated (J). If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then flag the quantitation limit for endrin as unreliable (R) and decomposition compounds as estimated (J).

If the retention time criteria is not met for the particular column used for analysis, the data may be flagged unreliable (R), but qualification of the data is left up to the professional judgment of the reviewer.

4.3 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run i.e. producing a linear calibration curve.

Action: The reviewer must verify that the initial calibration contained a minimum of five points. If less than five standards are used, qualify with R. If a target displays an RSD>20% or r<0.99(r2<0.99), then apply R to all associated data. Only one passing column or detector is required for non-detects.

4.4 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 85-115%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 115%, apply J to all positives. For a result <85% and >80%, apply J/UJ. For recovery < 80%, apply J/R. Only one passing column or detector is required for non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a result >65% and <70%, apply J/UJ. For recovery < 65%, apply J/R. Only one passing column or detector is required for non-detects.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or analytes were not detected, then a deficiency must be entered in the report, and qualify the non-detects R.

4.5 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every twelve hours. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is: D or mean D ≤ 15% with a maximum %D<20% for a specific compound.

Action: If mean %D > 15% and D > 30% then R. If mean %D ≤ 15% and D > 30% then J. For D>20% with a negative bias apply J to positives and R to non-detects; and with a

positive bias apply J to positives only. Only one passing column or detector is required for non-detects.

4.6 METHOD BLANK

Criteria: If a Pesticide/PCB is found in the blank but not in the sample, no action is taken. Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X should apply.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 5X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 5 times the amount in any blank will be qualified "B", meaning that compound was found in the blank and may be present due to laboratory contamination.

4.7 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 30% and the lower limit (LL), flag J/UJ. For a recovery < 30%, flag J/R.

4.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: The MS/MSD results should not be used alone to qualify data. The reviewer should consider other quality control results in conjunction with professional judgment. Some sample results may be qualified "J", meaning estimated values due-to matrix interference. However, if all parameters were met for the sample analysis such as ICV, CCV, MDL Check, LCS, and surrogate recoveries, but MS/MSD failed the QC limits, then the data must not be qualified. The only data that could be qualified are those of the sample that was utilized for the MS/MSD. The reviewer must be careful in qualifying data based on the MS/MSD results. The reviewer must establish a link between the MS/MSD failure and the sample data for justifying data qualification.

The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

4.9 SURROGATE RECOVERY

Criteria: Samples are spiked with surrogate compounds prior to addition of extracting solvents to samples. The surrogate recoveries for pesticide analyses must be within acceptable limits of 50-150%.

Action: When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R.

4.10 TARGET ANALYTE CONFIRMATION

Criteria: Whenever a positive hit is detected in one column it must be confirmed on a confirmation 2nd column. The higher of the two corresponding results shall be reported unless a positive interferent is suspected on this result's column.

Action: The RPD must be ≤ 40%. If the RPD > 40% apply J to positive results.

4.11 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

4.12 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

5.0 NITROAROMATICS AND NITRAMINES BY HPLC (8330) VALIDATION PROCEDURE

5.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with "J", meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

5.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run and when producing a linear calibration curve.

Action: The reviewer must verify that the initial calibration standards contain both the target compounds and surrogate compounds (system monitoring compounds). The relative standard deviation (RSD) for each target analyte must be ≤ 20%.

If a target displays an RSD>20% or r<0.99 (r^2<0.99), then apply R to all associated data. Only one passing column or detector is required for non-detects.

5.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 85-115%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 115%, apply J. For a result <85% and >80%, apply J/UJ. For recovery < 80%, apply J to all positives and R to all non-detects. Only one passing column or detector is required for non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the %D is ≤ 30%.

Action: For results greater than 130%, apply J to all positives. For a result <70% and >65%, apply J/UJ. For recovery < 65% apply J/R. Only one passing column or detector is required for non-detects.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or analytes were not detected, then a deficiency must be entered in the report, and qualify the non-detect R.

5.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every twelve hours. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria are: D or mean D ≤ 15% with a maximum %D<30% for a specific compound.

Action: For D>15% with a negative bias apply J to positives and R to non-detects; and with a positive bias apply J to positives only. Only one passing column or detector is required for non-detects.

5.5 RETENTION TIME

Criteria: Retention time windows must be within 3 standard deviations from three checks on three consecutive days, as per SW-846 8000B, section 7.6.

Action: If retention time is outside the retention time windows, results will be rejected "R".

5.6 METHOD BLANK

Criteria: Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X rule should apply.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 5X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 5 times the amount in any blank 5 will be qualified “B”, meaning that compound was found in the blank and may be present due to laboratory contamination.

5.7 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 30% and the lower limit (LL), flag J/UJ. For a recovery < 30%, flag J/R.

5.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: The MS/MSD results should not be used alone to qualify data. The reviewer should consider other quality control results in conjunction with professional judgment. Some sample results may be qualified "J", meaning estimated values due-to matrix interference. However, if all parameters were met for the sample analysis such as ICV, CCV, MDL Check, LCS, and surrogate recoveries, but MS/MSD failed the QC limits, then the data must not be qualified. The only data that could be qualified are those of the sample that was utilized for the MS/MSD. The reviewer must be careful in qualifying data based on the MS/MSD results. The reviewer must establish a link between the MS/MSD failure and the sample data for justifying data qualification. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

5.9 SURROGATE RECOVERY

Criteria: Samples are spiked with surrogate compounds prior to addition of extracting solvents to samples. The surrogate recoveries compounds must be within acceptable limits of 50-150%.

Action: When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R.

5.10 TARGET ANALYTE CONFIRMATION

Criteria: Whenever a positive hit is detected in one column it must be confirmed on a confirmation 2nd column.

Action: The RPD must be ≤ 40%. If the RPD > 40% apply J to positive results.

5.11 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

5.12 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

6.0 POLYAROMATIC HYDROCARBON (8310) VALIDATION PROCEDURE

6.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with "J", meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

6.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run and when producing a linear calibration curve.

Action: The reviewer must verify that the initial calibration standards contain both the target compounds and surrogate compounds (system monitoring compounds). The relative standard deviation (RSD) for each target analyte must be ≤ 20%.

If a target displays an RSD>20% or r<0.995 (r^2<0.99), then apply R to all associated data. Only one passing column or detector is required for non-detects.

6.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 85-115%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 115%, apply J. For a result <85% and >80%, apply J/UJ. For recovery < 80% apply J to all positives and R to all non-detects. Only one passing column or detector is required for non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a result <70% and >65%, apply J/UJ. For recovery < 65%, apply J/R. Only one passing column or detector is required for non-detects.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or analytes were not detected, then a deficiency must be entered in the report, and qualify non-detect with R.

6.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every twelve hours. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is: D or mean D ≤ 15% with a maximum %D<30% for up to 5% of non-contaminants of concern.

Action: For D>15% with a negative bias apply J to positives and R to non-detects; and with a positive bias apply J to positives only. Only one passing column or detector is required for non-detects.

6.5 RETENTION TIME

Criteria: Retention time windows must be within 3 standard deviations from three checks on three consecutive days, as per SW-846 8000B, section 7.6.

Action: If retention time is outside the retention time windows, results will be rejected "R".

6.6 METHOD BLANK

Criteria: Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X should apply.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 5X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 5 times the amount in any blank will be qualified “B”, meaning that compound was found in the blank and may be present due to laboratory contamination.

6.7 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 30% and the lower limit (LL), flag J/UJ. For a recovery < 30%, flag J/R.

6.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: The MS/MSD results should not be used alone to qualify data. The reviewer should consider other quality control results in conjunction with professional judgment. Some sample results may be qualified "J", meaning estimated values due-to matrix interference. However, if all parameters were met for the sample analysis such as ICV, CCV, MDL Check, LCS, and surrogate recoveries, but MS/MSD failed the QC limits, then the data must not be qualified. The only data that could be qualified are those of the sample that was utilized for the MS/MSD. The reviewer must be careful in qualifying data based on the MS/MSD results. The reviewer must establish a link between the MS/MSD failure and the sample data for justifying data qualification. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

6.9 SURROGATE RECOVERY

Criteria: Samples are spiked with surrogate compounds prior to addition of extracting solvents to samples. The surrogate recoveries compounds must be within acceptable limits of 50-150%.

Action: When recovery is >UL flag positives with J. For recovery between 10% and the LL, flag J/UJ. For a surrogate recovery <10%, flag J/R.

6.10 TARGET ANALYTE CONFIRMATION

Criteria: Whenever a positive hit is detected in one column it must be confirmed on a confirmation 2nd column. The higher of the two corresponding results will be reported unless there is positive interference suspected on this result's column.

Action: The RPD must be ≤ 40%. If the RPD > 40% apply J for positive results. This would only pertain to a positive result.

6.11 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended

for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

6.12 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

7.0 ICP METALS (6010) VALIDATION PROCEDURE

7.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with "J", meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

7.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run and when producing a linear calibration curve.

Action: The reviewer must verify that the 3 standards and a blank were utilized in the initial calibration. The r ≥ 0.995. When <0.995, apply R.

7.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 90-110%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 110%, apply J. For a results < 90% and > 85%, apply J/UJ. For recovery < 85%, apply J to all positives and R to all non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a results < 65% and > 70%, apply J/UJ. For recovery < 65% apply J/R.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed, then a deficiency must be entered in the report, and qualification may be necessary for the none-detect analytes.

7.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every ten samples and at the end of an analytical sequence. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is 90-110%.

Action: For results greater than 110%, apply J. For a results < 90% and > 85%, apply J/UJ. For recovery < 85% apply J to all positives and R to all non-detects.

7.5 BLANKS

Criteria: The criteria for assessment of blanks are to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the sample. If contamination problems with any blank exist, all data associated with the analytical method must be carefully evaluated to determine whether or not there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data. There should be no contaminant in the blank(s) at > 1/2 MRL

Action: Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results above the MRL but <5 times the amount in the blank will be considered artifacts of contamination and qualified "B". The sample result will be regarded as non-detect.

The actual comparison of blank and sample results will be based on actual instrument values, particularly for soil matrix. This approach will eliminate variability in results due to sample weight/volume, percent moisture, etc., which vary from sample to sample. In instances where more than one blank is associated with a given sample, qualification should be based upon the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank values.

7.6 ICP INTERFERENCE CHECK SAMPLE (ICS)

Criteria: The ICP interference check sample verifies laboratory interelement and background corrective factors. An ICS must be run to determine interelemental effect on sample results. Results of the ICSAB solution must fall within the control limits of +20% of the true value. ICSA: No detection of other elements >1/2 MRL (up to 5% may exceed).

Recalculation of %R for one or more recoveries will be performed using the equation below and to verify that values agree with the laboratory reported values.

$$\text{ICS \%R} = \frac{\text{Found Solution}}{\text{True Solution}} \times 100$$

Action: Only for samples with concentrations approximate to [ICS]. For recovery >120%, apply J to positives that are known to be interfered by the failing metal. For recovery in between 50-79%, apply J to positives and UJ to non-detects that are known to be interfered by the failing metal. For recovery <50%, apply R to metals that are known to be interfered by the failing metal. For detections of other elements, use professional judgment. The results for non-spiked elements in the ICSA may be used to determine the interference and a factor of five may be used to measure the magnitude of interence.

7.7 LABORATORY CONTROL SAMPLE (LCS)

Criteria: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Action: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 60%and the lower limit (LL), flag J/UJ. For a recovery < 60%, flag J/R.

7.8 MATRIX SPIKE

Criteria: The MS results are designed to determine the precision of the analytical method on various matrices pre-digestion. Given that every matrix is unique, the use of MS for precision may be specific to individual samples. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more. In this case, the sample duplicate and serial dilution results will apply.

Action: For recoveries greater than 125%, flag positive associated sample results JI. For recoveries between 30-75% flag positive JI and non-detects UJI. For recoveries less than 30%, flag positive JI and non-detects RI. The I flag must also be incorporated when flagging due to matrix effects (ie: “JI”).

7.9 MATRIX DUPLICATE

Criteria: Duplicate analyses are indicators of laboratory precision based on each sample matrix. A control limit of 20%RPD shall be used for sample values >MRL.

Action: Flag all positives for the analytes out with a J in associated samples.

7.10 POST DIGESTION SPIKE

Criteria: A post digestion spike will be performed as needed to confirm matrix effect. The recovery = 75-125%.

Action: For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag non-detect R and positive J.

7.11 SERIAL DILUTION

Criteria: The Serial Dilution determines whether significant physical or chemical interferences exist due to sample matrix.

If the analyte concentration is sufficiently high, an analysis of a 4-fold dilution must agree within 10% Difference (%D) of the original results.

Action: When criteria are not met, qualify the associated positive data as estimated (J).

7.12 METHOD OF STANDARD ADDITIONS

Criteria: The Method of Standard Additions must have a minimum of three spiked sample concentration to create a linear regression. This method is employed when the degree of matrix interference affects the pre-spike sample, post-spike sample, matrix duplicate, and serial dilution results.

Action: As per Section 7 of this quidance, the method of standard additions must be performed when the criteria for certain QC elements are not met. If this method was not performed as required, flag non-detects R and positives J.

7.13 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

7.14 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

8.0 GFAA METALS VALIDATION PROCEDURE

8.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with “J”, meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data.. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

8.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run and when producing a linear calibration curve.

Action: The reviewer must verify that the 3 standards and a blank were utilized in the initial calibration. The r ≥ 0.995. When <0.995, apply R.

8.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 80-120%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 120%, apply J. For a results < 80% and > 75%, apply J/UJ. For recovery < 75% apply J to all positives and R to all non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a result >65% and <70%, apply J/UJ. For recovery < 65% apply J/R.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or when the analyte is not detected, then a deficiency must be entered in the report, and qualify non-detects with R.

8.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every ten samples and at the end of an analytical sequence. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is 80-120%.

Action: For results greater than 120%, apply J. For a results < 80% and > 75%, apply J/UJ. For recovery <75% apply J to all positives and R to all non-detects.

8.5 BLANKS

Criteria: The criteria for assessment of blanks are to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the sample. If contamination problems with any blank exist, all data associated with the analytical method must be carefully evaluated to determine whether or not there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data. There should be no contaminant in the blank(s) at > 1/2 MRL

Action: Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results above the MRL but <5 times the amount in the blank will be considered artifacts of contamination and qualified "B". The sample result will be regarded as non-detect.

The actual comparison of blank and sample results will be based on actual instrument values, particularly for soil matrix. This approach will eliminate variability in results due to sample weight/volume, percent moisture, etc., which vary from sample to sample. In instances where more than one blank is associated with a given sample, qualification should be based upon the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank values.

8.6 LABORATORY CONTROL SAMPLE (LCS)

Criteria: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria. The control limits are 80-120%

Action: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 50%and the lower limit (LL), flag J/UJ. For a recovery < 50%, flag J/R.

8.7 MATRIX SPIKE

Criteria: The MS results are designed to determine the precision of the analytical method on various matrices pre-digestion. Given that every matrix is unique, the use of MS for precision may be specific to individual samples. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more. In this case, the sample duplicate and serial dilution results will apply.

Action: For recoveries greater than 125%, flag positive associated sample results J. For recoveries between 30-75% flag positive J and non-detects UJ. For recoveries less than 30%, flag positive J and non-detects R. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

8.8 MATRIX DUPLICATE

Criteria: Duplicate analyses are indicators of laboratory precision based on each sample matrix. A control limit of 20%RPD shall be used for sample values >MRL.

Action: Flag all positives for the analytes out with a J in associated samples.

8.9 POST DIGESTION SPIKE

Criteria: A post digestion spike will be performed as needed to confirm matrix effect. The recovery = 75-125%.

Action: For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag positives J and non-detects R.

8.10 SERIAL DILUTION

Criteria: The Serial Dilution determines whether significant physical or chemical interferences exist due to sample matrix.

If the analyte concentration is sufficiently high, an analysis of a 4-fold dilution must agree within 10% Difference (%D) of the original results.

Action: When criteria are not met, qualify the associated positive data as estimated (J).

8.11 METHOD OF STANDARD ADDITIONS

Criteria: The Method of Standard Additions must have a minimum of three spiked sample concentration to create a linear regression. This method is employed when the degree of matrix interference affects the pre-spike sample, post-spike sample, matrix duplicate, and serial dilution results.

Action: As per Section 7 of this quidance, the method of standard additions must be performed when the criteria for certain QC elements are not met. If this method was not performed as required, flag non-detects R and positives J.

8.12 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

8.13 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

9.0 CVAA METALS VALIDATION PROCEDURE

9.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with “J”, meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

9.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run and when producing a linear calibration curve.

Action: The reviewer must verify that the 5 standards and a blank were utilized in the initial calibration. The r ≥ 0.995. When <0.995, apply R.

9.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 80-120%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 120%, apply J. For a results < 80% and > 75%, apply J/UJ. For recovery < 75% apply J to all positives and R to all non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a results >65% and <70%, apply J/UJ. For recovery < 65% apply J/R.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or the analyte is not detected, then a deficiency must be entered in the report, and qualify the non-detects R.

9.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every ten samples and at the end of an analytical sequence. Continuing calibration provides a means to measure or check performance on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is 80-120%.

Action: For results greater than 120%, apply J. For a results < 80% and > 75%, apply J/UJ. For recovery <75% apply J to all positives and R to all non-detects.

9.5 BLANKS

Criteria: The criteria for assessment of blanks are to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the sample. If contamination problems with any blank exist, all data associated with the analytical method must be carefully evaluated to determine whether or not there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data. There should be no contaminant in the blank(s) at > 1/2 MRL

Action: Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results above the MRL but <5 times the amount in the blank will be considered artifacts of contamination and qualified "B". The sample result will be regarded as non-detect.

The actual comparison of blank and sample results will be based on actual instrument values, particularly for soil matrix. This approach will eliminate variability in results due to sample weight/volume, percent moisture, etc., which vary from sample to sample. In instances where more than one blank is associated with a given sample, qualification should be based upon the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank values.

9.6 LABORATORY CONTROL SAMPLE (LCS)

Criteria: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria. The control limits are 80-120%

Action: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 50% and the lower limit (LL), flag J/UJ. For a recovery < 50%, flag J/R.

9.7 MATRIX SPIKE

Criteria: The MS results are designed to determine the precision of the analytical method on various matrices pre-digestion. Given that every matrix is unique, the use of MS for precision may be specific to individual samples. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more. In this case, the sample duplicate and serial dilution results will apply.

Action: For recoveries greater than 125%, flag positive associated sample results J. For recoveries between 30-75% flag positive J and non-detects UJ. For recoveries less than 30%, flag positive J and non-detects R. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

9.8 MATRIX DUPLICATE

Criteria: Duplicate analyses are indicators of laboratory precision based on each sample matrix. A control limit of 20%RPD shall be used for sample values >MRL.

Action: Flag all positives for the analytes out with a J in associated samples.

9.9 POST DIGESTION SPIKE

Criteria: A post digestion spike will be performed as needed to confirm matrix effect. The recovery = 75-125%.

Action: For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag positives J and non-detects R.

9.10 SERIAL DILUTION

Criteria: The Serial Dilution determines whether significant physical or chemical interferences exist due to sample matrix.

If the analyte concentration is sufficiently high, an analysis of a 4-fold dilution must agree within 10% Difference (%D) of the original results.

Action: When criteria are not met, qualify the associated positive data as estimated (J).

9.11 METHOD OF STANDARD ADDITIONS

Criteria: The Method of Standard Additions must have a minimum of three spiked sample concentration to create a linear regression. This method is employed when the degree of matrix interference affects the pre-spike sample, post-spike sample, matrix duplicate, and serial dilution results.

Action: As per Section 7 of this quidance, the method of standard additions must be performed when the criteria for certain QC elements are not met. If this method was not performed as required, flag non-detects R and positives J.

9.12 CONTROL CHARTS

The use of control charts is one of the most common statistical tools to monitor the quality of the analysis. For small sample delivery groups, LCS data should be obtained from the laboratory for the period the samples were analyzed, in order for the validator to prepare control charts. However, control charts for surrogate recoveries, and MS/MSD obtained from the laboratory will be of little or no value since these charts would represent different matrices. It is recommended for the reviewer to plot control chart for the surrogate recoveries, MS/MSD, and the RPDs. Any outliers on the control charts (lies outside of the upper/lower Warning limits) should be investigated by the reviewer. In most cases there are causes for these outliers rather than matrix effect. The Validator must provide his findings for the cause(s) behind the outliers. Control charts should be included in an appendix attached to validation reports.

9.13 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

10.0 CYANIDE VALIDATION PROCEDURE

10.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with "J", meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

10.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run and when producing a linear calibration curve.

Action: The reviewer must verify that the 6 standards and a blank were utilized in the initial calibration. The r ≥ 0.995. When <0.995, apply R.

10.3 INITIAL CALIBRATION VERIFICATION (ICV)

Criteria: The Validator must check that initial calibration verification was prepared from a 2nd source, and the recovery is within 85-115%. If the ICV was prepared from the same source of the initial calibration, then qualify data with "R", and enter a comment as a major deficiency in the laboratory procedure.

Action: For results greater than 115%, apply J. For a results < 85% and > 80%, apply J/UJ. For recovery < 80% apply J to all positives and R to all non-detects.

Criteria: The Validator must check the "D" for the QC/MRL (low ICV) and that the recovery is within 70-130%.

Action: For results greater than 130%, apply J to all positives. For a results >65% and <70%, apply J/UJ. For recovery < 65%, apply J/R.

Analyst must assure the data user that the MDL has not deviated by detecting analytes at levels of the MDL check (2 X MDL). If MDL check was not performed or the analyte was not detected, then a deficiency must be entered in the report, and qualify the non-detects R.

10.4 CONTINUING CALIBRATION VERIFICATION (CCV)

Criteria: In order to ensure that the initial calibration curve is still usable for determining the concentration of the target compounds during analysis, the analyst must conduct a CCV every ten samples and at the end of an analytical sequence. Continuing calibration provides a means to measure or check performance on a daily basis. It also

provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The acceptance criteria is 80-120%.

Action: For results greater than 120%, apply J. For a results < 80% and > 75%, apply J/UJ. For recovery <75% apply J to all positives and R to all non-detects.

10.5 BLANKS

Criteria: The criteria for assessment of blanks are to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the sample. If contamination problems with any blank exist, all data associated with the analytical method must be carefully evaluated to determine whether or not there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data. There should be no contaminant in the blank(s) at > 1/2 MRL

Action: Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results above the MRL but <5 times the amount in the blank will be considered artifacts of contamination and qualified "B". The sample result will be regarded as non-detect.

The actual comparison of blank and sample results will be based on actual instrument values, particularly for soil matrix. This approach will eliminate variability in results due to sample weight/volume, percent moisture, etc., which vary from sample to sample. In instances where more than one blank is associated with a given sample, qualification should be based upon the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank values.

10.6 LABORATORY CONTROL SAMPLE (LCS)

Criteria: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria. The control limits are 80-120%

Action: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 50% and the lower limit (LL), flag J/UJ. For a recovery < 50%, flag J/R.

10.7 MATRIX SPIKE

Criteria: The MS results are designed to determine the precision of the analytical method on various matrices pre-digestion. Given that every matrix is unique, the use of MS for precision may be specific to individual samples. Spike recovery (%R) must be within the limits of 80-120%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more. In this case,

the sample duplicate and serial dilution results will apply.

Action: For recoveries greater than 125%, flag positive associated sample results J. For recoveries between 30-75% flag positive J and non-detects UJ. For recoveries less than 30%, flag positive J and non-detects R. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

10.8 MATRIX DUPLICATE

Criteria: Duplicate analyses are indicators of laboratory precision based on each sample matrix. A control limit of 20%RPD shall be used for sample values >MRL.

Action: Flag all positives for the analytes out with a J in associated samples.

10.9 POST DIGESTION SPIKE

Criteria: A post digestion spike will be performed as needed to confirm matrix effect. The recovery = 75-125%.

Action: For positive associated results where recovery is >125% or between 30-75%, flag with J. For non-detect associated results where recovery is between 30-74%, flag UJ. All results associated with an analyte recovery <30%, flag positives J and non-detects R.

10.10 SERIAL DILUTION

Criteria: The Serial Dilution determines whether significant physical or chemical interferences exist due to sample matrix.

If the analyte concentration is sufficiently high, an analysis of a 4-fold dilution must agree within 10% Difference (%D) of the original results.

Action: When criteria are not met, qualify the associated positive data as estimated (J).

10.11 METHOD OF STANDARD ADDITIONS

Criteria: The Method of Standard Additions must have a minimum of three spiked sample concentration to create a linear regression. This method is employed when the degree of matrix interference affects the pre-spike sample, post-spike sample, matrix duplicate, and serial dilution results.

Action: As per Section 7 of this quidance, the method of standard additions must be performed when the criteria for certain QC elements are not met. If this method was not performed as required, flag non-detects R and positives J.

10.12 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method

using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

11.0 SPECIAL PARAMETERS VALIDATION PROCEDURE

11.1 HOLDING TIMES

Criteria: See Appendix D

Action: If holding times are exceeded, all positive results shall be qualified with “J”, meaning estimated values, and non-detects with "UJ". If holding times are exceeded by twice the holding time, the reviewer must use professional judgment to determine the validity of the data. Qualify positive results ≥ 10 MRL with "J", positive hits < 10MRL and none- detect with "R".

11.2 INITIAL CALIBRATION

Criteria: Initial calibration is designed to measure instrument performance and demonstrate that the instrument is capable of generating acceptable values. This is performed at the beginning of the analytical run.

Action: As applicable for the analysis.

11.3 METHOD BLANK

Criteria: Blank samples must be free of any contaminants. All positive hits must be checked for possible lab contaminants, by checking presence of contaminants in the blanks. The 5X and 10X rule should apply. Apply the 10X rule for common lab contaminants.

Action: Blanks must be evaluated based on the levels with respect to the levels of the Method Reporting Limit (MRL). Method blanks should not be considered contaminated when levels of compounds appear at or below 1/2 MRL levels. If levels of contaminant are > 1/2 MRL, then make a comment in the report that these blanks are contaminated. The Validator must evaluate sample results to determine the levels of contamination in the analytical batch. If the analyte has concentrations ≥ than 10X the levels of the compound in the blank, then the compound should be considered present in the sample, and should not be qualified. However, any sample result below 10 times the amount in any blank for common contaminants, or 5 times the amount for other compounds will be qualified “B”, meaning that compound was found in the blank and may be present due to laboratory contamination.

11.4 LABORATORY CONTROL SAMPLE (LCS)

Action: Since the LCS is prepared by spiking target compounds of interest in a pure matrix that is interference free, meeting the QC limits is of vital importance in assuring that the instrument and the method are within the optimum and acceptance criteria.

Criteria: Limits are provided in Appendix C. Analysis should not be conducted when LCS results are out of the QC limits. When recovery is > upper limit (UL) flag positives with J. For recovery between 50% and the lower limit (LL), flag J/UJ. For a recovery < 50%, flag J/R.

11.5 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

Criteria: The MS/MSD results, as applicable, are designed to determine the precision of the analytical method on various matrices. Given that every matrix is unique, the use of MS/MSD for precision may be specific to individual samples.

Action: As applicable to analysis. The I flag must also be incorporated when flagging due to matrix effects (ie: "JI").

11.6 DATA QUALITY

The reviewer must provide a table summarizing percent usability for each analytical method using the following formula:

$$\%\text{Usability} = \frac{[(\#\text{ usable samples}) \times (\text{total analytes})] - \text{unusable analytes}}{[(\#\text{ total samples}) \times (\text{total analytes})]} \times 100$$

Analytical data will be acceptable when % usability is ≥ 90. If % usability < 90, then the data is not acceptable, and the Validator may consider validating more analytical batches.

ATTACHMENT B
DATA VALIDATION CHECK LIST

VOLATILE ORGANIC ANALYSIS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group (SDG): _____

	Yes	No
1. Holding Time:		
(a) Were samples preserved?	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]
2. Was the BFB tune performed at the beginning of each 12-hour period during which samples were analyzed?	[ ]	[ ]
3. Was mass assignment based on m/z 95?	[ ]	[ ]
4. Indicate if BFB ions abundance relative to m/z 95 base peak met the ions abundance criteria:		

m/z	Acceptance Criteria	Yes	No
50	15.0 - 40.0 %	[ ]	[ ]
75	30.0 - 66.0 %	[ ]	[ ]
95	**100%, Base Peak**	[ ]	[ ]
96	5.0 - 9.0%	[ ]	[ ]
173	<2.0% of m/z 174	[ ]	[ ]
174	>50%	[ ]	[ ]
175	5.0 - 9.0% of mass 174	[ ]	[ ]
176	95.0 - 101.0% of m/z 174	[ ]	[ ]
177	5.0 - 9.0% of m/z 176	[ ]	[ ]

The relative ion abundance of m/z 95/96, m/z 174/176, and 176/177 are of critical importance.

The relative ion abundance of m/z 50 and 75 are of lower importance.

	Yes	No
5. Initial Calibration:		
• Did the initial calibration consist of five standards?	[ ]	[ ]
• Did the System Performance Check Compounds (SPCC) meet the minimum mean response factor (RF)?		
RF		
Chloromethane 0.1	[ ]	[ ]
1,1-Dichloroethane 0.1	[ ]	[ ]
Bromoform 0.1	[ ]	[ ]
Chlorobenzene 0.3	[ ]	[ ]
1,1,2,2-Tetrachloroethane 0.3	[ ]	[ ]
• Did the RSD meet the criteria ≤ 30% for each individual Calibration Check Compound (CCC)?		
1,1-Dichloroethene	[ ]	[ ]
Chloroform	[ ]	[ ]
1,2-Dichloropropane	[ ]	[ ]
Toluene	[ ]	[ ]
Ethylbenzene	[ ]	[ ]
Vinyl chloride	[ ]	[ ]
• Are the RSDs for the remaining target analytes ≤ 15% or r ≥ 0.99 with a mean RSD ≤ 15% with a maximum RSD ≤ 20%?	[ ]	[ ]
If the answer is "No", are the mean RSDs ≤ 15%?	[ ]	[ ]
• Was manual integration "M" performed?	[ ]	[ ]
If the answer is "Yes", check for supporting documents.	[ ]	[ ]
• Was the manual integration necessary?		
If the answer is "No", contact the laboratory inquiring about the reasons behind the manual integration, and **inform the District Chemist immediately if there were no valid reasons**.	[ ]	[ ]
6. QCMDL:	[ ]	[ ]
• Was MDL Check performed?		
7. QCMRL:	[ ]	[ ]

	Yes	No
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours?	[ ]	[ ]
• Was the QC/MRL between 70-130% R	[ ]	[ ]
• For the non-contaminants of concern was the QC/MRL between 60-140% (Sporadic Marginal Failure)	[ ]	[ ]

8. Initial Calibration Verification (ICV):

• Is the mid level (2nd source) recovery within 80 - 120% for contaminants of concern ? • Is the mid level (2nd source) recovery within 60-140% for non-contaminants of concern (Sporadic Marginal Failure)?	[ ]	[ ]

9. Continuing Calibration Verification (CCV):

• Was CCV conducted every 12 hours?	[ ]	[ ]
• Did SPCC meet the RF values?	[ ]	[ ]

RF

Chloromethane	0.1	[ ]	[ ]
1,1-Dichloroethane	0.1	[ ]	[ ]
Bromoform	0.1	[ ]	[ ]
Chlorobenzene	0.3	[ ]	[ ]
1,1,2,2-Tetrachloroethane	0.3	[ ]	[ ]

• Did the CCC meet the minimum requirements (D ≤ 20%)?	[ ]	[ ]
1,1-Dichloroethene	[ ]	[ ]
Chloroform	[ ]	[ ]
1,2-Dichloropropane	[ ]	[ ]
Toluene	[ ]	[ ]
Ethylbenzene	[ ]	[ ]
Vinyl chloride	[ ]	[ ]
• Primary Evaluation: Was the mean, Drift or D ≤ 20% from the initial calibration?	[ ]	[ ]

• Alternative Evaluation: Maximum allowable Drift/D for

	Yes	No
each target analyte is ≤ 30% when mean D ≤ 20%?	[ ]	[ ]

10. Sample Analysis:

• Was the RRT of an identified component within ± 0.06 RRT units of the RRT of the standard component?	[ ]	[ ]
• Did the abundance of ions in the sample spectra agree within 30% of the major ions (> 10% of the base ion) in the standard spectra?	[ ]	[ ]
• Were the internal standard areas within the QC limits (from -50% to +200%)?	[ ]	[ ]

11. Sample Quality Control:

• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS/MSD: Were the percent recoveries within limits?	[ ]	[ ]
Were the RPD within control limits?	[ ]	[ ]
System Monitoring Compounds (Surrogates): are surrogate recoveries within QC limits (50-150%)?	[ ]	[ ]

12. Comments (attach additional sheets if necessary):

_____

Validated/Reviewed by:
Signature:

_____ Date: _____

Name: _____

SEMIVOLATILE ORGANIC ANALYSIS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Sample Holding Time:		
(a) Were samples extracted within holding time?	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]
2. Instrument Tuning:		
Was the DFTPP tune performed at the beginning of each 12-hour period during which samples were analyzed?	[ ]	[ ]
3. Ion Mass Assignments:		
Was mass assignment based on m/z 198?	[ ]	[ ]
4. Ion Abundance:		
Indicate if DFTPP ions abundance relative to m/z 198 base peak met the ions abundance criteria:		
m/z — Acceptance Criteria		
51 — 30.0 - 60.0 %	[ ]	[ ]
68 — < 2% of mass 69	[ ]	[ ]
70 — < 2% of mass 69	[ ]	[ ]
127 — 40-60%	[ ]	[ ]
197 — < 1%	[ ]	[ ]
198 — 100%, Base peak	[ ]	[ ]
199 — 5-9%	[ ]	[ ]
275 — 10 - 30%	[ ]	[ ]
365 — > 1%	[ ]	[ ]
441 — present but < mass 443	[ ]	[ ]
442 — > 40%	[ ]	[ ]
443 — 17-23% of mass 442	[ ]	[ ]

		Yes	No
5.0 Initial Calibration:			
• Did the initial calibration consist of five or more standards?	5-stds	[ ]	[ ]
	more	[ ]	[ ]
If the calibration curve consists of 5-standards, check validity of the calibration model.			
Was the linear model applied?		[ ]	[ ]
• Did the followings System Performance Check Compounds (SPCC) meet the minimum mean response factor (RF)?			
	RF		
N-nitroso-di-n-propylamine	0.05	[ ]	[ ]
Hexachlorocyclopentadiene	0.05	[ ]	[ ]
2,4-dinitrophenol	0.05	[ ]	[ ]
4-nitrophenol	0.05	[ ]	[ ]
• Did the RSD meet the criteria ≤ 30% for the followings each individual Calibration Check Compound (CCC)?			
Base/Neutral Fraction:			
Acenaphthene		[ ]	[ ]
1,4-Dichlorobenzene		[ ]	[ ]
Hexachlorobutadiene		[ ]	[ ]
Diphenylamine		[ ]	[ ]
Di-n-octylphthalate		[ ]	[ ]
Fluoranthene		[ ]	[ ]
Benzo(a)pyrene		[ ]	[ ]
Acid Fraction:			
4-Chloro-3-methylphenol		[ ]	[ ]
2,4-Dichlorophenol		[ ]	[ ]
2-Nitrophenol		[ ]	[ ]
Phenol		[ ]	[ ]
Pentachlorophenol		[ ]	[ ]
2,4,6-Trichlorophenol		[ ]	[ ]
• Are the RSDs for the remaining target analytes ≤ 15%?		[ ]	[ ]
• If the answer is "No", are the mean RSDs ≤ 15% or r ≥ 0.99 with a mean RSD ≤ 15% with a maximum RSD ≤ 30%?		[ ]	[ ]
		[ ]	[ ]

	Yes	No
• Was manual integration "M" performed?	[ ]	[ ]
If the answer is "Yes", check for supporting documents.		
• Was the manual integration necessary?	[ ]	[ ]
If the answer is "No", contact the laboratory inquiring about the reasons behind the manual integration, and **inform the District Chemist immediately if there were no valid reasons**.		

6. QCMDL:

• Was MDL Check performed?	[ ]	[ ]

7. QCMRL:

• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours?	[ ]	[ ]
• Was the QC/MRL between 70-130% R	[ ]	[ ]
• For the non-contaminants of concern was the QC/MRL between 50-150% (Sporadic Marginal Failure)?	[ ]	[ ]

8. Initial Calibration Verification (ICV):

• Is the mid level (2nd source) recovery within 70-130% for contaminants of concern ?	[ ]	[ ]
• Is the mid level (2nd source) recovery within 50-150% for non-contaminants of concern (Sporadic Marginal Failure)?	[ ]	[ ]

9. Continuing Calibration Verification (CCV):

• Was CCV conducted every 12 hours?	[ ]	[ ]
• Did any of SPCC meet the minimum RF values?	[ ]	[ ]

		Yes	No
N-nitroso-di-n-propylamine	0.05	[ ]	[ ]
Hexachlorocyclopentadiene	0.05	[ ]	[ ]
2,4-dinitrophenol	0.05	[ ]	[ ]
4-nitrophenol	0.05	[ ]	[ ]

- Did the CCC meet the minimum requirements (D ≤ 20%) for the followings?

Base/Neutral Fraction:

Acenaphthene	[ ]	[ ]
1,4-Dichlorobenzene	[ ]	[ ]
Hexachlorobutadiene	[ ]	[ ]
Diphenylamine	[ ]	[ ]
Di-n-octylphthalate	[ ]	[ ]
Fluoranthene	[ ]	[ ]
Benzo(a)pyrene	[ ]	[ ]

Acid Fraction:

4-Chloro-3-methylphenol	[ ]	[ ]
2,4-Dichlorophenol	[ ]	[ ]
2-Nitrophenol	[ ]	[ ]
Phenol	[ ]	[ ]
Pentachlorophenol	[ ]	[ ]
2,4,6-Trichlorophenol	[ ]	[ ]

- Primary Evaluation: Was Drift or D ≤ 20% calculated from the initial calibration? [ ] [ ]
- Alternative Evaluation: Maximum allowable Drift/D for each target analyte is ≤ 30%. [ ] [ ]

10. Sample Analysis:

- Was the RRT of an identified component within ± 0.06 RRT units of the RRT of the standard component? [ ] [ ]
- Did the abundance of ions in the sample spectra agree within 30% of the major ions (> 10% of the base ion) in the standard spectra? [ ] [ ]
- Were the internal standard areas within the QC limits (from -50% to +200%)? [ ] [ ]

11. Sample Quality Control:

	Yes	No
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS/MSD: Were the percent recoveries within limits?	[ ]	[ ]
Were the RPD within control limits?	[ ]	[ ]
• System Monitoring Compounds (Surrogates): are surrogate recoveries within QC limits?	[ ]	[ ]

12. Comments (attach additional sheets if necessary):

_____
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_____

Validated/Reviewed by:

Signature: _____ Date: _____

Name: _____

POLY CHLORINATED BIPHENYLS (PCB/AROCLORS) CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time:		
(a) Were samples extracted within holding time?	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of five standards?	[ ]	[ ]
• Did Aroclors 1016 and 1260 meet the RSD ≤ 20% or the r ≥ 0.99?	[ ]	[ ]
• Was manual integration "M" performed? If the answer is "Yes", check for supporting documents.	[ ]	[ ]
• Was the manual integration necessary?	[ ]	[ ]
If the answer is "no", contact the laboratory inquiring about the reasons behind the manual integration, and **inform the District Chemist immediately if there were no valid reasons.**		
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
4. QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours??	[ ]	[ ]
• Was the QC/MRL between 70-130% R	[ ]	[ ]
5. Initial Calibration Verification (ICV):		
Is the mid level (2nd source) recovery within 85 - 115%?	[ ]	[ ]

	Yes	No
6. Continuing Calibration Verification (CCV):		
• Was CCV conducted every 12 hours?	[ ]	[ ]
• Was Drift or D ≤ 15% from the initial calibration with a maximum %D < 20% for a specific compound?	[ ]	[ ]
7. Sample Analysis:		
• Was the RRT of an identified component within the retention time window created as SW-846 requires?	[ ]	[ ]
• Were samples with levels higher than the calibration range (E), diluted and re-analyzed?	[ ]	[ ]
• Were identified Aroclors confirmed on a second GC column?	[ ]	[ ]
• Were individual Aroclor standards used to determine the pattern of the peaks? (Individual Aroclors are 1221, 1232, 1242, 1248, and 1254. Both Aroclor 1016, and 1260 can be used from the mixed calibration standards.)	[ ]	[ ]
• Was RPD of target analyte conformation ≤ 40?	[ ]	[ ]
8. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS/MSD: Were the percent recoveries within limits?	[ ]	[ ]
Were the RPDs within control limits?	[ ]	[ ]
• System Monitoring Compounds (Surrogates): are surrogate recoveries within QC limits?	[ ]	[ ]

9. Comments (attach additional sheets if necessary):

Validated/Reviewed by:

Signature: Date:

Name:

ORGANOCHLORINE PESTICIDES ANALYSIS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time:		
(a) Were samples extracted within holding time?	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]
2. DDT/Endrin Breakdown:		
• Was breakdown ≤ 15%?	[ ]	[ ]
3. Initial Calibration:		
• Did the initial calibration consist of five standards?	[ ]	[ ]
• Did all compounds meet the RSD ≤ 20% or r ≥ 0.99?	[ ]	[ ]
• Was manual integration "M" performed? If the answer is "Yes", check for supporting documents.	[ ]	[ ]
• Was the manual integration necessary?	[ ]	[ ]
If the answer is "no", contact the laboratory inquiring about the reasons behind the manual integration, and **inform the District Chemist immediately if there were no valid reasons.**		
4. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
5. QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours??	[ ]	[ ]
• Was the QC/MRL between 70-130% R	[ ]	[ ]

	Yes	No
6. Initial Calibration Verification (ICV):		
• Is the mid level (2nd source) recovery within 85 - 115%?	[ ]	[ ]
7. Continuing Calibration Verification (CCV):		
• Was CCV conducted every 12 hours?	[ ]	[ ]
• Was Drift or D ≤ 15% from the initial calibration with a maximum D ≤ 20% for a specific compound?	[ ]	[ ]
8. Sample Analysis:		
• Was the RRT of an identified component within the retention time window created as SW-846 requires?	[ ]	[ ]
• Were samples with levels higher than the calibration range (E), diluted and re-analyzed?	[ ]	[ ]
• Were identified compounds confirmed on a second GC column?	[ ]	[ ]
• Was RPD of target analyte confirmation ≤ 40?	[ ]	[ ]
9. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS/MSD: Were the percent recoveries within limits?	[ ]	[ ]
Were the RPD within control limits?	[ ]	[ ]
• System Monitoring Compounds (Surrogates): are surrogate recoveries within QC limits?	[ ]	[ ]

10. Comments (attach additional sheets if necessary):

Validated/Reviewed by:

Signature: Date:

Name:

NITROAROMATICS & NITRAMINE DATA ANALYSIS (EXPLOSIVE RESIDUES) CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time: Were samples analyzed within holding time?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of five standards?	[ ]	[ ]
• Did the RSD meet the criteria ≤ 20% for each individual Calibration Compound or r ≥ 0.99?	[ ]	[ ]
• Was manual integration "M" performed? If the answer is "Yes", check for supporting documents.	[ ]	[ ]
• Was the manual integration necessary?	[ ]	[ ]
If the answer is "no", contact the laboratory inquiring about the reasons behind the manual integration, and **inform the District Chemist immediately if there were no valid reasons.**		
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
4. QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours??	[ ]	[ ]
• Was the percentage "D" for QC/MRL ≤ 30%?	[ ]	[ ]
5. Initial Calibration Verification (ICV):	[ ]	[ ]

	Yes	No
• Was the ICV made of a 2nd source?	[ ]	[ ]
• Was the mid level (2nd source) recovery within 85 - 115%?		
6. Continuing Calibration Verification (CCV): {Daily calibration}		
• Was midpoint calibration standard conducted at the beginning of the day?	[ ]	[ ]
• Was midpoint calibration standard conducted every ten samples or every twelve hours?	[ ]	[ ]
• Was midpoint calibration standard conducted after the last sample of the day?	[ ]	[ ]
• Did the CCV meet the minimum requirements (D ≤ 15% with a maximum D ≤ 20% for a specific compound if the mean D ≤ 15%)?	[ ]	[ ]
7. Sample Analysis:		
• Was the RRT of an identified component within the retention time window created as SW-846 requires?	[ ]	[ ]
• Were all identified hits, above the initial calibration curve, diluted and reanalyzed?	[ ]	[ ]
• Were all identified hits confirmed on a second column?	[ ]	[ ]
• Was RPD of target analyte confirmation ≤ 40?	[ ]	[ ]
• Was there a shoulder on the 2,4,6-TNT peak?	[ ]	[ ]
If the answer is "Yes", then tetryl decomposition is suspected. Peak height rather than peak area should be used for calculating TNT concentration. If teryl was identified in aqueous samples, was pH adjusted to <3? If the answer is "No", then check for tetryl decomposition, and qualify hits with **"J"** accordingly.	[ ]	[ ]
8. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]

	Yes	No
• MS/MSD: Were the percent recoveries within limits?	[ ]	[ ]
Were the RPDs within control limits?		
• System Monitoring Compounds (Surrogates): Were surrogate recoveries within QC limits?	[ ]	[ ]

9. Comments (attach additional sheets if necessary):

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Validated/Reviewed by:

Signature: _____ Date: _____

Name: _____

POLYAROMATIC HYDROCARBONS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time: Were samples analyzed within holding time?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of five standards?	[ ]	[ ]
Did the RSD meet the criteria ≤ 20% for each individual Calibration Compound or r ≥0.99	[ ]	[ ]
• Was manual integration "M" performed? If the answer is "Yes", check for supporting documents.	[ ]	[ ]
• Was the manual integration necessary?	[ ]	[ ]
If the answer is "no", contact the laboratory inquiring about the reasons behind the manual integration, and **inform the District Chemist immediately if there were no valid reasons.**		
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
4. QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours??	[ ]	[ ]
• Was the QC/MRL between 70-130% R	[ ]	[ ]
5. Initial Calibration Verification (ICV):		
• Was the ICV made of a 2nd source?	[ ]	[ ]

	Yes	No
• Was the mid level (2nd source) recovery within 85 - 115%?	[ ]	[ ]
6. Continuing Calibration Verification (CCV): {Daily calibration}		
• Was midpoint calibration standard conducted at the beginning of the day?	[ ]	[ ]
• Was midpoint calibration standard analyzed every ten samples or every twelve hours?	[ ]	[ ]
• Was midpoint calibration standard conducted after the last sample of the day?	[ ]	[ ]
• Did the CCV meet the minimum requirements (D ≤ 15% with a maximum D ≤ 20% for a specific compound if the mean D ≤ 15%)?	[ ]	[ ]
7. Sample Analysis:		
• Was the RRT of an identified component within the retention time window created as SW-846 requires?	[ ]	[ ]
• Were all identified hits, above the initial calibration curve, diluted and reanalyzed?	[ ]	[ ]
• Were all identified hits confirmed on a second column?	[ ]	[ ]
8. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS/MSD: Were the percent recoveries within limits?	[ ]	[ ]
Were the RPDs within control limits?	[ ]	[ ]
• System Monitoring Compounds (Surrogates): Were surrogate recoveries within QC limits?	[ ]	[ ]

9. Comments (attach additional sheets if necessary):

_____

_____

_____

	Yes	No

Validated/Reviewed by:

Signature: _____ Date: _____

Name: _____

ICP METALS ANALYSIS (6010) CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time:		
• Were samples analyzed within holding time (6-Months)?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of		
One calibration standard and a blank?	[ ]	[ ]
three calibration standards and a blank?	[ ]	[ ]
• Was R ≥ 0.995	[ ]	[ ]
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours??	[ ]	[ ]
• Was the QC/MRL between 70-130% R?	[ ]	[ ]
Common Elements can be between the MRL and 2X MRL level (Fe, Al, Mg and Ca)	[ ]	[ ]
4. Initial Calibration Verification (ICV):		
• Is the mid level (2nd source) recovery within 90 - 110%?		
5. Initial Calibration Blank (ICP):		

	Yes	No
• Were analytes in the blank ≤ 1/2 MRL?	[ ]	[ ]

6. Interelement Check Standard:

• Was ICS-A (interferents only) conducted at the beginning of analytical sequence?	[ ]	[ ]
• Was ICS-AB results within QC limits (80-120)?	[ ]	[ ]

7. Continuing calibration Blank (CCB):

• Was CCB conducted every 10 samples?	[ ]	[ ]
• Was CCB conducted at end of the analytical sequence?	[ ]	[ ]
• Were analytes ≤ 1/2 MRL?	[ ]	[ ]

8. Continuing Calibration Verification (CCV):

• Was CCV conducted every 10 samples?	[ ]	[ ]
• Was CCV conducted at end of the analytical sequence?	[ ]	[ ]
• Was the %R between 90-110?	[ ]	[ ]

9. Sample Analysis:

• Were samples with levels higher than the calibration range (E), diluted and re-analyzed?	[ ]	[ ]

10. Sample Quality Control:

• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS: Were the percent recoveries within limits?	[ ]	[ ]
• MD: Were the RPDs within control limits?	[ ]	[ ]

11. Serial Dilution:

• Was serial dilution (1:4) conducted when needed?	[ ]	[ ]

	Yes	No
• Was there an agreement between diluted and undiluted results (<10%)?	[ ]	[ ]

12. Method of Standard Addition (MSA):

• Was MSA performed on samples suspected of matrix effect (R ≥ 0.995)?	[ ]	[ ]

13. Comments (attach additional sheets if necessary):

Validated/Reviewed by:

Signature: _____ Date: _____

Name: _____

GFAA METALS ANALYSIS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time:		
• Were samples analyzed within holding time?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of		
One calibration standard and a blank?	[ ]	[ ]
three calibration standards and a blank?	[ ]	[ ]
• Was R ≥ 0.995	[ ]	[ ]
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
4. QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours?	[ ]	[ ]
• Was the QC/MRL between 70-130% R?	[ ]	[ ]
5. Initial Calibration Verification (ICV):		
• Is the mid level (2nd source) recovery within 80-120%?	[ ]	[ ]
6. Initial calibration Blank (ICP):		
• Were analytes in the blank ≤ 1/2 MRL?	[ ]	[ ]

	Yes	No
7. Continuing calibration Blank (CCB):		
• Was CCB conducted every 10 samples?	[ ]	[ ]
• Was CCB conducted at end of the analytical sequence?	[ ]	[ ]
• Were analytes ≤ 1/2 MRL?	[ ]	[ ]
8. Continuing Calibration Verification (CCV):		
• Was CCV conducted every 10 samples?	[ ]	[ ]
• Was CCV conducted at end of the analytical sequence?	[ ]	[ ]
• Was the %R between 80-120?	[ ]	[ ]
9. Sample Analysis:		
• Were samples with levels higher than the calibration range (E), diluted and re-analyzed?	[ ]	[ ]
10. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS: Were the percent recoveries within limits?	[ ]	[ ]
• MD: Were the RPDs within control limits?	[ ]	[ ]
11. Serial Dilution:		
• Was serial dilution (1:4) conducted when needed?	[ ]	[ ]
• Was there an agreement between diluted and undiluted results (<10%)?	[ ]	[ ]

12. Comments (attach additional sheets if necessary):

Validated/Reviewed by:

Signature: Date:

Name:

CVAA METALS ANALYSIS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time:		
• Were samples analyzed within holding time?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of		
One calibration standard and a blank?	[ ]	[ ]
five calibration standards and a blank?	[ ]	[ ]
• Was R ≥ 0.995	[ ]	[ ]
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
4. QCMRL:		
• Were QC/MRL run at the beginning and end of every daily sequence or every 12 hours?	[ ]	[ ]
• Was the QC/MRL between 70-130% R?	[ ]	[ ]
5. Initial Calibration Verification (ICV):		
• Is the mid level (2nd source) recovery within 80-120%?	[ ]	[ ]
6. Initial calibration Blank (ICP):		
• Were analytes in the blank ≤ 1/2 MRL?	[ ]	[ ]

	Yes	No
7. Continuing calibration Blank (CCB):		
• Was CCB conducted every 10 samples?	[ ]	[ ]
• Was CCB conducted at end of the analytical sequence?	[ ]	[ ]
• Were analytes ≤ 1/2 MRL?	[ ]	[ ]
8. Continuing Calibration Verification (CCV):		
• Was CCV conducted every 10 samples?	[ ]	[ ]
• Was CCV conducted at end of the analytical sequence?	[ ]	[ ]
• Was the %R between 80-120?	[ ]	[ ]
9. Sample Analysis:		
• Were samples with levels higher than the calibration range (E), diluted and re-analyzed?	[ ]	[ ]
10. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS: Were the percent recoveries within limits?	[ ]	[ ]
• MD: Were the RPDs within control limits?	[ ]	[ ]

11. Comments (attach additional sheets if necessary):

Validated/Reviewed by:

Signature: Date:

Name:

CYANIDE ANALYSIS CHECKLIST

Project Name: _____

Laboratory: _____

Batch Number(s): _____

Sample Delivery Group: _____

	Yes	No
1. Holding Time:		
• Were samples analyzed within holding time?	[ ]	[ ]
2. Initial Calibration:		
• Did the initial calibration consist of		
One calibration standard and a blank?	[ ]	[ ]
Six calibration standards and a blank?	[ ]	[ ]
• Was R ≥ 0.995	[ ]	[ ]
3. QCMDL:		
• Was MDL Check performed?	[ ]	[ ]
4. QCMRL:		
• Were QC/MRL run at the beginning of every daily sequence??	[ ]	[ ]
• Was the QC/MRL between 70-130% R?	[ ]	[ ]
5. Initial Calibration Verification (ICV):		
• Is the mid level (2nd source) recovery within 80-120%?	[ ]	[ ]
7. Initial calibration Blank (ICP):		
• Were analytes in the blank ≤ 1/2 MRL?	[ ]	[ ]

	Yes	No
7. Continuing calibration Blank (CCB):	[ ]	[ ]
• Was CCB conducted every 10 samples?	[ ]	[ ]
• Was CCB conducted at end of the analytical sequence?	[ ]	[ ]
• Were analytes ≤ 1/2 MRL?		
8. Continuing Calibration Verification (CCV):		
• Was CCV conducted every 10 samples?	[ ]	[ ]
• Was CCV conducted at end of the analytical sequence?	[ ]	[ ]
• Was the %R between 80-120?	[ ]	[ ]
9. Sample Analysis:		
• Were samples with levels higher than the calibration range (E), diluted and re-analyzed?	[ ]	[ ]
12. Sample Quality Control:		
• Method Blanks: Were target analytes ≤ 1/2 MRL?	[ ]	[ ]
• LCS: Were the percent recoveries for LCS within the limits?	[ ]	[ ]
• MS: Were the percent recoveries within limits?	[ ]	[ ]
• MD: Were the RPDs within control limits?	[ ]	[ ]

13. Comments (attach additional sheets if necessary):

	Yes	No

Validated/Reviewed by:

Signature: _____ Date: _____

Name: _____

SECTION VI
CHEMICAL DATA QUALITY ASSESSMENT REPORT (CDQAR)

The text of CDQAR was entered from US Army Corps Manual, Chemical Quality Assurance for HTRW Projects, EM 200-1-6, 10 October 1997.

Chapter 5.0 CHEMICAL DATA QUALITY ASSESSMENT REPORTS

5.1 Introduction

In this chapter, the requirements for CDQARs are defined. Each district is responsible for evaluation of chemical data quality, including determination of contract compliance, data usability and data quality objective attainment. The district's data evaluation is documented in the CDQAR. Districts with insufficient staff chemist resources to prepare CDQARs should rely upon the HTRW design district, the CMQAL, or the HTRW-CX for chemistry support. CDQARs should be prepared by the district project chemist for both contractor-executed and in-house projects.

5.2 Evaluation of Data Quality

The district project chemist has three general benchmarks for evaluation of project data: usability, DQOS, and contract compliance. The district project chemist must first determine if data are usable. Data usability is assessed using some form of data review followed by evaluation of other factors; general data review procedures are described in Chapter 3 of this EM. The district project chemist must also determine if project DQOs have been met. DQOs are summarized in the SAP; the chemist should review this summary and compare it to the project data to determine if DQOs were attained. Contract compliance should also be assessed by the district project chemist, to ensure that stated requirements for data quality have been met. The district project chemist should draw on 0 applicable sources of information to conduct the data evaluation. Good supporting documents might include the daily quality report, the contractor's assessment of data quality, results from PE samples, field oversight findings, and/or project-specific laboratory audits.

5.3 Documentation of Data Quality

The district project chemist documents chemical data quality determinations in a CDQAR.

a. Preparation of CDQAR. The CDQAR may be prepared in a variety of formats. The format for documentation of data quality shall be determined by the district project chemist on a project- specific basis. This allows the district project chemist flexibility to choose the most appropriate format for each HTRW project. Common formats include:

- a memorandum for record
- a separate report to the data users
- a memorandum to data user and/or PM and/or TM and/or customer
- an integral section of project report (prepared by or reviewed and approved by district project chemist
- an appendix to the project report (prepared by or reviewed and approved by district project chemist

b. Documentation. Documentation will typically include the following elements, as applicable:

- description of project background and purpose - summary of DQOs
- summary of sampling activities
- description of deficiencies in sampling, packaging, transportation, storage, or analysis
- restrictions on use of data
- statement of contract compliance or noncompliance
- data adequacy (including sensitivity requirements)
- lessons learned
- corrective actions taken

c. Documentation by Reference. If the above information is included in other documents, it may be incorporated by reference.

d. Assessment. The assessment of data usability and attainment of DQOs must be completed concurrent with or prior to completion of the draft project report.

Chemical Data Quality Assessment Report

(CDQAR)

Project:
Background:
Data Quality Objectives (DQO):
Date Samples Collected :
Method of Analysis:
Key Findings in QCSR

Key Findings in Data Validation Report:		
Key Findings in CQAR:		
Usability: Is data adequate for its intended use? Is there any restriction on use of data? If Yes, indicate the restrictions:	 Yes [ ] Yes [ ]	 No [ ] No [ ]
Contractual Compliance: Were data quality requirements met? If No, indicate the deficiencies:	 Yes [ ]	 No [ ]
Signature: District Chemist/Name Date:		

Appendix A
Independent 3rd Party Validation of Laboratory Sample Data Results Memorandum

DEPARTMENT OF THE ARMY
U. S. ARMY ENGINEER DISTRICT, LOUISVILLE
CORPS OF ENGINEERS
P. O. BOX 59
LOUISVILLE, KENTUCKY 40201-0059

31 October 2000

MEMORANDUM FOR: CELRL-ED-E-B

MEMORANDUM FROM: CELRL-OC

SUBJECT: Independent 3rd Party Validation of Laboratory Sample Data Results

1. The issue that has been presented concerns how to satisfy the requirement for independent third party validation of laboratory sample data results. This issue is independent of the quality control/quality assurance processes that have established criteria in place.

2. The main issue that is involved is conflict of interest either real or perceived. Both should be avoided at all times for several reasons. There are ethical issues and standards of professionalism, there are technical issues in requiring valid information upon which decisions can be based and there are legal issues in fulfilling our responsibilities under CERCLA or other environmental laws. If this process can be successfully challenged at any level it could require the process to be repeated costing time and money and depending on when this occurs in the process the cost could be significant.

3. Therefore, in the procurement process care should be exercised as to the relation between the sampling laboratory and the Data Validator. The party contracting with the Data Validator should have no vested interest in the outcome of the process. Therefore, the sampling laboratory should not subcontract out the data validation. Beyond that, one has to look at the relationships and make some common sense determinations. For example the Corps may have separate actions for sampling laboratory and a Data Validator. Depending on the circumstances, the TERC, as a cost reimbursable contractor with no vested interest in the outcome, may be able hire both the sampling laboratory and the Data Validator. In fixed price contracts one should exercise extreme care. A fixed price AE contractor who subcontracts separately with a sampling laboratory and a Data Validator can be financially impacted by the negative results of the Data Validator.

4. This concept extends not only to the contractual relations but also to the technical relationships. The sampling laboratory should not be communicating directly with the Data Validator concerning the process or results. The determination of the validation process and which sampling results are to be used in the validation process should be independent of the sampling laboratory.
5. Those involved in the process may be aware of additional circumstances where potential areas of conflict of interest arise in this area. The general guidance provided

above is just that – general. Individuals need to recognize the potential conflicts and use caution and common sense. This office is available to assist in evaluation and resolution of any concerns in this area.

Kevin M. Finley
Assistant District Counsel

Appendix B
CQAR Report and Forms

U.S. Army Corps of Engineers
Louisville District
Louisville, KY 40202

Subject: Chemical Quality Assurance Report

Project:

Submitted by:

POC: **Telephone Number:**

Date Sampled:

Method of Test or Specification:

REMARKS

The contractor for this project was_____ and the QC laboratory for primary and duplicate sample analysis was _____, located at _____. The QA laboratory was_____, located at_____ .

I. QC DATA EVALUATION:

A. Accuracy:

1) Volatiles Organic Analyses

Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

Subject: Chemical Quality Assurance Report
Project:

REMARKS

I. QC DATA EVALUATION (Continued):

2) Semivolatile Organic Analysis:

Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

3) PCB's Analysis:

Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

4) Explosive Residue Analysis:

Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

5) Propellants Analysis:

6) Metals (Total and Filtered) Analysis:

Discuss results of pre-, and post-digested spikes recoveries. Discuss if there is matrix interference on the results.

7) Cyanide Analysis:

Subject: Chemical Quality Assurance Report
Project:

REMARKS

I. QC DATA EVALUATION (Continued):

B. Precision

1) **Volatiles Organic Analyses**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

2) **Semivolatile Organic Analysis:**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

3) **PCB's Analysis:**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

4) **Explosive Residue Analysis:**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

5) **Propellants Analysis:**

6) **Metals (Total and Filtered) Analysis:**
Compare results of duplicate analysis, and indicate if calculated RPD are within the QC limits.

7) **Cyanide Analysis:**

Subject: Chemical Quality Assurance Report
Project:

REMARKS

I. QC DATA EVALUATION (Continued):

C. Laboratory Contamination

Discuss laboratory contaminants that appear in both samples and method blanks. Determine if the presence of the analyte in the sample is due to laboratory contamination or actually it is in the sample.

D. Holding Times

Indicate if all samples were extracted/digested within holding time, and were analyzed within the holding time. Summarize samples that were extracted/digested, and/or analyzed out of the holding time in a table at the end of the text.

II. QA DATA EVALUATION:

A. Accuracy:

1) Volatile Organic Analysis
Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

2) Semivolatile Organic Analysis:
Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

3) PCB's Analysis
Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

Subject: Chemical Quality Assurance Report
Project:

REMARKS

II. QA DATA EVALUATION (Continued):

4) **Explosive Residue Analysis:**
Discuss results of surrogate recoveries in relation to the QC limits. Also, plot Control Charts for the surrogate recoveries; and include them in an appendix. Discuss the recoveries of the surrogate within ±1 or 2 SD.

5) **Propellants Analysis:**

6) **Metals (Total and Filtered) Analysis:**
Discuss results of pre-, and post-digested spikes recoveries. Discuss if there is a matrix interference on the results.

7) **Cyanide Analysis:**

B. <u>Precision</u>

1) **Volatiles Organic Analyses**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

2) **Semivolatiles Organic Analysis:**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

3) **PCB's Analysis:**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

4) **Explosive Residues Analysis:**
Compare results of duplicate, and/or MS/MSD, and indicate if the calculated RPD are within the QC limits.

Subject: Chemical Quality Assurance Report
Project:

REMARKS

II. QA DATA EVALUATION (Continued):

5) **Propellants Analysis:**

6) **Metals (Total and Filtered) Analysis:**
Compare results of duplicate analysis, and indicate if calculated RPD are within the QC limits.

7) **Cyanide Analysis:**

C. Laboratory Contamination

Discuss laboratory contaminants that appear in both samples and method blanks. Determine if the presence of the analyte in the sample is due to laboratory contamination or actually it is in the sample.

D. Holding Times

Indicate if all samples were extracted/digested within holding time, and were analyzed within the holding time. Summarize samples that were extracted/digested, and/or analyzed out of the holding time in a table at the end of the text.

Subject: Chemical Quality Assurance Report
Project:

REMARKS

III. QA/QC DATA COMPARISON

1) **Volatiles Organic Analysis:**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

2) **Semivolatile Organic Analysis:**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

3) **PCB's Analysis:**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

4) **Explosive Residues Analysis**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

5) **Propellants Analysis:**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

Subject: Chemical Quality Assurance Report
Project:

REMARKS

III. QA/QC DATA COMPARISON (Continued):

6) **Metals Analysis:**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

7) **Cyanide Analysis:**
Compare results between the primary, duplicate and QA sample analysis for the positive hits. Determine if there are agreements, or minor, major disagreements between the results. If there are discrepancies between the results, investigate the cause of such disagreement. Use table 4-1, Criteria for Comparing Field QC and QA Sample Data.

IV. PROBLEMS

General Problems

V. Recommendation

VI. Conclusion:

Subject: Chemical Quality Assurance Report
Project:

REMARKS

VII. Summary

All analytical data were conducted based on SW-846 methodology except for the Wet Chemistry analytes were done by EPA 300 series methods. All Initial Calibration Verification (ICV), and Continuing Calibration Verification (CCV) were met per SW-846 requirements, and according to LCG dated _____.

Submitted By:_____

Prepared By:_____

Signature:_____ Date:_____

TABLES

OUT OF HOLDING TIME SUMMARY

Project: **Matrix:**

Laboratory Name:

Analysis/Sample Number	Date Collected	Date Received	Date Digested/ Extracted	Date Analyzed	Holding Timer
VOC/EPA8260A:					Not Acceptable
SVOC/EPA8270C:					
PCB/EPA8082:					
Pesticides/EPA8081A:					
Explosive Residues/EPA8330:					
Metal/EPA6010:					

DATA COMPARISON SUMMARY

DATA COMPARISON SUMMARY

Project:

List in the following table the results of positive hits in primary, duplicate, or QA sample, and their corresponding values. See example of methylene chloride under VOC in the table below.

Matrix: Water **Units:** µg/L

Analysis/Sample Number	Analyte	Results Primary Samples	Q	Results Duplicate Samples	Q	Results QA Samples	Q
VOC, EPA8260A: Sample #xxxxxx	Vinyl Chloride	1.9		1.3		< 1.0	U
SVOC, EPA8270C:							
PCB, EPA8082:							
PESTICIDES, EPA8081A:							
EXPLOSIVE RESIDUES, EPA8330:							
METAL/EPA6010B							

DATA COMPARISON TABLES

U.S. Army Corps of Engineers
1A
VOLATILE ORGANIC ANALYSIS DATA SHEET
QA/QC COMPARISON TABLE

Customer Sample ID

Project: _____

QC Lab Name: QA Lab Name:

QC-SAMPLE NO.: _____ QA-SAMPLE NO.: _____

QC-DUP NO.: _____

DATE RECEIVED: _____ DATE RECEIVED: _____

Date Sampled: _____
Date Analyzed: _____ Date Analyzed: _____
Method No.: Method No.:
Dilution factor: _____ Dilution Factor: _____

% Solids: % Solids:

Matrix: CONCENTRATION UNIT:

CAS NO.	COMPOUND	QC	Q	QC DUP	Q	QA	Q
67-64-1	Acetone	<				<	
71-43-2	Benzene	<				<	
108-86-1	Bromobenzene						
74-97-5	Bromochloromethane	<				<	
75-27-4	Bromodichloromethane	<				<	
75-25-2	Bromoform	<				<	
74-83-9	Bromomethane	<					
78-93-3	2-Butanone	<				<	
104-51-8	n-Butylbenzene	<				<	
135-98-8	sec-Butylbenzene	<				<	
98-06-6	tert-Butylbenzene	<				<	
75-15-0	Carbon disulfide	<				<	
56-23-5	Carbon tetrachloride	<				<	
108-90-7	Chlorobenzene	<					
124-48-1	Chlorodibromomethane	<				<	
75-00-3	Chloroethane	<				<	
67-66-3	Chloroform	<				<	
74-87-3	Chloromethane	<				<	
95-49-8	2-Chlorotoluene	<				<	
106-43-4	4-Chlorotoluene	<				<	
96-12-8	1,2-Dibromo-3-Chloropropane	<				<	
74-95-3	Dibromomethane						
106-93-4	1,2-Dibromoethane	<				<	
95-50-1	1,2-Dichlorobenzene	<				<	
541-73-1	1,3-Dichlorobenzene	<				<	
106-46-7	1,4-Dichlorobenzene	<				<	
75-71-8	Dichlorodifluoromethane	<				<	
75-34-3	1,1-Dichloroethane	<				<	
107-06-2	1,2-Dichloroethane	<				<	
75--35-4	1,1-Dichloroethene	<				<	
540-59-0	1,2-Dichloroethene (total)	<				<	
156-59-2	cis-1,2-Dichloroethene	<				<	
156-60-5	trans-1,2-Dichlorethene	<				<	
78-87-5	1,2-Dichloropropane	<				<	

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U.S. Army Corps of Engineers
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Jan-00
Customer Sample ID

VOLATILE ORGANIC ANALYSIS DATA SHEET
QA/QC COMPARISON TABLE

Project: _____ Date Sampled: _____

QC-SAMPLE No.: _____ QA-SAMPLE NO.: _____
QC-DUP No.: _____

Matrix: CONCENTRATION UNIT:

CAS NO.	COMPOUND	QC	Q	QC DUP	Q	QA	Q
142-28-9	1,3-Dichloropropane						
594-20-7	2,2-Dichloropropane						
563-58-6	1,1-Dichloropropene						
10061-01-5	cis-1,3-Dichloropropene	<				<	
10061-02-6	trans-1,3Dichloropropene	<				<	
100-41-4	Ethylbenzene						
87-68-3	Hexachlorobutadiene	<				<	
591-78-6	2-Hexanone	<				<	
98-82-8	Isopropylbenzene	<				<	
99-87-6	4-Isopropyltoluene	<					
75-09-2	Methylene chloride	<				<	
108-10-1	4-Methyl-2-pentanone	<				<	
91-20-3	Naphthalene	<				<	
103-65-1	n-Propylbenzene						
100-42-5	Styrene	<				<	
630-20-6	1,1,1,2-Tetrachloroethane	<				<	
79-34-5	1,1,2,2-Tetrachloroethane	<				<	
127-18-4	Tetrachloroethene	<					
108-88-3	Toluene	<				<	
87-61-6	1,2,3-Trichlorobenzene						
120-82-1	1,2,4-Trichlorobenzene	<				<	
71-55-6	1,1,1-Trichloroethane	<				<	
79-00-5	1,1,2-Trichloroethane	<				<	
79-01-6	Trichloroethene	<				<	
75-69-4	Trichlorofluoromethane	<				<	
96-18-4	1,2,3-Trichloropropane	<				<	
95-63-6	1,2,4-Trimethylbenzene	<				<	
108-67-8	1,3,5-Trimethylbenzene	<				<	
108-05-4	Vinyl acetate	<				<	
75-01-4	Vinyl chloride	<				<	
95-47-6	o-Xylene	<				<	
108-38-3	m-Xylene	<				<	
106-42-3	p-Xylene	<				<	
1330-20-7	Xylene (total)	<				<	

U.S. Army Corps of Engineers
1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
QA/QC COMPARISON TABLE

Customer Sample ID

Project: _____

QC-Lab Name: QA-Lab Name:

QC-SAMPLE NO: _____ QA-SAMPLE NO.: _____

QC-DUP NO.: _____

DATE RECEIVED: _____ DATE RECEIVED: _____

Date Sampled: _____
Date Extracted: _____ Date Extracted: _____
Date Analyzed: _____ Date Analyzed: _____

Analysis Method No.: Analysis Method No.: _____
Extraction Method No. Extraction Method No.: _____
Dilution Factor: _____ Dilution Factor: _____
% Solids: % Solids: _____

Matrix: CONCENTRATION UNIT:

CAS NO.	COMPOUND	QC	Q	QC DUP	Q	QA	Q
108-95-2	Phenol	<					
111-44-4	bis(2-Chloroethyl)ether	<					
95-57-8	2-Chlorophenol	<					
541-73-1	1,3-Dichlorobenzene	<					
106-46-7	1,4-Dichlorobenzene	<					
95-50-1	1,2-Dichlorobenzene	<					
95-48-7	2-Methylphenol	<					
108-60-1	2,2'-oxybis(1-Chloropropane)	<					
106-44-5	4-Methylphenol	<					
621-64-7	N-Nitroso-di-n-propylamine	<					
67-72-1	Hexachloroethane	<					
98-95-3	Nitrobenzene	<					
78-59-1	Isophorone	<					
88-75-5	2-Nitrophenol	<					
105-67-9	2,4-Dimethylphenol	<					
111-91-1	bis(2-Chloroethoxy)methane	<					
120-83-2	2,4-Dichlorophenol	<					
120-82-1	1,2,4-Trichlorobenzene	<					
91-20-3	Naphthalene						
106-47-8	4-Chloroaniline	<					
87-68-3	Hexachlorobutadiene	<					
59-50-7	4-Chloro-3-methylphenol	<					
91-57-6	2-Methylnaphthalene						
77-47-4	Hexachlorocyclopentadiene	<					
88-06-2	2,4,5Trichlorophenol	<					
95-95-4	2,4,6-Trichlorophenol	<					
91-58-7	2-Chloronaphthalene	<					
88-74-4	2-Nitroaniline	<					
131-11-3	Dimethylphthalate	<					
208-96-8	Acenaphthylene						
606-20-2	2,6-Dinitrotoluene	<					
99-09-2	3-Nitroaniline	<					

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U.S. Army Corps of Engineers
1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
QA/QC COMPARISON TABLE

Customer Sample ID

Project: _____ Date Sampled: _____

QC-SAMPLE No.: _____ QA-SAMPLE NO.: _____
QC-DUP No.: _____

% Solids: % Solids:

Matrix: CONCENTRATION UNIT:

CAS NO.	COMPOUND	QC	Q	QC DUP	Q	QA	Q
83-32-9	Acenaphthene	<					
121-14-2	2,4,-Dinitrotoluene	<				<	
84-66-2	Diethylphthalate	<				<	
7005-72-3	4-Chlorophenyl-phenyl ether	<				<	
86-73-7	Fluorene	<				<	
100-01-6	4-Nitroaniline	<				<	
534-52-1	4,6-Dinitro-2-methylphenol	<				<	
86-30-6	N-Nitrosodiphenylamine (1)	<				<	
101-55-3	4-Bromophenyl-phenylether	<				<	
118-74-1	Hexachlorobenzene	<				<	
87-86-5	Pentachlorophenol	<				<	
85-01-8	Phenanthrene					<	
120-12-7	Anthracene	<				<	
86-74-8	Carbazole	<				<	
84-74-2	Di-n-butylphthalate	<				<	
206-44-0	Fluoranthene					<	
129-00-0	Pyrene					<	
85-68-7	Butylbenzylphthalate	<				<	
91-94-1	3,3`-Dichlorobenzidine	<				<	
56-55-3	Benzo(a)anthracene					<	
218-01-9	Chrysene					<	
117-81-7	bis(2-Ethylhexyl)phthalate					<	
117-84-0	Di-n-octylphthalate	<				<	
205-99-2	Benzo(b)fluoranthene					<	
207-08-9	Benzo(k)fuoranthene					<	
50-32-8	Benzo(a)pyrene					<	
193-39-5	Indeno(1,2,3-cd)pyrene					<	
53-70-3	Dibenz(a,h)anthracene					<	
191-24-2	Benzo(g,h,i)perylene					<	

U.S. Army Corps of Engineers
1D
PCB ORGANICS ANALYSIS DATA SHEET
QA/QC COMPARISON TABLE

Customer Ssample ID

Project: _____

QC-Lab Name:

QA-Lab Name:

QC-SAMPLE NO.: _____

QA-SAMPLE NO: _____

QC-DUP NO: _____

DATE RECEIVED: _____

DATE RECEIVED: _____

Date Sampled: _____

Date Extracted: _____

Date Extracted: _____

Date Analyzed: _____

Date Analyzed: _____

Extraction Method No.:
Analysis Method No.:

Extraction Method No.:
Analysis Method No.:

% Solid:

% Solid:

Dilution Factor: _____

Dilution Factor:

Matrix:

CONCENTRATION UNITS:

CAS NO.	COMPOUND	QC		QC DUP	Q	QA	Q
1.	Aroclor-1016	<				<	
2.	Aroclor 1221	<				<	
3.	Aroclor 1232	<				<	
4.	Aroclor 1242	<				<	
5.	Aroclor 1248	<				<	
6.	Aroclor 1254	<				<	
7.	Aroclor 1260	<				<	

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U.S. Army Corps of Engineers
1D
PESTICIDE ORGANICS ANALYSIS DATA SHEET
QA/QC COMPARISON TABLE

Customer Sample ID

Project: _____

QC-Lab Name: QA-Lab Name:

QC-SAMPLE NO.: _____

QC-DUP NO: _____ QA-SAMPLE NO.: _____

DATE RECEIVED: DATE RECEIVED:

Date Sampled: _____

Date Extracted: _____ Date Extracted: _____

Date Analyzed: _____ Date Analyzed: _____

Extraction Method No.: Extraction method No.:
Analysis Mehtod No.: Analysis Mehtod No.:

% Solid: % Solid:

Dilution Factor: _____ Dilution Factor: _____

Matrix: CONCENTRATION UNITS:

CAS NO.	COMPOUND	QC	Q	QC DUP	Q	QA	Q
8.	alpha-BHC	<					
9.	beta-BHC						
10.	gamma-BHC (Lindane)	<					
11.	delta-BHC	<					
12.	Heptachlor	<					
13.	Aldrin	<					
14.	Heptachlor Epoxide	<					
15.	Endosulfan I	<					
16.	Dieldrin						
17.	4,4-'DDE	<					
18.	Endrin	<					
19.	Endosulfan II	<					
20.	4,4'-DDD	<					
21.	Endrin Aldehyde	<					
22.	Endosulfan Sulfate	<					
23.	4,4'-DDT	<					
24.	Endrin Ketone						
25.	4,4'-Methoxychlor						
26.	Chlordane, Technical						
27.	Toxaphene	<					

FORM I CQAR PEST Jan-00

U.S. Army Corps of Engineers
I EXP
NITROAROMATICS ANALYSIS DATA SHEET
CQAR QA/QC COMPARISON TABLE

Customer Sample ID

Project: _____

QC Lab Name: _____ QA Lab Name: _____

Date Sampled: _____ Method No.: SW846-8330

QC-SAMPLE NO: _____ QA-SAMPLE NO: _____

QC-DUP NO: _____

Date Received: _____ Date Received: _____

Date Extracted: _____ Date Extracted: _____

Date Analyzed: _____ Date Analyzed: _____

% Solid: _____ % Solid: _____

Dilution Factor: _____ Dilution Factor: _____

Matrix: _____ CONCENTRATION UNITS: _____

CAS NO.	COMPOUND	QC	Q	QC DUP	Q	QA	Q
99-35-4	1,3,5-TNB						
99-65-0	1,3-DNB						
118-96-7	2,4,6-TNT						
121-14-2	2,4-DNT						
606-20-2	2,6,-DNT						
355-72-78-2	2-Am-4,6-DNT						
88-72-2	2-Nitrotoluene						
99-08-1	3-Nitrotoluene						
1946-51-0	4-Am-2,6-DNT						
99-99-0	4-Nitrotoluene						
269-41-0	HMX						
98-95-3	Nitrobenzene						
121-82-4	RDX						
479-45-8	Tetryl						

FORM I EXPLOSIVES Jan-00

U.S. Army Corps of Engineers
2A
METAL ANALYSIS DATA SHEET
CQAR QA/QC COMPARISON TABLE

Customer Sample ID

Project:

QC-Lab Name:

QA-Lab Name:

QC-DUP No.:

QA-SAMPLE NO.:

Date Sampled:

DATE RECEIVED:

DATE RECEIVED:

% Solid:

% Solid:

Dilution Factor:

Dilution Factor:

Matrix:

Concentration Units:

Analyte	QC-Sample (S)	Q	C	QA-Sample (S)	Q	C	RPD	Q	M QC/QA
Aluminum									
Antimony	<								
Arsenic									
Barium									
Beryllium	<								
Cadmium	<								
Calcium									
Chromium	<								
Cobalt									
Copper	<								
Iron									
Lead									
Magnesium									
Manganese									
Mercury				<					
Nickel	<								
Potassium									
Selenium				<					
Silver	<			<					
Sodium									
Thallium									
Vanadium	<								
Zinc									

COMMENTS:

FORM VI-CQAR METL Jan-00

U.S. Army Corps of Engineers
2A
METAL ANALYSIS DATA SHEET
CQAR QA/QC COMPARISON TABLE

Customer Sample ID

Project:

QC-Lab Name: QA-Lab Name:

QC-DUP No.: QA-SAMPLE NO.:

Date Sampled:

DATE RECEIVED: DATE RECEIVED:

% Solid: % Solid:

Dilution Factor: Dilution Factor:

Matrix: Concentration Units:

Analyte	QC-DUP Sample (S)	Q	C	QA-Sample (S)	Q	C	RPD	Q	M QC/QA
Aluminum									
Antimony	<								
Arsenic									
Barium									
Beryllium	<								
Cadmium	<								
Calcium									
Chromium	<								
Cobalt									
Copper	<								
Iron									
Lead									
Magnesium									
Manganese									
Mercury				<					
Nickel	<								
Potassium									
Selenium				<					
Silver	<			<					
Sodium									
Thallium									
Vanadium	<								
Zinc									

COMMENTS:

FORM VI-CQAR METL Jan-00

Appendix C
LCS Recovery Limits, MDLs, RLs and PRGs for SW-846 Methods

The Risk Assessment values are based on current values as of October 2001. These values may change as updates are made to the PRG tables.

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8260B - VOCs

8260B		WATER (5030B-LOW)						SOIL (5035)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Acetone	1.9	10	ug/L	51	157	30	2.7	20	Ug/Kg	50	150	50
2	Acrolein	4.5	50	ug/L	50	150	30	13	50	Ug/Kg	50	150	50
3	Acrylonitrile	4.6	10	ug/L	50	150	30	2.5	50	Ug/Kg	50	150	50
4	Benzene	0.2	1	ug/L	75	126	30	0.3	5	Ug/Kg	75	126	50
5	Bromobenzene	0.2	1	ug/L	74	123	30	0.4	5	ug/Kg	75	123	50
6	Bromochloromethane	0.1	1	ug/L	75	127	30	0.8	5	ug/Kg	75	127	50
7	Bromodichloromethane	0.1	1	ug/L	70	130	30	0.4	5	ug/Kg	71	130	50
8	Bromoform	0.2	1	ug/L	72	136	30	0.3	5	ug/Kg	62	141	50
9	Bromomethane	0.1	1	ug/L	35	153	30	2.5	5	ug/Kg	57	153	50
10	t-Butanol	4.9	25	ug/L	50	150	30	26	50	ug/Kg	50	150	50
11	2-Butanone(MEK)	1.8	10	ug/L	45	150	30	2.8	20	ug/Kg	50	150	50
12	n-Butylbenzene	0.1	1	ug/L	75	126	30	0.3	5	ug/Kg	74	136	50
13	sec-Butylbenzene	0.2	1	ug/L	75	125	30	0.3	5	ug/Kg	75	125	50
14	tert-Butylbenzene	0.1	1	ug/L	75	125	30	0.3	5	ug/Kg	75	120	50
15	Carbon Disulfide	0.1	1	ug/L	74	123	30	0.3	5	ug/Kg	64	123	50
16	Carbon Tetrachloride	0.2	1	ug/L	71	132	30	0.4	5	ug/Kg	69	132	50
17	Chlorobenzene	0.1	1	ug/L	75	127	30	0.3	5	ug/Kg	75	126	50
19	Chloroethane	0.2	1	ug/L	72	129	30	3.2	10	ug/Kg	62	140	50
20	Chloroform	0.1	1	ug/L	74	127	30	0.4	5	ug/Kg	75	132	50
21	1-Chlorohexane	0.1	1	ug/L	75	132	30	0.3	5	ug/Kg	75	129	50
22	Chloromethane	0.4	1	ug/L	58	135	30	2.5	5	ug/Kg	56	144	50
23	2-Chlorotoluene	0.2	1	ug/L	75	121	30	0.5	5	ug/Kg	73	128	50
24	4-Chlorotoluene	0.2	1	ug/L	73	127	30	0.4	5	ug/Kg	75	127	50
25	DIPE	0.1	2	ug/L	50	150	30	0.7	5	ug/Kg	50	150	50
26	Dibromochloromethane	0.2	1	ug/L	74	145	30	1.1	10	ug/Kg	61	147	50
27	1,2-Dibromo-3-chloropropane	0.2	2	ug/L	75	132	30	0.6	5	ug/Kg	70	128	50
28	1,2-Dibromoethane	0.2	1	ug/L	75	127	30	0.6	5	ug/Kg	62	150	50
29	Dibromomethane	0.2	1	ug/L	76	132	30	0.8	5	ug/Kg	62	132	50
30	1,1-Dichloroethane	0.1	1	ug/L	75	133	30	0.5	5	ug/Kg	80	126	50
31	1,2-Dichloroethane	0.2	1	ug/L	67	132	30	0.9	5	ug/Kg	75	121	50
32	1,2-Dichlorobenzene	0.2	1	ug/L	73	120	30	0.3	5	ug/Kg	75	132	50
33	1,3-Dichlorobenzene	0.2	1	ug/L	75	122	30	0.3	5	ug/Kg	75	122	50
34	1,4-Dichloro-2-butene	0.3	10	ug/L	36	150	30	0.8	10	ug/Kg	50	150	50
35	1,4-Dichlorobenzene	0.1	1	ug/L	74	123	30	0.3	5	ug/Kg	74	126	50
36	Dichlorodifluoromethane	0.4	1	ug/L	59	134	30	2.8	10	ug/Kg	43	134	50
37	1,1-Dichloroethene	0.1	1	ug/L	75	125	30	0.5	5	ug/Kg	70	129	50
38	cis-1,2-Dichloroethene	0.1	1	ug/L	73	133	30	0.3	5	ug/Kg	70	129	50
39	trans-1,2-Dichloroethene	0.2	1	ug/L	75	134	30	0.3	5	ug/Kg	75	123	50
40	1,1-Dichloropropene	0.2	1	ug/L	75	135	30	1.1	5	ug/Kg	75	123	50
41	1,2-Dichloropropane	0.2	1	ug/L	75	127	30	0.4	5	ug/Kg	72	124	50
42	1,3-Dichloropropane	0.2	1	ug/L	75	133	30	0.7	5	ug/Kg	75	128	50

8260B		WATER (5030B-LOW)						SOIL (5035)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
43	2,2-Dichloropropane	0.2	1	ug/L	62	134	30	0.4	5	ug/Kg	75	134	50
44	cis-1,3-Dichloropropene	0.2	1	ug/L	73	132	30	0.3	5	ug/Kg	72	132	50
45	trans-1,3-Dichloropropene	0.2	1	ug/L	74	131	30	0.5	5	ug/Kg	69	131	50
46	ETBE	0.1	2	ug/L	50	150	30	0.8	5	ug/Kg	50	150	50
47	Ethyl Methacrylate	0.2	1	ug/L	50	150	30	0.8	5	ug/Kg	50	150	50
48	Ethylbenzene	0.1	1	ug/L	75	120	30	0.3	5	ug/Kg	75	120	50
49	2-Hexanone	1	10	ug/L	53	139	30	1.9	20	ug/Kg	55	144	50
50	Hexachlorobutadiene	0.2	1	ug/L	75	133	30	0.5	5	ug/Kg	55	133	50
51	Iodomethane	0.1	2	ug/L	50	150	30	1.5	5	ug/Kg	50	150	50
52	Isopropyl Benzene	0.2	1	ug/L	75	126	30	0.5	5	ug/Kg	76	126	50
53	p-Isopropyltoluene	0.2	1	ug/L	75	125	30	0.3	5	ug/Kg	74	130	50
54	Methylene Chloride	0.1	2	ug/L	69	118	30	1.4	10	ug/Kg	66	131	50
55	4-Methyl-2-Pentanone(MIBK)	1	10	ug/L	59	150	30	1.6	20	ug/Kg	50	150	50
56	MTBE	0.1	1	ug/L	59	129	30	1.2	5	ug/Kg	58	146	50
57	Naphthalene	0.3	1	ug/L	65	149	30	3.2	10	ug/Kg	49	160	50
58	n-Propylbenzene	0.2	1	ug/L	75	127	30	0.3	5	ug/Kg	74	136	50
59	Styrene	0.1	1	ug/L	75	130	30	0.5	5	ug/Kg	75	130	50
60	TAME	0.2	1	ug/L	50	150	30	0.9	5	ug/Kg	75	129	50
61	1,1,1,2-Tetrachloroethane	0.2	1	ug/L	75	127	30	0.5	5	ug/Kg	75	127	50
62	1,1,2,2-Tetrachloroethane	0.3	1	ug/L	68	129	30	0.3	5	ug/Kg	68	144	50
63	Tetrachloroethene	0.2	1	ug/L	75	129	30	0.4	5	ug/Kg	75	129	50
64	Toluene	0.1	1	ug/L	75	125	30	0.5	5	ug/Kg	75	125	50
65	1,1,1-Trichloroethane	0.1	1	ug/L	70	127	30	0.5	5	ug/Kg	75	133	50
66	1,1,2-Trichloroethane	0.2	1	ug/L	75	136	30	1.1	5	ug/Kg	71	129	50
67	1,2,3-Trichlorobenzene	0.2	1	ug/L	75	133	30	0.5	5	ug/Kg	64	129	50
68	1,2,4-Trichlorobenzene	0.2	1	ug/L	75	130	30	0.6	5	ug/Kg	69	139	50
69	Trichloroethene	0.2	1	ug/L	67	128	30	0.3	5	ug/Kg	69	128	50
70	Trichlorofluoromethane	0.2	1	ug/L	68	133	30	0.3	5	ug/Kg	61	139	50
71	1,2,3-Trichloropropane	0.2	1	ug/L	65	139	30	0.6	5	ug/Kg	61	145	50
72	1,1,2-Trichloro1,2,2-trifluoroethane	0.2	1	ug/L	50	150	30	1	5	ug/Kg	50	145	50
73	1,2,4-Trimethylbenzene	0.1	1	ug/L	75	123	30	0.3	5	ug/Kg	74	126	50
74	1,3,5-Trimethylbenzene	0.2	1	ug/L	75	121	30	0.4	5	ug/Kg	75	125	50
75	Vinyl Acetate	0.7	2	ug/L	30	150	30	0.4	5	ug/Kg	50	150	50
76	Vinyl Chloride	0.3	1	ug/L	73	134	30	2.3	5	ug/Kg	75	134	50
77	M,p-Xylene	0.2	2	ug/L	75	122	30	0.7	10	ug/Kg	75	124	50
78	o-Xylene	0.1	1	ug/L	75	118	30	0.3	5	ug/Kg	75	125	50

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8270C –SVOCs

8270C		WATER (3520C)						SOIL (3540C)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Acenaphthene	4.7	10	ug/L	31	120	30	109	330	ug/Kg	30	135	50
2	Acenaphthylene	4.9	10	ug/L	37	115	30	121	330	ug/Kg	33	120	50
3	Aniline	13	40	ug/L	30	127	30	160	660	ug/Kg	50	150	50
4	Anthracene	4.8	10	ug/L	45	118	30	83	330	ug/Kg	35	122	50
5	Benzo(a)anthracene	4.2	10	ug/L	43	138	30	83	330	ug/Kg	33	139	50
6	benzo(a)pyrene	4.1	10	ug/L	38	144	30	92	330	ug/Kg	30	144	50
7	Benzo(b)fluoranthene	3.8	10	ug/L	31	146	30	96	330	ug/Kg	30	140	50
8	Benzo(g,h,i)perylene	4.3	10	ug/L	35	129	30	149	330	ug/Kg	30	146	50
9	Benzo(k)fluoranthene	4.8	10	ug/L	40	127	30	167	330	ug/Kg	30	150	50
10	Benzoic Acid	2.5	10	ug/L	30	136	30	140	330	ug/Kg	30	160	50
11	Benzyl Alcohol	5.1	10	ug/L	30	115	30	174	330	ug/Kg	30	117	50
12	bis(2-chloroethoxy)methane	4.7	10	ug/L	30	115	30	128	330	ug/Kg	30	126	50
13	bis(2-chloroethyl)ether	5.1	10	ug/L	30	115	30	135	330	ug/Kg	30	121	50
14	bis(2-chloroisopropyl)ether	5.4	10	ug/L	50	150	30	143	330	ug/Kg	30	131	50
15	bis(2-Ethylhexyl)phthalate	9.9	20	ug/L	30	154	30	116	330	ug/Kg	34	149	50
16	4-Bromophenyl-phenylether	6.8	20	ug/L	43	118	30	132	330	ug/Kg	34	120	50
17	Butylbenzylphthalate	3.9	10	ug/L	37	136	30	88	330	ug/Kg	30	153	50
18	Carbazole	5	10	ug/L	49	126	30	83	330	ug/Kg	30	142	50
19	4-Chloro-3-methylphenol	4.7	10	ug/L	31	121	30	93	330	ug/Kg	30	125	50
20	4-Chloroaniline	4.6	10	ug/L	30	133	30	114	330	ug/Kg	32	121	50
21	2-Chloronaphthalene	4.8	10	ug/L	35	115	30	133	330	ug/Kg	32	115	50
22	2-Chlorophenol	4.6	10	ug/L	30	120	30	151	330	ug/Kg	30	115	50
23	4-Chlorophenyl-phenylether	5.2	10	ug/L	40	115	30	92	330	ug/Kg	33	118	50
24	Chrysene	4	10	ug/L	42	142	30	83	330	ug/Kg	33	142	50
25	Dibenzo(a,h)anthracene	4.7	10	ug/L	38	130	30	163	330	ug/Kg	34	148	50
26	Dibenzofuran	4.8	10	ug/L	40	115	30	109	330	ug/Kg	34	120	50
27	1,2-Dichlorobenzene	4.6	10	ug/L	30	120	30	149	330	ug/Kg	30	115	50
28	1,3-Dichlorobenzene	4.4	10	ug/L	30	120	30	147	330	ug/Kg	30	115	50
29	1,4-Dichlorobenzene	4.5	10	ug/L	30	115	30	161	330	ug/Kg	34	123	50
30	3,3'-Dichlorobenzidine	4.1	10	ug/L	30	160	30	159	660	ug/Kg	31	137	50
31	2,4-Dichlorophenol	4.4	10	ug/L	34	115	30	149	330	ug/Kg	32	121	50
32	Diethylphthalate	5.7	20	ug/L	43	132	30	85	330	ug/Kg	32	126	50
33	2,4-Dimethylphenol	4.4	10	ug/L	31	120	30	122	330	ug/Kg	33	119	50
34	Dimethylphthalate	5.5	20	ug/L	42	116	30	83	330	ug/Kg	36	124	50
35	Di-n-butylphthalate	4.8	10	ug/L	46	123	30	83	330	ug/Kg	36	135	50
36	4,6-Dinitro-2-methylphenol	6.5	20	ug/L	42	144	30	129	660	ug/Kg	30	160	50
37	2,4-Dinitrophenol	5.9	20	ug/L	29	146	30	167	660	ug/Kg	34	160	50
38	2,4-Dinitrotoluene	6.3	20	ug/L	34	151	30	83	330	ug/Kg	34	154	50
39	2-6-Dinitrotoluene	6.3	20	ug/L	43	122	30	83	330	ug/Kg	32	133	50
40	Di-n-octylphthalate	4.6	10	ug/L	36	151	30	85	330	ug/Kg	50	160	50
41	1,2-Diphenylhydrazine	4.5	10	ug/L	50	150	30	83	330	ug/Kg	50	150	50
42	Fluoranthene	5.3	10	ug/L	47	132	30	83	330	ug/Kg	32	122	50

8270C		WATER (3520C)						SOIL (3540C)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
43	Fluorene	4.9	10	ug/L	41	115	30	83	330	ug/Kg	32	127	50
44	Hexachlorobenzene	5.5	20	ug/L	42	123	30	167	330	ug/Kg	32	127	50
45	Hexachlorobutadiene	3.9	10	ug/L	30	120	30	142	330	ug/Kg	30	118	50
46	Hexachlorocyclopentadiene	5	10	ug/L	30	115	30	167	330	ug/Kg	30	123	50
47	Hexachloroethane	4	10	ug/L	30	120	30	170	330	ug/Kg	30	115	50
48	Indeno(1,2,3-cd)pyrene	4	10	ug/L	37	130	30	158	330	ug/Kg	34	147	50
49	Isophorone	4.6	10	ug/L	33	115	30	107	330	ug/Kg	34	115	50
50	2-Methylnaphthalene	4	10	ug/L	32	115	30	137	330	ug/Kg	30	120	50
51	2-Methylphenol	3.5	10	ug/L	30	116	30	118	330	ug/Kg	30	123	50
52	4-Methylphenol	3.9	10	ug/L	31	115	30	116	330	ug/Kg	30	127	50
53	Naphthalene	4.3	10	ug/L	30	119	30	142	330	ug/Kg	30	115	50
54	2-Nitroaniline	5.7	20	ug/L	36	140	30	108	660	ug/Kg	30	148	50
55	3-Nitroaniline	4.8	10	ug/L	30	138	30	92	660	ug/Kg	30	135	50
56	4-Nitroaniline	4.8	10	ug/L	30	140	30	167	330	ug/Kg	30	148	50
57	Nitrobenzene	5.1	10	ug/L	31	115	30	123	330	ug/Kg	30	115	50
58	2-Nitrophenol	4.7	10	ug/L	33	115	30	156	330	ug/Kg	30	126	50
59	4-Nitrophenol	5.1	10	ug/L	30	138	30	167	660	ug/Kg	36	147	50
60	n-Nitrosodimethylamine	4.2	10	ug/L	30	115	30	119	330	ug/Kg	36	115	50
61	n-Nitroso-di-n-propylamine	4.9	10	ug/L	30	132	30	136	330	ug/Kg	35	147	50
62	n-Nitrosodiphenylamine	4.8	10	ug/L	35	124	30	83	330	ug/Kg	50	134	50
63	Pentachlorophenol	6.4	20	ug/L	30	150	30	97	660	ug/Kg	30	160	50
64	Phenanthrene	5.7	20	ug/L	45	117	30	83	330	ug/Kg	35	119	50
65	Phenol	4.1	10	ug/L	30	115	30	126	330	ug/Kg	30	120	50
66	Pyrene	4.7	10	ug/L	35	139	30	83	330	ug/Kg	32	147	50
67	Pyridine	8.6	20	ug/L	50	150	30	167	330	ug/Kg	30	125	50
68	1,2,4-Trichlorobenzene	3.6	10	ug/L	30	120	30	138	330	ug/Kg	30	120	50
69	2,4,5-Trichlorophenol	4.3	10	ug/L	36	135	30	83	330	ug/Kg	34	128	50
70	2,4,6-Trichlorophenol	4.8	10	ug/L	39	115	30	133	330	ug/Kg	35	120	50

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8082 - PCBs

EPA 8082		WATER (3520C)						SOIL (3540C)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	PCB-1016	0.57	1	ug/L	58	141	50	18.16	50	ug/Kg	53	143	50
2	PCB-1221	0.25	1	ug/L	50	150	50	23.85	50	ug/Kg	50	150	50
3	PCB-1232	0.25	1	ug/L	50	150	50	16.65	50	ug/Kg	50	150	50
4	PCB-1242	0.25	1	ug/L	50	150	50	16.65	50	ug/Kg	50	150	50
5	PCB-1248	0.25	1	ug/L	50	150	50	16.65	50	ug/Kg	50	150	50
6	PCB-1254	0.25	1	ug/L	50	150	50	16.65	50	ug/Kg	50	150	50
7	PCB-1260	0.25	1	ug/L	71	143	50	9.83	50	ug/Kg	71	134	50

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8081A – Pesticides

8081A		WATER (3520C)						SOIL (3540C)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Aldrin	0.01148855	0.03	ug/L	53	128	30	0.28	2	ug/Kg	55	133	50
2	alpha-BHC	0.008156	0.03	ug/L	44	137	30	0.21	2	ug/Kg	50	120	50
3	beta-BHC	0.01279133	0.03	ug/L	50	135	30	0.25	2	ug/Kg	50	124	50
4	delta-BHC	0.0117307	0.03	ug/L	58	160	30	0.22	2	ug/Kg	50	146	50
5	gamma-BHC	0.00789013	0.03	ug/L	58	127	30	0.21	2	ug/Kg	50	129	50
6	alpha-Chlordane	0.005	0.03	ug/L	50	122	30	0.21	2	ug/Kg	50	120	50
7	gamma-Chlordane	0.00528226	0.03	ug/L	50	130	30	0.21	2	ug/Kg	50	127	50
8	4,4'-DDD	0.03161711	0.1	ug/L	50	137	30	0.45	4	ug/Kg	50	136	50
9	4,4'-DDE	0.03019408	0.1	ug/L	50	130	30	0.53	4	ug/Kg	50	120	50
10	4,4'-DDT	0.01920071	0.03	ug/L	50	145	30	1.33	4	ug/Kg	55	160	50
11	Dieldrin	0.07654355	0.1	ug/L	50	124	30	0.50	4	ug/Kg	50	126	50
12	Endosulfan I	0.02529045	0.03	ug/L	50	160	30	0.69	2	ug/Kg	50	144	50
13	Endosulfan II	0.01	0.1	ug/L	50	144	30	0.42	4	ug/Kg	50	135	50
14	Endosulfan Sulfate	0.01142581	0.1	ug/L	50	160	30	0.45	4	ug/Kg	50	160	50
15	Endrin	0.01	0.1	ug/L	50	137	30	0.91	4	ug/Kg	50	159	50
16	Endrin Aldehyde	0.01	0.1	ug/L	30	160	30	0.42	4	ug/Kg	39	121	50
17	Endrin Ketone	0.01054346	0.1	ug/L	50	150	30	0.56	4	ug/Kg	50	160	50
18	Heptachlor	0.00787991	0.03	ug/L	48	150	30	0.59	2	ug/Kg	58	147	50
19	Heptachlor Epoxide	0.005	0.1	ug/L	50	127	30	2.08	4	ug/Kg	50	130	50
20	Methoxychlor	0.08333115	1	ug/L	50	160	30	2.93	20	ug/Kg	50	160	50

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8151 Herbicides

8151		WATER											
PARAMETER		MDL	RL	UNIT	LL	UL	RPD	MDL	RL	UNIT	LL	UL	RPD
1	2,4,5-T	0.16	0.4	ug/L	45	45	30	4.997	10	ug/Kg	48	155	50
2	2,4,5-TP(Silvex)	0.181	0.4	ug/L	46	46	30	4.804	10	ug/Kg	46	147	50
3	2,4-D	0.147	0.4	ug/L	33	33	30	5.018	10	ug/Kg	33	138	50
4	2,4-DB	0.217	0.4	ug/L	27	27	30	7.63	20	ug/Kg	52	160	50
5	Dalapon	0.078	0.4	ug/L	160	20	30	3.892	20	ug/Kg	20	160	50
6	Dicamba	0.563	0.8	ug/L	37	37	30	5.466	20	ug/Kg	47	145	50
7	Dichloroprop	0.157	0.4	ug/L	48	48	30	4.826	10	ug/Kg	46	138	50
8	Dinoseb	0.039	0.4	ug/L	20	20	30	1.628	10	ug/Kg	20	125	50
9	MCPA	6.275	200	ug/L				365.289	2000	ug/Kg			
10	MCPP	8.172	200	ug/L				249.456	2000	ug/Kg			

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8330 - Explosives

8330		WATER						SOIL					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	4-Am-2,6-DNT	0.2	1	ug/L	58	159	30	0.066	0.4	mg/kg	50	159	50
2	2-Am-4,6-DNT	0.2	1	ug/L	53	120	30	0.115	0.4	mg/kg	50	120	50
3	1,3-DNB	0.1	1	ug/L	54	120	30	0.634	0.4	mg/kg	50	120	50
4	2,4-DNT	0.1	1	ug/L	58	136	30	0.089	0.4	mg/kg	50	136	50
5	2,6-DNT	0.2	1	ug/L	52	144	30	0.128	0.4	mg/kg	50	144	50
6	HMX	0.2	1	ug/L	45	140	30	0.168	0.4	mg/kg	60	140	50
7	Nitrobenzene	0.1	1	ug/L	49	120	30	0.052	0.4	mg/kg	50	120	50
8	2-Nitrotoluene	0.2	1	ug/L	52	120	30	0.098	0.4	mg/kg	59	120	50
9	3-Nitrotoluene	0.3	1	ug/L	48	136	30	0.108	0.4	mg/kg	50	136	50
10	4-Nitrotoluene	0.2	1	ug/L	46	136	30	0.947	0.4	mg/kg	58	136	50
11	RDX	0.2	1	ug/L	39	120	30	0.126	0.4	mg/kg	59	120	50
12	Tetryl	0.2	1	ug/L	30	120	30	0.91	0.4	mg/kg	37	120	50
13	1,3,5-TNB	0.2	1	ug/L	53	135	30	0.57	0.4	mg/kg	50	135	50
14	2,4,6-TNT	0.1	1	ug/L	37	120	30	0.083	0.4	mg/kg	44	120	50

LCS Recovery Limits, MDLs and RLs for SW-846 Method 8310 – Polyaromatic Hydrocarbons

8310		WATER						SOIL					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Acenaphthene	0.5	2	ug/L	57	125	30	11.711	100	ug/Kg	60	127	50
2	Acenaphthylene	1	4	ug/L	61	125	30	16.668	200	ug/Kg	62	125	50
3	Anthracene	0.031	0.2	ug/L	60	125	30	1.045	10	ug/Kg	66	125	50
4	Benzo(a)anthracene	0.036	0.2	ug/L	73	126	30	1.44	10	ug/Kg	74	133	50
5	Benzo(a)pyrene	0.055	0.2	ug/L	54	128	30	1.665	10	ug/Kg	55	131	50
6	Benzo(b)fluoranthene	0.055	0.4	ug/L	75	125	30	1.665	20	ug/Kg	75	125	50
7	Benzo(g,h,i)perylene	0.088	0.4	ug/L	71	128	30	3.796	20	ug/Kg	73	130	50
8	Benzo(k)fluoranthene	0.042	0.2	ug/L	75	125	30	2.258	10	ug/Kg	75	127	50
9	Chrysene	0.031	0.2	ug/L	75	125	30	1.665	10	ug/Kg	75	125	50
10	Dibenzo(a,h)anthracene	0.068	0.4	ug/L	75	125	30	5.33	20	ug/Kg	75	127	50
11	Fluoranthene	0.058	0.4	ug/L	74	125	30	2.384	20	ug/Kg	74	126	50
12	Fluorene	0.066	0.4	ug/L	73	125	30	1.665	20	ug/Kg	71	125	50
13	Indeno(1,2,3-cd)pyrene	0.052	0.2	ug/L	75	125	30	1.484	10	ug/Kg	73	130	50
14	Naphthalene	0.5	2	ug/L	52	125	30	15.026	100	ug/Kg	67	158	50
15	Phenanthrene	0.05	0.2	ug/L	75	125	30	2.109	10	ug/Kg	75	126	50
16	Pyrene	0.093	0.2	ug/L	62	132	30	4.801	10	ug/Kg	67	135	50

LCS Recovery Limits, MDLs and RLs for SW-846 Method 6010B - Metals

6010B		WATER (3010A)						SOIL (3050B)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Aluminum	50.000	200	ug/L	80	120	20	5	20	mg/Kg	80	120	20
2	Antimony	50.000	100	ug/L	80	120	20	3.9	10	mg/Kg	80	120	20
3	Arsenic	25.000	100	ug/L	80	120	20	2.7	10	mg/Kg	80	120	20
4	Barium	1.000	10	ug/L	80	120	20	0.3	1	mg/Kg	80	120	20
5	Beryllium	1.000	10	ug/L	80	120	20	0.3	1	mg/Kg	80	120	20
6	Boron	10.000	10	ug/L	80	120	20	2.3	10	mg/Kg	80	120	20
7	Cadmium	5.000	10	ug/L	80	120	20	0.4	1	mg/Kg	80	120	20
8	Calcium	150.000	1000	ug/L	80	120	20	12	100	mg/Kg	80	120	20
9	Chromium	6.000	20	ug/L	80	120	20	0.8	2	mg/Kg	80	120	20
10	Cobalt	11.000	20	ug/L	80	120	20	1	2	mg/Kg	80	120	20
11	Copper	5.000	20	ug/L	80	120	20	0.8	2	mg/Kg	80	120	20
12	Iron	6.000	1000	ug/L	80	120	20	10	20	mg/Kg	80	120	20
13	Lead	25.000	100	ug/L	80	120	20	2.1	10	mg/Kg	80	120	20
14	Magnesium	50.000	1000	ug/L	80	120	20	16	100	mg/Kg	80	120	20
15	Manganese	3.000	100	ug/L	80	120	20	0.4	1	mg/Kg	80	120	20
16	Molybdenum	6.000	100	ug/L	80	120	20	0.9	5	mg/Kg	80	120	20
17	Nickel	15.000	20	ug/L	80	120	20	0.5	2	mg/Kg	80	120	20
18	Potassium	750.000	5000	ug/L	80	120	20	72	500	mg/Kg	80	120	20
19	Selenium	53.000	1000	ug/L	80	120	20	4.6	10	mg/Kg	80	120	20
20	Silver	10.000	20	ug/L	80	120	20	1.1	2	mg/Kg	80	120	20
21	Sodium	70.000	1000	ug/L	80	120	20	18	100	mg/Kg	80	120	20
22	Strontium	3.000	10	ug/L	80	120	20	0.3	1	mg/Kg	80	120	20
23	Thallium	52.000	100	ug/L	80	120	20	1.9	5	mg/Kg	80	120	20
24	Tin	50.000	100	ug/L	80	120	20	2.9	5	mg/Kg	80	120	20
25	Vanadium	5.000	20	ug/L	80	120	20	0.8	2	mg/Kg	80	120	20
26	Zinc	27.000	100	ug/L	80	120	20	0.3	1	mg/Kg	80	120	20

LCS Recovery Limits, MDLs and RLs for SW-846 Method 6010B Trace – Metals

6010B-TRACE		WATER (3010A)						SOIL (3050B)					
	PARAMETER	MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Arsenic	1.676	10	ug/L	80	120	20	0.15	1	mg/Kg	80	120	20
2	Cadmium	0.500	10	ug/L	80	120	20	0.1	1	mg/Kg	80	120	20
3	Copper	1.000	10	ug/L	80	120	20	0.22	1	mg/Kg	80	120	20
4	Lead	1.299	10	ug/L	80	120	20	0.2	1	mg/Kg	80	120	20
5	Manganese	1.000	10	ug/L	80	120	20	0.15	1	mg/Kg	80	120	20
6	Selenium	2.680	10	ug/L	80	120	20	0.47	1	mg/Kg	80	120	20
7	Thallium	3.707	10	ug/L	80	120	20	0.61	2	mg/Kg	80	120	20
8	Vanadium	2.162	10	ug/L	80	120	20	0.15	1	mg/Kg	80	120	20

LCS Recovery Limits, MDLs and RLs for SW-846 Method 7470/7471 Mercury

7470/7471		WATER (3010A)						SOIL (3050B)					
PARAMETER		MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Mercury	0.05	0.5	mg/L	80	120	20	0.017	0.1	mg/Kg	80	120	20

LCS Recovery Limits, MDLs and RLs for SW-846 Method 9010/9014 Cyanide

9010/9014		WATER (3010A)						SOIL (3050B)					
PARAMETER		MDL	RL	Unit	LL	UL	RPD	MDL	RL	Unit	LL	UL	RPD
1	Cyanide	0.003	0.01	mg/L	80	120	20	0.138	1	mg/Kg	80	120	20

Reporting Limits Above Risk Assessment Levels

Matrix: Soil/Sediment

EPA 8260B **Volatile Organic Analytes/25 ml Volume**		
	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
Bromomethane	0.148	
Chloromethane	0.0785	
Ethylbenzene	0.1	
1,1-Dichloroethane	0.575	
1,3,5-Trimethylbenzene	0.121	
EPA 8270C **Semivolatile Organic Analytes:**		
Acenaphthene	6.7	
Acenaphthylene	5.9	
Benzo(a)anthracene	31.7	
Benzo(a)pyrene	31.9	
Benzo(g,h,i)perylene	170	
Benzo(k)fluoranthene	240	
Bis(2-chloroethyl)ether		0.21
Chrysene	57.1	
Dibenzo(a,h)anthracene	6.22	
1,2-Dichlorobenzene	231.32	
3,3'-Dichlorobenzidine	28.22	
2.4-Dichlorophenol	133.63	
Diethylphthalate	8.04	
2,4-Dimethylphenol	304.5	
Dimethylphthalate	24.95	
di-n-butylphthalate	110.5	
Methylphenol	0.8	
2,4-Dinitrophenol	1.33	
2,4-Dinitrotoluene	75.13	
2,6-Dinitrotoluene	20.62	
Fluoranthene	111.3	
Fluorene	19	
Hexachlorobenzene	20	0.3
Indeno(1,2,3-cd)pyrene	200	
2-methylnaphthalene	20.2	
2-Methylphenol	0.826	
4-methylphenol	0.808	
Naphthalene	34.6	
2-Nitroaniline	0.222	

Reporting Limits Above Risk Assessment Levels

Matrix: Soil/Sediment

EPA 8270C Continued Semivolatile Organic Analytes:	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
3-Nitroanaline	0.222	
4-Nitroaniline	0.222	
2-Nitrophenol	7.77	
4-Nitrophenol	7.78	
n-Nitrosodimehtylamine		0.0095
Phenanthrene	41.9	
Phenol	27.3	
Pyrene	53	
2,4,5-Trichlorophenol	85.6	
2,4,6-trichlorophenol	84.8	
EPA 8082 PCBS	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
PCB-1016		
PCB-1221		
PCB-1232		
PCB-1242		
PCB-1248		
PCB-1254		
PCB-1260		
EPA 8081A Pesticides		
Gamma-BHC	0.94	
Alpha-chlordane	0.03	
Gamma-Chlordane	0.03	
4,4'-DDD	0.004	
4,4'-DDE	0.004	
4,4'-DDT	0.004	
Dieldrin	0.01	0.003
Endosulfan I	0.17	
Endosulfan II	0.1	
Endrin	0.014	
Heptachlor	0.26	
Heptachlor Epoxide	0.008	
Methoxychlor	3.5	

EPA 8151 Herbicides	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
2,4,5-T		
2,4,5-TP(Silvex)		
2,4-D		
2,4-DB		
Dalapon		
Dicamba		26000
Dichloroprop		
Dinoseb		
MCPA		
MCPP		
EPA 8330 Explosives	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
4-Am-2,6-DNT		
2-Am-4,6-DNT		
1,3-DNB		
2,4-DNT		
2,6-DNT		
HMX		
Nitrobenzene		
2-Nitrotoluene		
3-Nitrotoluene		
4-Nitrotoluene		
RDX		
Tetryl		
1,3,5-TNB		
2,4,6-TNT		
EPA 8310 Polyaromatic Hydrocarbons	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
Acenaphthene		
Acenaphthylene		
Anthracene		
Benzo(a)anthracene		
Benzo(a)pyrene		
Benzo(b)fluoranthene		
Benzo(g,h,i)perylene		
Benzo(k)fluoranthene		
Chrysene		
Dibenzo(a,h)anthracene		
Fluoranthene		
Fluorene		
Indeno(1,2,3-cd)pyrene		
Naphthalene		

EPA 8310 Polyaromatic Hydrocarbons	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
Phenanthrene		
Pyrene		
EPA 6010B Metal Analytes	Target Ecological PRG (mg/kg)	Target Human Health PRG (mg/kg)
Aluminum		
Antimony	2	
Arsenic	5.9	0.39
Barium		
Beryllium		
Boron		
Cadmium	0.5	
Calcium		
Chromium (VI)	0.4	
Cobalt		
Copper		
Iron		
Lead		
Magnesium		
Manganese		
Molybdenum		
Nickel		
Potassium		
Selenium	0.4	
Silver	0.09	
Sodium		
Strontium		
Thallium	0.4	
Tin		
Vanadium		
Zinc		
EPA 6010B (Trace Analysis) Metal Analytes	Target Ecological PRG (µg/kg)	Target Human Health PRG (mg/kg)
Arsenic		0.39
Cadmium	0.5	
Chromium VI	0.4	
Selenium	0.4	
Thallium	0.4	
EPA 7471 Mercury		
Mercury		
EPA 9010/9014 Cyanide		
Cyanide	.001	

PRGS and Method Reporting Limits – Method 8260B – VOCs
Soil and Sediment

EPA 8260									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(ug/kg)	**(ug/kg)**	**Yes/No**	**(ug/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Acetone	453.4	20	Yes			1600	Yes		
Acrolein		50				0.1	Yes		
Acrylonitrile		50				0.21	Yes		
Benzene	141.5	5	Yes			0.65	Yes		
Bromobenzene		5				28	Yes		
Bromochloromethane		5							
Bromodichloromethane	1.13	5	No	1.09	Yes	1	Yes		
Bromoform		5				62	Yes		
Bromomethane	0.148	5	No	6	No	3.9	Yes		
t-Butanol		50							
2-Butanone (MEK)	136.9	20	Yes						
n-Butylbenzene		5				140	Yes		
sec-Butylbenzene		5				110	Yes		
tert-Butylbenzene		5				130	Yes		
Carbon Disulfide	133.9	5	Yes			360	Yes		
Carbon Tetrachloride	35.7	5	Yes			0.24	Yes		
Chlorobenzene	61.9	5	Yes			150	Yes		
Chloroethane	58600	10	Yes			3	Yes		
Chloroform	27	5	Yes			0.24	Yes		
1-Chlorohexane		5							
Chloromethane	0.0785	5	No	7.5		1.2	Yes		
2-Chlorotoluene		5			No				
4-Chlorotoluene		5							
DIPE		5							
Dibromochloromethane	267.61	10	Yes			1.1	Yes		
chloropropane		5				170	Yes		
1,2-Dibromoethane		5				0.007	Yes		
Dibromomethane		5							
1,1-Dichloroethane	0.575	5	No	1.46	No	590	Yes		
1,2-Dichloroethane	54.18	5	Yes			0.35	Yes		
1,2-Dichlorobenzene	231.3	5	Yes			370	Yes		
1,3-Dichlorobenzene	3010	5	Yes			13	Yes		
1,4-Dichloro-2-betene		10							
1,4-Dichlorobenzene	1450	5	Yes			3.4	Yes		
Dichlorodifluoromethane		10				94	Yes		
1,1-Dichloroethene	23.27	5	Yes			0.054	Yes		
cis-1,2-Dichloroethene		5				43	Yes		
trans-1,2-Dichloroethene		5				63	Yes		
1,1-Dichloropropene		5							
1,2-Dichloropropane	351.6	5	Yes			0.35	Yes		

EPA 8260B									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(ug/kg)	**(ug/kg)**	**Yes/No**	**(ug/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
1,3-Dichloropropane		5							
2,2-Dichloropropane		5							
cis-1,3-Dichloropropene	2.9	5		0.75	Yes	0.7	Yes		
Dichloropropene		5							
ETBE		5							
Ethyl Methacrylate		5				140	Yes		
Ethylbenzene	0.1	5	No	0.98	No	230	Yes		
2-Hexanone	1010	20	Yes						
Hexachlorobutadiene	1380	5	Yes						
Iodomethane		5							
Isopropyl benzene		5							
p-Isopropyltoluene		5							
Methylene Chloride	1260	10	Yes			8.9	Yes		
Pentanone		20							
MTBE		5							
Napthalene		10							
n-Propylbenzene		5				140	Yes		
Styrene	444.9	5	Yes			1700	Yes		
TAME		5							
Tetrachloroethane		5				0.38	Yes		
Tetrachloroethene	195.8	5	Yes			5.7	Yes		
Toluene	52500	5	Yes			520	Yes		
1,1,1-Trichloroethane	246.8	5	Yes			630	Yes		
1,1,2-Trichloroethane	673.5	5	Yes			0.84	Yes		
1,2,3-Trichlorobenzene		5							
1,2,4-Trichlorobenzene	11700	5	Yes			650	Yes		
Trichloroethene	179.5	5	Yes			2.8	Yes		
Trichloroflurorometh ane		5				390	Yes		
1,2,3-Trichloropropane		5				0.0014	No	0.0017	No
trifluoroethane		5							
1,2,4-Trimethylbenzene		5				52	Yes		
1,3,5-Trimethylbenzene	0.121	5	No	1.3	No	21	Yes		
Vinyl Acetate		5				430	Yes		
Vinyl Chloride	2	5	No	6.9	No	0.15	Yes		
m,p-Xylene	1	10	No	2.2	No	210	Yes		
o-Xylene	1	5	No	0.9	Yes	210	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8270C – SVOCs
Soil and Sediment

EPA 8270									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(ug/kg)	**(ug/kg)**	**Yes/No**	**(ug/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Acenaphthene	6.71	330	No	328	No	3700	Yes		
Acenapthylene	5.9	330	No	363	No				
Aniline	N/A	660				85	Yes		
Anthracene	453.4	330	Yes			22000	Yes		
Benzo(a)anthracene	31.7	330	No	250	No	0.62	Yes		
Benzo(a)pyrene	31.9	330	No	275	No	0.062	No	0.275	Yes
Benzo(b)fluoranthene	10400	330	Yes			0.62	Yes		
Benzo(g,h,i)perylene	170	330	No	446	No				
Benzo(k)fluoranthene	240	330	No	500	No	6.2	Yes		
Benzoic Acid		330				100000	Yes		
Benzyl Alcohol		330				18000	Yes		
Chloroethoxymethane		330							
bis(2-chloroethyl)ether		330				0.21	No	0.404	No
Chloroisopropylether		330							
ethylhexylphthalate		330							
phenylether		330							
Butylbenzylphthalate	4190	330	Yes			12000	Yes		
Carbazole		330				24	Yes		
4-Chloro-3-methylphenol	388	330	Yes						
4-Chloroaniliine		330				240	Yes		
2-Chloronaphthalene		330				3900	Yes		
2-Chlorophenol		330				63	Yes		
phenylether		330							
Chrysene	57.1	330	No	250	No	62	Yes		
Dibenzo(a,h)anthracene	6.22	330	No	488	No	0.062	No	0.488	Yes
Dibenzofuran	1520	330	Yes			290	Yes		
1,2-Dichlorobenzene	231.32	330	No	446	No	370	Yes		
1,3-Dichlorobenzene	3010	330	Yes			13	Yes		
1,4-Dichlorobenzene	1450	330	Yes			3.4	Yes		
3,3'-Dichlorobenzidine	28.22	660	No	477	No	1.1	Yes		
2,4-Dichlorophenol	133.63	330	No	447	No	180	Yes		
Diethylphthalate	8.04	330	No	255	No	49000	Yes		
2,4-Dimethylphenol	304.5	330	No	366	No	1200	Yes		
Dimethylphthalate	24.95	330	No	250	No	100000	Yes		
di-n-buylphthalate	110.5	330	No	250	No	6100	Yes		
methylphenol	0.8	660	No	386	No	3100	Yes		
2,4-Dinitrophenol	1.33	660	No	500	No	120	Yes		
2,4-Dinitrotoluene	75.13	330	No	250	No	120	Yes		
2,6-Dinitrotoluene	20.62	330	No	250	No	61	Yes		
Di-n-octylphthalate	40600	330	Yes			1200	Yes		
1,2-Diphenylhydrazine		330				0.61	Yes		
EPA 8270									

Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL *3	PRG Attainable
	(ug/kg)	(ug/kg)	Yes/No	(ug/kg)	Yes/No	(mg/kg)	Yes/No	(mg/kg)	Yes/No
fluoranthene	111.3	330	No	250	No	2300	Yes		
Fluorene	19	330	No	250	No	2600	Yes		
Hexachlorobenzene	20	330	No	500	No	0.3	No	0.5	No
hexachlorobutadiene	1380	330	Yes			6.2	Yes		
Hexachloroethane	2230	330	Yes			35	Yes		
Indeno(1,2,3-cd)pyrene	200	330	No	474	No	0.62	Yes		
Isophorone	422.36	330	Yes			510	Yes		
2-Methylnaphthalene	20.2	330	No	410	No				
2-Methylphenol	0.826	330	No	355	No	3100	Yes		
4-Methylphenol	0.808	330	No	350	No	310	Yes		
Naphthalene	34.6	330	No	426	No	56	Yes		
2-Nitroaniline	0.222	660	No	324	No	3.5	Yes		
3-Nitroaniline	0.222	660	No	276	No				
4-Nitroaniline	0.222	330	No	500	No				
Nitrobenzene	487.6	330	Yes			20	Yes		
2-Nitrophenol	7.77	330	No	469	No				
4-Nitrophenol	7.78	660	No	500	No	490	Yes		
n-Nitrosodimethylamine		330				0.0095	No	0.357	No
Pentachlorophenol	30100	660	Yes			3	Yes		
Phenanthrene	41.9	330	No	250	No				
Phenol	27.3	330	No	378	No	37000	Yes		
Pyrene	53	330	No	250	No				
Pyridine		330				61	Yes		
1,2,4-Trichlorobenzene	11700	330	Yes			650	Yes		
2,4,5-Trichlorophenol	85.6	330	No	250	No	6100	Yes		
2,4,6-Trichlorophenol	84.8	330	No	400	No	44	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8082 PCBs
Soil and Sediment

EPA 8082									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(mg/kg)	**(ug/kg)**	**Yes/No**	**(ug/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
PCB-1016	7	50	Yes			3.9	Yes		
PCB-1221		50				0.22			
PCB-1232		50				0.22			
PCB-1242		50				0.22			
PCB-1248	30	50	Yes			0.22	Yes		
PCB-1254	60	50	Yes			0.22	Yes		
PCB-1260	5	50	Yes			0.22	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SquiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8081A – Pesticides
Soil and Sediment

EPA 8081A									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(ug/kg)	**(ug/kg)**	**Yes/No**	**(ug/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Aldrin	2	2	Yes			0.029	Yes		
alpha-BHC	6	2	Yes						
beta-BHC	5	2	Yes						
delta-BHC		2							
gamma-BHC	0.94	2	No	0.63	yes				
alpha-Chlordane	0.03	2	No	0.63	no				
gamma-Chlordane	0.03	2	No	0.63	no				
4,4'-DDD	0.004	4	No	1.35	no	2.4	Yes		
4,4'-DDE	0.004	4	No	1.59	no	1.7	Yes		
4,4'-DDT	0.004	4	No	3.99	no	1.7	Yes		
Dieldrin	0.01	4	No	1.5	no	0.003	No	0.001	No
Endosulfan I	0.17	2	No	2	no	370	Yes		
Endosulfan II	0.1	4	No	1.26	no				
Endosulfan Sulfate	34.6	4	Yes						
Endrin	0.014	4	No	2.73	no	18	Yes		
Endrin Aldehyde		4							
Endrin Ketone		4							
Heptachlor	0.26	2	No	1.77	no	0.11	Yes		
Heptachlor Epoxide	0.008	4	No	4	no	0.053	Yes		
Methoxychlor	3.5	20	No	8.79	no	310	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8151 - Herbicides
Soil and Sediment

EPA 8151									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(ug/kg)	**(ug/kg)**	**Yes/No**	**(ug/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
2,4,5-T		10							
2,4,5-TP(Silvex)	108.8	10	Yes						
2,4-D	27.25	10	Yes			690	Yes		
2,4-DB		20				490	Yes		
Dalapon		20				1800	Yes		
Dicamba		20				26000	Yes		
Dichloroprop		10							
Dinoseb	21.8	10	Yes			61	Yes		
MCPA		2000							
MCPP		2000							

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8330 - Explosives
Soil and Sediment

EPA 8330									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(mg/kg)	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
4-Amino-2,6 DNT		0.4							
2-Amino-4,6 DNT		0.4							
1,3-DNB	3	0.4	Yes			6.1	Yes		
2,4 DNT	0.6	0.4	Yes			120	Yes		
2,6 DNT		0.4				61	Yes		
HMX	5	0.4	Yes			3100	Yes		
Nitrobenzene		0.4				20	Yes		
2-Nitrotoluene	149	0.4	Yes			370	Yes		
3-Nitrotoluene	149	0.4	Yes			370	Yes		
4-Nitrotoluene	149	0.4	Yes			370	Yes		
RDX	1	0.4	Yes			4.4	Yes		
Tetryl	4	0.4	Yes						
1,3,5-TNB	1	0.4	Yes			1800	Yes		
2,4,6-TNT	5	0.4	Yes			16	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8310 – Polyaromatic Hydrocarbons Soil and Sediment

EPA 8310									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(ug/kg)	**Ug(/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Acenaphthene	6.71	100	No			3700	Yes		
Acenaphthylene	5.9	200	No						
Anthracene	453.4	10	Yes			22000	Yes		
Benzo(a)anthracene	31.7	10	Yes			0.62	Yes		
Benzo(a)pyrene	31.9	10	Yes			0.062	Yes		
Benzo(b)fluoranthene	10400	20	Yes			0.62	Yes		
Benzo(g,h,i)perylene	170	20	Yes						
Benzo(k)fluoranthene	240	10	Yes			6.2	Yes		
Chrysene	57.1	10	Yes			62	Yes		
Dibenzo(a,h)anthracene	6.22	20	No			0.062	Yes		
Fluoranthene	111.3	20	Yes			2300	Yes		
Fluorene	19	20	Yes			2600	Yes		
Indeno(1,2,3-cd)pyrene	200	10	Yes			0.62	Yes		
Naphthalene	34.6	100	No			56	Yes		
Phenanthrene	51.9	10	Yes						
Pyrene	53	10	Yes						

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 6010B - Metals
Soil and Sediment

EPA 6010									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(mg/kg)	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Aluminum		20				76000	Yes		
Antimony	2	10	No	10	No	31	Yes		
Arsenic	5.9	10	No	8.07	No	0.39	No	8.07	No
Barium	218	1	Yes			5400	Yes		
Beryllium	2.5	1	Yes			150	Yes		
Boron		10				5500	Yes		
Cadmium	0.5	1	No	1	No	37	Yes		
Calcium		100							
Chromium III	26	2	Yes			100000	Yes		
Chromium VI	0.4	2	No	2	No	30	Yes		
Cobalt	21	2	Yes			4700	Yes		
Copper	16	2	Yes						
Iron	20	20	Yes			23000	Yes		
Lead	31	10	Yes			400	Yes		
Magnesium		100							
Manganese	460	1	Yes			1800	Yes		
Molybdenum		5				390	Yes		
Nickel	16	2	Yes			1600	Yes		
Potassium		500							
Selenium	0.4	10	No	10	No	390	Yes		
Silver	0.09	2	No	2	No	390	Yes		
Sodium		100							
Strontium		1				47000	Yes		
Thallium	0.4	5	No	5	No	5.2	Yes		
Tin		5				47000	Yes		
Vanadium	52	2	Yes			550	Yes		
Zinc	112	1	Yes			23000	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 6010B TRACE - Metals
Soil and Sediment

EPA 6010B TRACE									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(mg/kg)	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Aluminum									
Antimony	2.0								
Arsenic	5.9	1.0	Yes			0.39	No		
Barium									
Beryllium									
Boron									
Cadmium	0.5	1.0	No			37	Yes		
Calcium									
Chromium									
Cobalt									
Copper	16.0	1.0	Yes						
Iron									
Lead	31.0	1.0				400	Yes		
Magnesium									
Manganese	460.0	1.0	Yes			1800	Yes		
Molybdenum									
Nickel									
Potassium									
Selenium	0.4	1.0	No			390	Yes		
Silver									
Sodium									
Strontium									
Thallium	0.4	2.0	No			5.2	Yes		
Tin									
Vanadium	52	1.0	Yes			550	Yes		
Zinc									

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 7471 - Mercury
Soil and Sediment

EPA 7471									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(mg/kg)	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Mercury	0.1	0.1	Yes			23	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 9010/9014 - Cyanide
Soil and Sediment

EPA 9010/9014									
Parameter	**Target Ecological PRG**	**RL**	**PRG Attainable**	**MDL *3**	**PRG Attainable**	**HH PRG**	**PRG Attainable**	**MDL *3**	**PRG Attainable**
	(mg/kg)	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**	**(mg/kg)**	**Yes/No**
Cyanide	0.001	1	No			11	Yes		

NOTES:
Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8260B – VOCs
Surface and Groundwater

EPA 8260B Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	water ug/L	Yes/No	ug/L	Yes/No
Acetone	78000	10	yes	5.7	yes			5.7		610	yes	5.7	yes
Acrolein	0.205	50	no	13.5	no	320	yes	13.5	yes	0.042	no	13.5	no
Acrylonitrile	0.89	10	no	13.8	no	0.059	no	13.8	no	0.039	no	13.8	no
Benzene	114	1	yes	0.6	yes	1.2	yes	0.6	yes	0.35	no	0.6	no
Bromobenzene		1		0.6				0.6		2	yes	0.6	yes
Bromochloromethane		1		0.3				0.3				0.3	
Bromodichloromethane		1		0.3				0.3		0.18	no	0.3	no
Bromoform	466	1	yes	0.6	yes	4.3	yes	0.6	yes	8.5	yes	0.6	yes
Bromomethane		1		0.3				0.3		8.7	yes	0.3	yes
t-Butanol		25		14.7				14.7		3600	yes	14.7	yes
2-Butanone (MEK)		10		5.4				5.4				5.4	
n-Butylbenzene		1		0.3				0.3		61	yes	0.3	yes
sec-Butylbenzene		1		0.6				0.6		61	yes	0.6	yes
tert-Butylbenzene		1		0.3				0.3		61	yes	0.3	yes
Carbon Disulfide	84.1	1	yes	0.3	yes			0.3		1000	yes	0.3	yes
Carbon Tetrachloride	5.9	1	yes	0.6	yes	0.25	no	0.6	no	0.17	no	0.6	no
Chlorobenzene	10	1	yes	0.3	yes	680	yes	0.3	yes	110	yes	0.3	yes
Chloroethane	230000	1	yes	0.6	yes			0.6		4.6	yes	0.6	yes
Chloroform	79	1	yes	0.3	yes	5.7	yes	0.3	yes	0.16	no	0.3	no

EPA 8260B Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	water ug/L	Yes/No	ug/L	Yes/No
1-Chlorohexane		1		0.3				0.3				0.3	
Chloromethane		1		1.2				1.2		1.5	yes	1.2	yes
2-Chlorotoluene		1		0.6				0.6		120	yes	0.6	yes
4-Chlorotoluene		1		0.6				0.6				0.6	
DIPE		2		0.3				0.3				0.3	
Dibromochloromethane	6400	1	yes	0.6	yes			0.6		0.13	no	0.6	no
1,2-Dibromo-3-chloropropane		2		0.6				0.6		170	yes	0.6	yes
1,2-Dibromoethane	22.5	1	yes	0.6	yes			0.6		0.00076	no	0.6	no
Dibromomethane		1		0.6				0.6				0.6	
1,1-Dichloroethane	47	1	yes	0.3	yes			0.3		810	yes	0.3	yes
1,2-Dichloroethane	190	1	yes	0.6	yes	0.38	no	0.6	no	0.12	no	0.6	no
1,2-Dichlorobenzene	87	1	yes	0.6	yes			0.6		370	yes	0.6	yes
1,3-Dichlorobenzene	11	1	yes	0.6	yes			0.6		5.5	yes	0.6	yes
1,4-Dichloro-2-butene		10		0.9				0.9		0.0012	no	0.9	no
1,4-Dichlorobenzene	43	1	yes	0.3	yes			0.3		0.5	no	0.3	yes
Dichlorodifluoromethane		1		1.2				1.2		390	yes	1.2	yes
1,1-Dichloroethene	78	1	yes	0.3	yes	0.057	no	0.3	no	810	yes	0.3	yes
cis-1,2-Dichloroethene		1		0.3				0.3		61	yes	0.3	yes
trans-1,2-Dichloroethene	310	1	yes	0.6	yes			0.6		120	yes	0.6	yes
1,1-Dichloropropene		1		0.6				0.6		0.4	no	0.6	no
1,2-Dichloropropane	380	1	yes	0.6	yes	0.52	no	0.6	no	0.16	no	0.6	no
1,3-Dichloropropane		1		0.6		10	yes	0.6	yes			0.6	
2,2-Dichloropropane		1		0.6				0.6				0.6	

EPA 8260B Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
cis-1,3-Dichloropropene	7.9	1	yes	0.6	yes			0.6				0.6	
Dichloropropene		1		0.6				0.6				0.6	
ETBE		1		0.3				0.3				0.3	
Ethyl Methacrylate		1		0.6				0.6		550	yes	0.6	yes
Ethylbenzene	17.2	1	yes	0.3	yes	3100	yes	0.3	yes	1300	yes	0.3	yes
2-Hexanone	1710	10	yes	3	yes			3				3	
Hexachlorobutadiene	0.223	1	no	0.6	no			0.6		0.86	no	0.6	yes
Iodomethane		2		0.3				0.3				0.3	
Isopropyl benzene		1		0.6				0.6				0.6	
p-Isopropyltoluene		1		0.6				0.6				0.6	
Methylene Chloride	430	2	yes	0.3	yes	4.7	yes	0.3	yes	4.3	yes	0.3	yes
4-Methyl-2-Pentanone		10		3				3				3	
MTBE		1		0.3				0.3				0.3	
Napthalene	44	1	yes	0.9	yes			0.9				0.9	
n-Propylbenzene		1		0.6				0.6		61	yes	0.6	yes
Styrene	56	1	yes	0.3	yes			0.3		1600	yes	0.3	yes
TAME		1		0.6				0.6				0.6	
1,1,1,2-Tetrachloroethane	13	1	yes	0.6	yes	0.17	no	0.6	no	0.055	no	0.6	no
1,1,2,2-Tetrachloroethane	8.9	1	yes	0.9	yes	0.8	no	0.9	no	1.1	yes	0.9	yes
Toluene	253	1	yes	0.3	yes	6800	yes	0.3	yes	720	yes	0.3	yes
1,1,1-Trichloroethane	88	1	yes	0.3	yes			0.3		540	yes	0.3	yes
1,1,2-Trichloroethane	650	1	yes	0.6	yes	0.6	no	0.6	yes	0.2	no	0.6	no

EPA 8260B Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	water ug/L	Yes/No	ug/L	Yes/No
1,2,3-Trichlorobenzene		1		0.6				0.6				0.6	
1,2,4-Trichlorobenzene	69.2	1	yes	0.6	yes			0.6		190	yes	0.6	yes
Trichloroethene	75	1	yes	0.6	yes			0.6				0.6	
Trichloroflurormethane		1		0.6				0.6		1300	yes	0.6	yes
1,2,3-Trichloropropane	12.11	1	yes	0.6	yes			0.6		1600	yes	0.6	yes
trifluoroethane		1		0.6				0.6				0.6	
1,2,4-Trimethylbenzene		1		0.3				0.3		12	yes	0.3	yes
1,3,5-Trimethylbenzene		1		0.6				0.6		12	yes	0.6	yes
Vinyl Acetate	248.03	2	yes	2.1	yes			2.1		410	yes	2.1	yes
Vinyl Chloride	9.2	1	yes	0.9	yes	2	yes	0.9	yes	0.041	no	0.9	no
m,p-Xylene	117	2	yes	0.6	yes			0.6		1400	yes	0.6	yes
o-Xylene	117	1	yes	0.3	yes			0.3		1400	yes	0.3	yes

Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8270C – SVOCs
Surface and Groundwater

EPA 8270 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Acenaphthene	9.9	10	yes	15	no	1200	yes	15	yes			15	
Acenapthylene	4840	10	yes	15	yes			15				15	
Aniline	0.44	40	no	39	no			39		12	no	39	no
Anthracene	0.029	10	no	14.4	no	9600	yes	14.4	yes	1800	yes	14.4	yes
Benzo(a)anthracene	0.839	10	no	12.6	no	0.0044	no	12.6	no	0.092	no	12.6	no
Benzo(a)pyrene	0.014	10	no	12.3	no	0.0044	no	12.3	no	0.0092	no	12.3	no
Benzo(b)fluoranthene	9.07	10	no	11.4	no	0.0044	no	11.4	no	0.092	no	11.4	no
Benzo(g,h,i)perylene	7.64	10	no	12.9	no			12.9				12.9	
Benzo(k)fluoranthene	0.0056	10	no	14.4	no	0.0044	no	14.4	no	0.92	no	14.4	no
Benzoic Acid		10		7.5				7.5		150000	yes	7.5	yes
Benzyl Alcohol	281.24	10	yes	15.3	yes			15.3		11000	yes	15.3	yes
Chloroethoxymethane		10		13.9				13.9				13.9	
bis(2-chloroethyl)ether		10		15.3		0.031	no	15.3	no	0.0098	no	15.3	no
Chloroisopropylether		10		16.2		1400	yes	16.2	yes			16.2	
ethylhexylphthalate		20		29.7				29.7				29.7	
4-Br-Ph-phenylether		20		20.4				20.4				20.4	
Butylbenzylphthalate	49	10	yes	11.7	yes	3000	yes	11.7	yes	7300	yes	11.7	yes
Carbazole		10		15				15		3.4	no	15	no
4-Chloro-3-methylphenol		10		14.4				14.4				14.4	
4-Chloroaniliine	231.97	10	yes	14.4	yes			14.4		150	yes	14.4	yes
2-Chloronaphthalene	0.396	10	no	14.4	no	1700	yes	14.4	yes	490	yes	14.4	yes
2-Chlorophenol	8.8	10	no	14.4	no			14.4		30	yes	14.4	yes
4-Cl-Ph-phenylether		10		15.6				15.6				15.6	
Chrysene	0.033	10	no	12	no	0.0044	no	12	no	9.2	no	12	no

EPA 8270 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Dibenzo(a,h)anthracene	0.0016	10	no	14.4	no	0.0044	no	14.4	no	0.0092	no	14.4	no
Dibenzofuran	20	10	yes	14.4	yes			14.4		24	yes	14.4	yes
1,2-Dichlorobenzene	87	10	yes	13.8	yes	2700	yes	13.8	yes	370	yes	13.8	yes
1,3-Dichlorobenzene	11	10	yes	13.2	no	400	yes	13.2	yes	5.5	no	13.2	no
1,4-Dichlorobenzene	43	10	yes	13.5	yes	400	yes	13.5	yes	0.5	no	13.5	no
3,3'-Dichlorobenzidine	99.75	10	yes	12.3	yes	0.04	no	12.3	no	0.15	no	12.3	no
2,4-Dichlorophenol	18	10	yes	13.2	yes	93	yes	13.2	yes	110	yes	13.2	yes
Diethylphthalate	3	20	no	17.1	no	23000	yes	17.1	yes	29000	yes	17.1	yes
2,4-Dimethylphenol	100.17	10	yes	13.2	yes	540	yes	13.2	yes	730	yes	13.2	yes
Dimethylphthalate	73	20	yes	16.5	yes	313000	yes	16.5	yes	360000	yes	16.5	yes
di-n-buylphthalate	3	10	no	14.4	no	2700	yes	14.4	yes			14.4	
4,6-Dinitro-2-methylphenol		20		19.5				19.5				19.5	
2,4-Dinitrophenol	4.07	20	no	17.7	no	70	yes	17.7	yes	73	yes	17.7	yes
2,4-Dinitrotoluene	230	20	yes	18.9	yes	0.11	no	18.9	no	73	yes	18.9	yes
2,6-Dinitrotoluene	42	20	yes	18.9	yes			18.9		36	yes	18.9	yes
Di-n-octylphthalate	30	10	yes	13.8	yes			13.8		730	yes	13.8	yes
1,2-Diphenylhydrazine		10		13.5		0.04	no	13.5	no	0.084	no	13.5	no
Fluoranthene	8.1	10	no	15.9	no	300	yes	15.9	yes	1500	yes	15.9	yes
Fluorene	3.9	10	no	14.7	no	1300	yes	14.7	yes	240	yes	14.7	yes
Hexachlorobenzene	0.00024	20	no	16.5	no	0.00075	no	16.5	no	0.042	no	16.5	no
Hexachlorobutadiene	0.223	10	no	11.7	no	0.44	no	11.7	no	0.86	no	11.7	no
Hexachlorocyclopentadiene		10		15				15				15	
Hexachloroethane	30.5	10	yes	12	yes	1.9	no	12	no	4.8	no	12	no
Indeno(1,2,3-cd)pyrene	4.31	10	no	12	no	0.0044	no	12	no	0.092	no	12	no
Isophorone	900	10	yes	13.8	yes	36	yes	13.8	yes	71	yes	13.8	yes

EPA 8270 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
2-Methylnaphthalene		10		12				12				12	
2-Methylphenol		10		10.5				10.5		1800	yes	10.5	yes
4-Methylphenol		10		11.7				11.7		180	yes	11.7	yes
Naphthalene	44	10	yes	12.9	yes			12.9				12.9	
2-Nitroaniline		20		17.1				17.1		2.1	no	17.1	no
3-Nitroaniline		10		14.4				14.4				14.4	
4-Nitroaniline		10		14.4				14.4				14.4	
Nitrobenzene	740	10	yes	15.3	yes	17	yes	15.3	yes	3.4	no	15.3	no
2-Nitrophenol	13.5	10	yes	14.1	no			14.1				14.1	
4-Nitrophenol	35	10	yes	15.3	yes			15.3		290	yes	15.3	yes
n-Nitrosodimethylamine		10		12.6		0.00069	no	12.6	no	0.0013	no	12.6	no
Pentachlorophenol	5.23	20	no	19.2	no			19.2		0.56	no	19.2	no
Phenanthrene	2.1	20	no	17.1	no			17.1				17.1	
Phenol	100	10	yes	12.3	yes			12.3		22000	yes	12.3	yes
Pyrene	0.3	10	no	14.1	no	960	yes	14.1	yes	180	yes	14.1	yes
Pyridine	2380	20	yes	25.8	yes			25.8		36	yes	25.8	yes
1,2,4-Trichlorobenzene	69.2	10	yes	10.8	yes	260	yes	10.8	yes	190	yes	10.8	yes
2,4,5-Trichlorophenol		10		12.9				12.9		3600	yes	12.9	yes
2,4,6-Trichlorophenol	2	10	no	14.4	no	2.1	no	14.4	no	6.1	no	14.4	no

Blank Cell indicates no value available in current sources
NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs
HH PRGs selected from Region 9 PRG
Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8082 – PCBs
Surface and Groundwater

EPA 8082 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
PCB-1016		1								0.96			
PCB-1221	0.28	1	No							0.34	No		
PCB-1232	0.58	1	No							0.34	No		
PCB-1242	0.053	1	No							0.34	No		
PCB-1248	0.81	1	No							0.34	No		
PCB-1254	0.33	1	No							0.34	No		
PCB-1260	94	1	Yes							0.34	No		
TOTAL PCB	0.00017									0.34			

Blank Cell indicates no value available in current sources

NOTE: Change of Units ug/kg to mg/kg from Eco to HH PRGs

HH PRGs selected from Region 9 PRG

Eco PRGs selected as most conservative from Region 5 EDQL, Ontario MOE, NOAA SQuiRTs, and NOAA ER-Ls

PRGS and Method Reporting Limits – Method 8081 – Pesticides
Surface and Groundwater

EPA 8081 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Aldrin	0.031	0.1	yes			0.00013	no	0.03	no	0.004	no	0.03	no
alpha-BHC	12.38	0.1	yes										
beta-BHC	0.495	0.1	yes										
delta-BHC	666.67	0.1	yes										
gamma-BHC	0.01	0.1	no	0.024	No	0.019	no	0.015	yes				
alpha-Chlordane	0.00029	0.1	no	0.015	No	0.0022	no	0.015	no	0.19	yes	0.015	
gamma-Chlordane	0.00029	0.1	no	0.015	No	0.0022	no	0.015	no	0.19	yes	0.015	
4,4'-DDD	0.0011	0.2	no	0.096	No	0.00083	no	0.096	no	0.28	yes	0.096	
4,4'-DDE	4.51E-09	0.2	no	0.09	No	0.00059	no	0.09	no	0.2	yes	0.09	
4,4'-DDT	0.001	0.2	no	0.057	No	0.00059	no	0.057	no	0.2	yes	0.057	
Dieldrin	0.000026	0.2	no	0.21	No	0.00014	no	0.21	no	0.0042	no	0.21	no
Endosulfan I	0.003	0.1	no	0.075	No	110	yes	0.075		220	yes	0.075	
Endosulfan II	0.003	0.2	no	0.03	Yes	110	yes	0.03		220	yes	0.03	
Endosulfan Sulfate	2.22	0.2	yes			110	yes	0.033				0.033	
Endrin	0.002	0.2	no	0.03	No	0.76	yes	0.03		11	yes	0.03	
Endrin Aldehyde	0.15	0.2	no	0.03	Yes	0.76	yes	0.03				0.03	
Endrin Ketone		0.2						0.033				0.033	
Heptachlor	0.00039	0.1	no	0.024	No	0.00021	no	0.024	no	0.015	no	0.024	no
Heptachlor Epoxide	0.00048	0.1	no	0.015	No	0.0001	no	0.015	no	0.0074	no	0.015	no
Methoxychlor	0.005	1	no	0.246	No	100	yes	0.246		180	yes	0.246	

Blank Cell indicates no value available in current sources

HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)

HH PRGs GW (groundwater) are taken from the MCLs

Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 8151 – Herbicides
Surface and Groundwater

EPA 8151 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
2,4,5-T													
2,4,5-TP (Silvex)	326.64	0.4	Yes	0.6	Yes								
2,4-D		0.4											
2,4-DB		0.4											
Dalapon		0.4								1100	Yes	0.24	Yes
Dicamba		0.4								1100	yes	0.24	Yes
Dichloroprop		0.4											
Dinoseb	0.39	0.4	Yes	0.36	Yes					36	Yes	0.36	Yes
MCPA		200											
MCPP		200											

Blank Cell indicates no value available in current sources
HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)
HH PRGs GW (groundwater) are taken from the MCLs
Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 8330 – Explosives
Surface and Groundwater

EPA 8330 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
4-Amino-2,6 DNT													
2-Amino-4,6 DNT	20	1	yes	0.6	Yes								
1,3-DNB	20	1	yes	0.3	Yes					3.6	yes	0.6	yes
2,4 DNT	230	1	yes	0.3	Yes	0.11	no	0.3	no	73	yes	0.3	yes
2,6 DNT	42	1	yes	0.6	Yes					36	yes	0.3	yes
HMX										1800	yes	0.6	yes
Nitrobenzene		1		0.6		17	yes	0.6	yes	3.4	yes	0.6	yes
2-Nitrotoluene										61	yes	0.6	yes
3-Nitrotoluene										61	yes	0.6	yes
4-Nitrotoluene										61	yes	0.9	yes
RDX		1								0.61	no	0.6	yes
Tetryl													
1,3,5-TNB	11	1	yes	0.6	Yes					1100	yes	0.6	yes
2,4,6-TNT	90	1	yes	0.3	Yes					2.2	yes	0.3	yes

Blank Cell indicates no value available in current sources
HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)
HH PRGs GW (groundwater) are taken from the MCLs
Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 8310 – Polyaromatic Hydrocarbons
Surface and Groundwater

EPA 8151 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Acenaphthene	9.9	10	Yes			1200	Yes						
Acenaphthylene	4840	10	Yes										
Anthracene	0.029	10	No			9600	Yes			1800	Yes		
Benzo(a)anthracene	0.839	10	No			0.0044	No			0.092	No		
Benzo(a)pyrene	0.014	10	No			0.0044	No			0.0092	No		
Benzo(b)fluoranthene	9.07	10	No			0.0044	No			0.092	No		
Benzo(g,h,i)perylene	7.64	10	No										
Benzo(k)fluoranthene	0.0056	10	No			0.0044	No			0.92	No		
Chrysene	0.033	10	No			0.0044	No			9.2	No		
Dibenzo(a,h)anthracene	0.0016	10	No			0.0044	No			0.0092	No		
Fluoranthene	8.1	10	No			300	Yes			1500	Yes		
Fluorene	3.9	10	No			1300	Yes			240	Yes		
Indeno(1,2,3-cd)pyrene	4.31	10	No			0.0044	No			0.092	No		
Naphthalene	44	10	Yes										
Phenanthrene	2.1	20	No										
Pyrene	0.3	10	No			960	Yes			180	Yes		

Blank Cell indicates no value available in current sources
HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)
HH PRGs GW (groundwater) are taken from the MCLs
Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 6010B – ICP Metals
Surface and Groundwater

EPA 6010B Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Aluminum	750(WQC)	200	yes	150	Yes					36000	yes	150	yes
Antimony	31 (EDQL)	100	no	150	No	14	no	150	no	15	no	150	no
Arsenic	53 (EDQL)	100	no	75	No	0.018	no	75	no	0.045	no	75	no
Barium	5000 (EDQL)	10	yes	3	No					2600	yes	3	yes
Beryllium	7.60 (EDQL)	10	no	3	No					73	yes	3	yes
Boron										3300			
Cadmium	0.66 (EDQL)	10	no	15	No								
Calcium													
Chromium III	74 (WQC)	20	yes	18	Yes					55000	yes	18	yes
Chromium VI	11 (WQC)	20	no	18	No					0.16	no	18	no
Cobalt	5.0 (EDQL)	20	no	33	No					2200	yes	33	yes
Copper	5.0 (EDQL)	1000	no	15	No	1300	yes	15	yes	1400	yes	15	yes
Iron	1000 (WQC)			18	Yes	300	yes	18	yes	11000	yes	18	yes
Lead	1.3 (EDQL)	100	no	75	No								
Magnesium													
Manganese						50				880	yes		
Molybdenum										180			
Nickel	29.0 (EDQL)	20	no	60	No	610	yes	60	yes	730	yes	60	yes
Potassium		5000											
Selenium	5.0 (EDQL)	1000	no	159	No	170	no	159	yes	180	yes	159	yes
Silver	1.0 (EDQL)	20	no	30	No					180	yes	30	yes

EPA 6010B Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Sodium		1000											
Strontium										22000			
Thallium	0.56 (EDQL)	100	no	1.68	No	1.7	no	1.68	yes	2.4	no	1.68	no
Tin	73.0 (EDQL)	100	no	219	No					22000	yes	219	yes
Vanadium	19.0 (EDQL)	20	no	15	yes					260	yes	15	yes
Zinc	58.90 (EDQL)	100	no	176.7	No	9100	yes	176.7	yes	11000	yes	176.7	yes

Blank Cell indicates no value available in current sources
HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)
HH PRGs GW (groundwater) are taken from the MCLs
Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 6010B TRACE– ICP Metals
Surface and Groundwater

EPA 6010B TRACE Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Aluminum													
Antimony													
Arsenic	53	10	Yes			0.018	No			0.045	No		
Barium													
Beryllium	7.6	10	No							73	Yes		
Boron													
Cadmium	0.66	10	No										
Calcium													
Chromium III	74	10	Yes							55000	Yes		
Chromium VI	11	10	Yes							0.16	No		
Cobalt	5.0	10	No							2200	Yes		
Copper	5.0	10	No			1300	Yes			1400	Yes		
Iron	1000	10	Yes			300	Yes			1100	Yes		
Lead	1.3	10	No										
Magnesium													
Manganese		10				50	Yes			880	Yes		
Molybdenum										180	Yes		
Nickel	29.0	10	Yes			610	Yes			730	Yes		
Potassium													
Selenium	5.0	10	No			170	Yes			180	Yes		
Silver	1.0	10	No							180	Yes		
Sodium													
Strontium													
Thallium	0.56	10	No			1.7	No			2.4	No		

EPA 6010B TRACE Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Tin	73.0	10	Yes							22000	Yes		
Vanadium	19.0	10	Yes							260	Yes		
Zinc													

Blank Cell indicates no value available in current sources
HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)
HH PRGs GW (groundwater) are taken from the MCLs
Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 7470 - Mercury
Surface and Groundwater

EPA 7470 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Mercury	0.0013	500	no	150	No								

Blank Cell indicates no value available in current sources
HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)
HH PRGs GW (groundwater) are taken from the MCLs
Eco PRGs selected from the Region 5 EDQLs

PRGS and Method Reporting Limits – Method 9010/9014 Cyanide
Surface and Groundwater

EPA 9010/9014 Parameter	Target Ecological PRG	RL	PRG Attainable	MDL *3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable	HH PRG	PRG Attainable	MDL * 3	PRG Attainable
						Surface				Ground			
	ug/L	ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No	Ug/L	Yes/No	ug/L	Yes/No	ug/L	Yes/No
Cyanide	5.2	10	no	9	No	700				6.2			

Blank Cell indicates no value available in current sources

HH PRGs for surface water are combined consumption of water and organism from the Water Quality Criteria (US EPA, 1999)

HH PRGs GW (groundwater) are taken from the MCLs

Eco PRGs selected from the Region 5 EDQLs

Appendix D
Sample Container, Preservation, and Holding Times

Sample Containers, Preservation, and Hold Times

CONVENTIONALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Acidity	100	P, G	Cool, 4° C	14 Days
Alkalinity	100	P, G	Cool, 4° C	14 Days
Anions	50	P, G	None	28 days for Br, F, CL, and SO4; 48 hours for NO3, NO2, and PO4
Total Solids	50	P, G	Cool, 4° C	7 Days
Ash Content @ 750° C	25	P, G	Cool, 4° C	---
Biochemical Oxygen Demand	500	P, G	Cool, 4° C	48 Hours
Boron	20	P	Cool, 4° C	6 Months
Bromide	1000	P, G	Cool, 4° C	28 Days
BTU	10	P, G	Cool, 4° C	---
Formaldehyde	20	G	Cool, 4° C	7 Days
Chloride	25	P, G	Cool, 4° C	28 Days
Chloride, Total Residual	100	P, G	Cool, 4° C	6 Hours
Cyanide (midi)	50	P, G	Cool, 4° C, NaOH, pH>12	14 Days
Cyanide, Amenable to Chlorination (midi)	100	P,G	Cool, 4° C, NaOH, pH>12	14 Days
Chemical Oxygen Demand	25	G	Cool, 4° C, H_2SO_4, pH<2	28 Days
Color, Platinum-Cobalt	50	P, G	Cool, 4° C	48 Hours
Coliform, Fecal	120	P, G	Cool, 4° C, $Na_2S_2O_3$	6 Hours
Fecal Streptococcus	100	P, G	Cool, 4° C, $Na_2S_2O_3$	6 Hours
Coliform, Total	100	P, G	Cool, 4° C, $Na_2S_2O_3$	6 Hours
Specific Conductance	100	P, G	Cool, 4° C	28 Days
Corrosivity	500	P, G	Cool, 4° C	---
Corrosivity (pH)	40	P, G	Cool, 4° C	---
Chromium, Trivalent	200	P, G	Cool, 4° C	---
Chromium, Hexavalent	150	P, G	Cool, 4° C	24 Hours
Dissolved Oxygen	300	G	Cool, 4° C	6 Hours
Fluoride	25	P, G	Cool, 4° C	28 Days
Ignitability	75	P, G	Cool, 4° C	---
Fluoride, Total (Distilled/Non-Distilled)	300 / 25	P, G	Cool, 4° C	28 Days
Hardness	100	P, G	Cool, 4° C, HNO3, pH<2	6 Months
Iodide	400	P, G	Cool, 4° C	24 Hours
Surfactants (MBAS)	100	P, G	Cool, 4° C	48 Hours
Coliform Fecal (MPN)	100	P, G	$Na_2S_2O_3$	6 Hours
Nitrogen, Ammonia (Distilled/Non-Distilled)	500 / 20	P, G	Cool, 4° C, H_2SO_4, pH<2	28 Days
Nitrogen, Nitrite	50	P, G	Cool, 4° C	48 Hours
Nitrogen, Nitrate	75	P, G	Cool, 4° C	48 Hours
Nitrogen, Nitrate-Nitrite	25	P, G	Cool, 4° C, H_2SO_4, pH<2	28 Days
Nitrogen, Organic	1000	P, G	Cool, 4° C, H_2SO_4, pH<2	28 Days
Threshold Odor	1000	G	Cool, 4° C	48 Hours
Oil and Grease	1000	G	Cool, 4° C, H_2SO_4, pH<2	28 Days
Petroleum Hydrocarbons	1000	G	Cool, 4° C, H_2SO_4, pH<2	28 Days
Perchlorate	50	P, G	None	28 days
Percent Moisture	25 g	P, G	Cool, 4° C	---
Percent Solids	25 g	P, G	Cool, 4° C	---
Paint Filter Liquids Test	100 g	P, G	Cool, 4° C	---

NOTES
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap

Sample Containers, Preservation, and Hold Times

CONVENTIONALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Phenolics, Total	500	Amber, G	Cool, 4° C, H_2SO_4, pH< 2	28 Days
Phosphorus, Total	50	P, G	Cool, 4° C, H_2SO_4, pH< 2	28 Days
pH Lab	40	P , G	Cool, 4° C	6 Hours
Orthophosphate	50	P, G	Cool, 4° C	48 Hours
Reactivity, Cyanide	10	P, G	Cool, 4° C	---
Reactivity, Sulfide	10	P, G	Cool, 4° C	---
Sulfite	50	P, G	Cool, 4° C	24 Hours
Settleable Solids	1000	P, G	Cool, 4° C	48 Hours
Silica, Dissolved	100	P	Cool, 4° C	28 Days
Sulfate	100	P, G	Cool, 4° C	28 Days
Specific Gravity	50	P, G	Cool, 4° C	---
Total (Organic) Sulfur	10	P, G	Cool, 4° C	---
Sulfide	200	P, G	Cool, 4° C, Zinc Acetate, NaOH, pH> 9	7 Days
Total Dissolved Solids	50	P, G	Cool, 4° C	7 Days
Total Suspended Solids	200	P, G	Cool, 4° C	7 Days
Turbidity	50	P, G	Cool, 4° C	48 Hours
Volatile Dissolved Solids	50	P, G	Cool, 4° C	7 Days
Total Volatile Solids	50	P, G	Cool, 4° C	7 Days
Volatile Suspended Solids	200	P, G	Cool, 4° C	7 Days

VOLATILE ORGANICS (VOA) - WATER

METHOD	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
8015M (GRO)	40 ml	G, Septa Caps	Cool, 4° C, HCl, pH< 2	14 Days
602/8021	40 ml	G, Septa Caps	Cool, 4° C, HCl, H_2SO_4, pH< 2	14 Days
603/8030	40 ml	G, Septa Caps	Cool, 4° C , $Na_2S_2O_3$, pH 4-5	14 Days
8260B	40 ml	G, Septa Caps	Cool, 4° C, HCl, pH< 2	14 Days
624	40 ml	G, Septa Caps	Cool, 4° C, $Na_2S_2O_3$	7 Days

NOTES
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap

Sample Containers, Preservation, and Hold Times

SEMIVOLATILE ORGANICS - WATER

METHOD	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
8015B Modified (DRO)	1000 ml	G	Cool, 4° C, H_2SO_4, pH< 2	14 Days
608/8081/8082	1000 ml	G	Cool, 4° C	7 Days
610/8100/8310	1000 ml	G	Cool, 4° C	7 Days
615/8151	1000 ml	G	Cool, 4° C	7 Days
625/8270C	1000 ml	G	Cool, 4° C	7 Days
8330	1000 ml	G	Cool, 4° C	7 days

METALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
All Metals (26)	1000 ml	P, G	HNO_3, pH< 2	6 Months
Mercury	100 ml	P, G	HNO_3, pH< 2, 4°	28 Days
Lead (furnace)	100 ml	P, G	HNO_3, pH< 2	6 Months
Chromium, Hexavalent	150 ml	P, G	Cool, 4° C	24 Hours

TCLP - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
TCLP Volatiles	250	G	Cool, 4° C	14 Days
TCLP Semi-Volatiles	1000	G	Cool, 4° C	14 Days
TCLP Pesticides	1000	G	Cool, 4° C	14 Days
TCLP Herbicides	1000	G	Cool, 4° C	14 Days
TCLP Metals	500	P, G	Cool, 4° C	6 Months*

*mercury is 28 days

NOTES
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. Triple the volumes above for MS/MSD samples

Sample Containers, Preservation, and Hold Times

SOIL

METHOD	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Coliform Fecal	4 oz	G	Cool, ,4° C	6 Hours
Chromium, Hexavalent	4 oz	G	Cool, 4° C	24 Hours
5035	5 g	P, G	Cool, 4° C	48 Hours
TCLP-VOA	4 oz	G	Cool, 4° C	14 Days
TCLP-SV	4 oz	G	Cool, 4° C	14 Days
TCLP-Pest/Herb	4 oz	G	Cool, 4° C	14 Days
TCLP-Metals (except Hg)	4 oz	G	Cool, 4° C	6 Months
Total Metals (except Hg)	4 oz	G	Cool, 4° C	6 Months
TPH	4 oz	G	Cool, 4° C	28 Days
Hg (Total & TCLP)	4 oz	G	Cool, 4° C	28 Days
Volatiles (5030)	4 oz	G	Cool, 4° C	14 Days
Volatiles (5035)	3 – Encores	G	Cool, 4° C	48 hours
Semi-Volatiles	4 oz	G	Cool, 4° C	14 Days
Conventionals (where applicable)	4 oz	G	Cool, 4° C	14 Days

*All soils stored at 4° C

NOTES
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap