

**QUALITY ASSURANCE PROJECT PLAN  
SAMPLING AND ANALYSIS PLAN - PART C**

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**Wenatchee Tree Fruit Research Center (TFREC) Test Plot  
Remediation  
Wenatchee, Washington  
DACA67-95-G-0001-029**

**Approvals:**

<b>Title</b>	<b>Signature</b>	<b>Date</b>
<b>Laboratory QA/QC Manager, Sound Analytical</b>		
<b>Laboratory QA/QC Manager, Cascade Analytical</b>		
<b>Laboratory QA/QC Manager, North Coast Analytical</b>		
<b>Laboratory QA/QC Manager, Columbia Analytical Services</b>		
<b>GSA Chemical Data Quality Manager</b>		



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Prepared by

**GARRY STRUTHERS ASSOCIATES, INC.**

Construction Management ♦ Environmental Sciences ♦ Project Management ♦ Engineering

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## **1. PURPOSE AND PROJECT DESCRIPTION**

The Quality Assurance Project Plan (QAPP) extensively references the Field Sampling Plan segment of the Sampling and Analysis Plan (SAP), the Laboratory Quality Assurance/Quality Control (QA/QC) Plan for each laboratory, and laboratory standard operating procedures. Laboratory validation documents, including the QA/QC Plan for each laboratory, have been submitted separately. The laboratory QA/QC plans are not project specific. The purpose of the QAPP is to outline data requirements, Data Quality Objectives (DQOs), and the practices and controls that are necessary during the sampling and analysis to maintain the required data quality.

### **1.1 SITE BACKGROUND**

The overall goal of the project is to remove soil contaminated above State of Washington Model Toxics Control Act ( MTCA Method B). Analyses will also be performed for federal and State of Washington waste designation.

### **1.2 PROJECT ACTIVITIES**

Refer to the Work Plan for the overall project description. The sampling work phases are described in the Field Sampling Plan.

## **2. PROJECT ORGANIZATION AND RESPONSIBILITIES**

The Statement of Chemical Qualifications (SOCQ) discusses organizational structure and individual responsibilities for project data quality management activities. The SOCQ (RAMP Section 2A) is incorporated by reference into this section.

## **3. DATA QUALITY OBJECTIVES**

### **3.1 BACKGROUND**

Refer to the Work Plan and Field Sampling Plan for background and a project outline. Refer to the Statement of Chemical Qualifications for a summary of the decisions to be made and types of data collected.

### **3.2 QA OBJECTIVES FOR CHEMICAL DATA MEASUREMENT**

The Field Sampling Plan provides a listing of all project health-based removal action levels (MTCA Method B cleanup values) and soil disposal action levels (Dangerous Waste Designation).

#### **3.2.1 Primary DQOs**

The primary data quality objective of the remedial action is to provide data of known and sufficient quality to accomplish the following:

- Determine the volume of soil within the test plot area that contains pesticides of concern (POCs) at concentrations greater than their respective MTCA Method B cleanup levels.
- Collect sufficient soil data to confirm that no significant residual concentrations of contaminants remain on site above MTCA Method B cleanup levels.
- Collect sufficient waste designation data to segregate wastes by categories and document proper disposal.

**3.2.2 Secondary DQOs**

The secondary data quality objective of the remedial action is to comply with the following remediation derived waste requirements:

- That incidental wastes generated during the removal, including all soil removed from the test plot area and wastewater, are disposed appropriately.

**3.3 PERFORMANCE REQUIREMENTS FOR ON-SITE SOIL IMMUNOCHEMICAL ANALYSIS**

Performance requirements for on-site soil sampling and analysis includes the following:

Two on-site immunochemical analyses, one for DDT and one for Cyclodienes, will be performed by GSA.

**3.3.1 Summary Reactivity/Sensitivity Information on Immunochemistry Kits**

A preliminary pilot study to evaluate the site-specific soil cross reactivity with the DDT and cyclodiene immunoassay kits was carried out by the Seattle District Corps. The characteristics of the kits selected for on-site immunochemical measurement during the removal action are summarized below.

The range of sensitivities of various pesticides in the reactivity group for the cyclodienes kit is shown in Table 1.

**Table 1  
Immunoassay Sensitivity for the Cyclodiene Reactivity Group**

<b>Constituent</b>	<b>LLD* (ppb)</b>	<b>Constituent</b>	<b>LLD (ppb)</b>
Dieldrin	6	Endrin	6
Aldrin	20	Toxaphene	200
Heptachlor	6	Gamma - BHC	600
Chlordane	14	Alpha - BHC	2,000
Endosulfan I	6	Delta - BHC	2,000
Endosulfan II	6		

\* LLD = lower limit of detection

The calibration range to be used for this project is as follows:

<b>Calibration Range</b>	<b>Low</b>	<b>Middle</b>	<b>High (ppb)</b>
Dieldrin/Endrin	18	86	512
Chlordane	40	200	1,200

The range of sensitivities of various pesticides in the reactivity group for the DDT kit is shown in Table 2.

**Table 2**  
**1**  
**Immunoassay Sensitivity for the DDT Reactivity Group**

<b>Constituent</b>	<b>LLD* (ppm)</b>	<b>Constituent</b>	<b>LLD (ppm)</b>
p,p DDT	0.04	Chlorobenzilate	0.03
p,p-DDD	0.01	Dicofol	0.14
p,p-DDE	0.18	Tetradifon	1.2
o,p DDT	4	Thiobencarb	5
o,p-DDD	0.4	Tebuconazole	7
o,p-DDE	3	Neburon	17
DDA	0.002	Chloroxuron	24
Chloropropylate	0.007	Monolinuron	25
		Diclofop	70

\* LLD = lower limit of detection

The calibration range to be used for this project is as follows:

<u>Calibration Range</u>	<u>Low</u>	<u>Medium</u>	<u>High (ppm)</u>
DDT	0.8	4.0	40.0

### 3.3.2 Immunochemistry Kit Data Quality Objectives

The performance requirements for the immunoassay tests are outlined in Table 3.

**Table 3**  
**Immunochemistry Kit Quantitative Data Quality Objectives**

<b>Compound</b>	<b>Matrix and Sample Type</b>	<b>Correlation with Definitive Analysis (RPD and r<sup>2</sup>)</b>	<b>Accuracy (LCS Recovery, %)</b>	<b>Precision (Duplicate RPD)</b>
DDT - Method 4042	Soil	≤50 > 0.90	60-140*	≤50
Cyclodienes - Method 4041	Soil	≤ 50 > 0.90	60-140*	≤ 50

\* Verification of analytical accuracy will be based on a mixed pesticide standard and a computed value based on the sensitivities for the reactivity groups given above. If the mean LCS recovery is not near 100%, further evaluation will be performed to assess the accuracy. Ultimately, the correlation of the site samples with the definitive analysis results will compensate for systematic differences (i.e. bias) and the LCS will primarily maintain a control on the batch-to-batch variability.

### **3.4 PERFORMANCE REQUIREMENTS FOR SOIL, WATER, AND TCLP DEFINITIVE ANALYSES**

#### **3.4.1 Soil Characterization and Cleanup Confirmation**

SW-846 Methods or equivalent documented methods will be used for all definitive confirmation sampling. Soxhlet extraction SW-846 Method 3540 or 3541, and appropriate cleanup as required by the interferences encountered will be used for all soil samples to be analyzed for organochlorine pesticides and organophosphorus pesticides. All pesticides listed on the quantitation limit tables will be reported by the laboratory. Modifications and equivalency of methods is described in Section 6.0 below.

#### **3.4.2 Waste Designation**

Soil samples to be collected for waste characterization analysis will be analyzed for total OC and OP pesticides (Methods 8081 and 8141), carbamates (8141 modified), and paraquat (SM-8-10) for State waste designation and extracted using the SW-846 TCLP extraction Method 1311. Waste sample TCLP extracts will be analyzed for all RCRA regulated organochlorine pesticides (Method 8081), and metals (Method 6010 and 7471) as appropriate.

A decontamination wastewater composite sample (and field blind duplicate) will be collected following completion of all site activities that could produce decontamination wastewater. The wastewater samples will be analyzed for organochlorine pesticides, organophosphorus pesticides, regulated metals, and total suspended solids. Quantitation limits for the methods selected must be below MTCA Method B groundwater screening values and Federal MCLs in order to decide whether water can be disposed on the ground.

#### **3.4.3 Quantitation Limits, Precision and Accuracy Goals**

Analytical quantitation limit requirements and quantitative data quality objectives are listed in Table 4. For the purpose of this project the method detection limit (MDL) will be defined as the lowest amount of an analyte that can be detected as defined in 40 CFR136 Appendix B. The quantitation limit will be no lower than the lowest non-zero standard in the calibration curve.

Quantitation limits for analyses must be below the MTCA Method B soil removal action level for excavation decisions and applicable federal and State Dangerous Waste Designation levels for waste designation decisions.

The accuracy, precision, completeness, and detection limit objectives for the analytical procedures are listed in Table 4. Data quality objectives will be met through adhering to required sampling methodology, required laboratory analytical methods, and data review. Data are accepted and rejected based on the data quality objectives. If the data are near the regulatory limit and could be affected by variability and accuracy measures, such as low recovery for spikes or surrogates, then further evaluation will be made. Audits will be initiated when data quality objectives are not being met.

Completeness is evaluated for each laboratory report in regard to the proportion of data qualified by the laboratory or Chemical Data Quality Manager (not to exceed 20% without corrective action). The completeness goal for unqualified data is therefore 80%. Some qualified data may be judged by the chemical Data Quality Manager as usable from the perspective of overall project objectives and decisions. The goal for usable data for this project is 98%, the high percentage reflecting the limited extent of the overall data set to be acquired.

Comparability will be measured using the RPD on single sample comparisons and through linear regression evaluation. Analytical confirmation sampling is being performed for the immunoassay test kits (IAA) during the focused removal and site characterization phases. In general, confirmation between two methods will be acceptable if a pair of results are within a relative percent difference (RPD) of 50%. If the concentration of one of the results is at or below the quantitation limit, the percent difference criteria are not appropriate and the pair of results will have to be evaluated on a case-by-case basis. Additionally, by plotting the pairs of results as a scatter plot and applying linear regression, the average performance of the confirmational analysis can be established. The data quality objective for the coefficient of regression is as follows:

$$R^2 > 0.9$$

Correlation anomalies serve to initiate investigation of sources for the differences observed and do not necessarily lead to data qualification. Any systematic bias discovered for application of the IAA at this site will be used to re-align the excavation plan in order to successfully remove contamination above the clean-up standards based on the extensive IAA characterization data set. The realignment of the IAA action levels is described in the Field Sampling Plan.

Field blanks will be taken during confirmation sampling to monitor sampling equipment cleanliness. The objective for all field blanks is to be below detection for the particular analytical method.

Representativeness of the analytical results will be highly dependent on the sampling procedures (discussed in the Field Sampling Plan). Field duplicates and laboratory matrix spike duplicates are analyzed to monitor the performance of the sampling and analytical processes. Comparability of these data is controlled by using standard test procedures and carefully evaluating modifications applied. Blind performance evaluation samples will be used to further evaluate comparability of results.

**Table 4**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/ Quantitation Limit
IAA - Cyclodienes	100 ug/kg	EPA 4041	EPA 4041	Sample spikes not applicable.	<20%	NA	<50	100%	40 ug/kg
IAA - DDT	5000 ug/kg	EPA 4042	EPA 4042	Sample spikes not applicable.	<20%	NA	<50	100%	800 ug/kg
<b>OC Pesticides</b>									ug/kg
Aldrin	5.9X10 <sup>-2</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	.1/5
α-BHC	7.7X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
β-BHC	7.7X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
δ-BHC	7.7X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
γ-BHC (Lindane)	7.7X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	5/20
Chlordane (technical)	6.7X10 <sup>-3</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	2/10
4,4'-DDD	4.2X10 <sup>0</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	5/20
4,4'-DDE	2.9X10 <sup>0</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	5/20
4,4'-DDT	2.9X10 <sup>0</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	5/20
2,4'-DDD *	4.2X10 <sup>0</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	5/20

**Table 4 (Continued)**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/Quantitation Limit
2,4'-DDE *	2.9X10 <sup>0</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130-	<30%	98%	5/20
2,4'-DDT *	2.9X10 <sup>0</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
Dieldrin	6.3X10 <sup>-2</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	.1/1
Endosulfan I	4.8X10 <sup>2</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	.5/2
Endosulfan II	4.8X10 <sup>2</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	.5/2
Endosulfan Sulfate	4.8X10 <sup>2</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	.5/2
Endrin	4X10 <sup>-1</sup> mg/kg <sup>α</sup>	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	.2/1
Endrin Aldehyde	2.4X10 <sup>1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
Heptachlor	2.2X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	60-140	<30%	75-130	<30%	98%	5/20
Heptachlor Epoxide	1.1X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
Methoxychlor	4X10 <sup>2</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
Toxaphene	9.1X10 <sup>-1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
Endrin Ketone	2.4X10 <sup>1</sup> mg/kg	3540B/3620 A	8081A	-	<30%	-	<30%	98%	5/20
<b>Surrogates:</b>									
DCBP		3540B/3620	8081A	65-135	<30%	65-135	<30%		

**Table 4 (Continued)**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/Quantitation Limit
		A							
TCMX		3540B/3620 A	8081A	65-135	<30%	65-135	<30%		
<b>OP Pesticides</b>									ug/kg
Azinphos Methyl	3.2X10 <sup>0</sup> mg/kg	EPA 3540	8141	-	<30%	-	<30%	98%	100/200
Diazinon	7.2X10 <sup>1</sup> mg/kg	EPA 3540	8141	-	<30%	-	<30%	98%	100/200
Dichlorovos	3.4X10 <sup>0</sup> mg/kg	EPA 3540	8141	-	<30%	-	<30%	98%	100/200
Disulfoton	3.2X10 <sup>0</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
Parathion Methyl	2X10 <sup>1</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
Parathion	4.8X10 <sup>2</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
Paraoxon-ethyl *	4.8X10 <sup>2</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
Paraoxon-methyl *	2X10 <sup>1</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
dimethoate	1.6X10 <sup>1</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
ethion *	4X10 <sup>1</sup> mg/kg	EPA 3540	8141	-	<30%	-	<30%	98%	100/200
malathion	1.6X10 <sup>3</sup> mg/kg	EPA 3540	8141	60-140	<30%	75-130	<30%	98%	100/200
<b>Surrogate: Tributyl Phosphate or Triphenyl Phosphate</b>		EPA 3540	8141	65-135	<30%	65-135	<30%	98%	
<b>Carbamates/ MTCA Method B Standard</b>									ug/kg
cabofuran	4X10 <sup>2</sup> mg/kg	EPA 3540	8141 mod.	60-140	<30%	60-140	<30%	98%	1000/5000
carbaryl	8X10 <sup>3</sup> mg/kg	EPA 3540	8141 mod.	60-140	<30%	60-140	<30%	98%	1000/5000
<b>Paraquat/ MTCA Method B Standard</b>									ug/kg
paraquat	3.6X10 <sup>2</sup> mg/kg	EPA 3540	8321 mod.	60-140	<30%	60-140	<30%		1000/5000

**Table 4 (Continued)**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/Quantitation Limit
<b>TCLP (mg/L in Extract)</b>									mg/L
Endrin/	2X10 <sup>-2</sup> mg/L	1311/3510 B	8081A	60-140	<30%	75-130	< 50%	98%	0.0001/0.001
Gamma BHC (lindane)	4X10 <sup>-1</sup> mg/L	1311/3510B	8081A	60-140	<30%	75-130	<50%	98%	0.01/0.1
Arsenic	5 mg/L	1311/3010	6010	80-120	<30%	80-120	<50%	98%	0.2/1
Selenium	1 mg/L	1311/3010	6010	80-120	<30%	80-120	< 50%	98%	0.1/0.5
Barium	100 mg/L	1311/3010	6010	80-120	<30%	80-120	<50%	98%	1/5
Cadmium	1 mg/L	1311/3010	6010	80-120	<30%	80-120	<50%	98%	0.1.0.5
Chromium	5 mg/L	1311/3010	6010	80-120	<30%	80-120	< 50%	98%	0.2/1
Lead	5 mg/L	1311/3010	6010	80-120	<30%	80-120	<50%	98%	0.2/1
Silver	5 mg/L	1311/3010	6010	80-120	<30%	80-120	< 50%	98%	0.2/1
Mercury	0.2 mg/L	1311/3010	6010	80-120	<30%	80-120	<50%	98%	0.01/0.05
<b>Wastewater Analysis</b>									ug/L
Arsenic	5X10 <sup>0</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	<50%	98%	.2/1
Antimony	6.4X10 <sup>0</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.2/1
Selenium	8X10 <sup>1</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.5/2
Beryllium	8X10 <sup>1</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	<50%	98%	.1/.5
Cadmium	5X10 <sup>0</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.1/.5
Chromium	5X10 <sup>1</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.2/1
Copper	5.9X10 <sup>2</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	<50%	98%	.1/.5
Lead	5X10 <sup>0</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.1/.5
Thallium	1.1X10 <sup>0</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	<50%	98%	.1/.5
Nickel	3.2X10 <sup>2</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.1/.5
Silver	8X10 <sup>1</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	<50%	98%	.1/.5
Zinc	4.8X10 <sup>3</sup> ug/L	EPA 3010	EPA 6020	80-120	<30%	80-120	< 50%	98%	.1/.5

**Table 4 (Continued)**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/ Quantitation Limit
Mercury	2X10 <sup>0</sup> ug/L	EPA 7471	EPA 7471	80-120	<30%	80-120	<50%	98%	.5/2
Total Suspended Solids	NA	NA	EPA 160	see LCS	<30%	40-160	< 50%	98%	1/10 mg/L
<u>OC Pesticides</u>									ug/L
Aldrin		3510B/3620 A	8081A	60-140	<30%	75-130	<50%	98%	0.02/0.1
α-BHC		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
β-BHC		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
δ-BHC		3510B/3620 A	8081A	-	<30%	-	<50%	98%	0.02/0.1
γ-BHC (Lindane)		3510B/3620 A	8081A	60-140	<30%	75-130	< 50%	98%	0.02/0.1
Chlordane (technical)		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
4,4'-DDD		3510B/3620 A	8081A	60-140	<30%	75-130	<50%	98%	0.02/0.1
4,4'-DDE		3510B/3620 A	8081A	60-140	<30%	75-130	< 50%	98%	0.02/0.1
4,4'-DDT		3510B/3620 A	8081A	60-140-	<30%	75-130	<50%	98%	0.02/0.1
2,4'-DDD *		3510B/3620 A	8081A	60-140	<30%	75-130	< 50%	98%	0.02/0.1
2,4'-DDE *		3510B/3620 A	8081A	60-140	<30%	75-130	<50%	98%	0.02/0.1
2,4'-DDT *		3510B/3620 A	8081A	60-140	<30%	75-130	<50%	98%	0.02/0.1
Dieldrin		3510B/3620	8081A	60-140	<30%	75-130	< 50%	98%	0.02/0.1

**Table 4 (Continued)**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/Quantitation Limit
		A							
Endosulfan I		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
Endosulfan II		3510B/3620 A	8081A	-	<30%	-	<50%	98%	0.02/0.1
Endosulfan Sulfate		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
Endrin		3510B/3620 A	8081A	60-140	<30%	75-130	< 50%	98%	0.02/0.1
Endrin Aldehyde		3510B/3620 A	8081A	-	<30%	-	<50%	98%	0.02/0.1
Heptachlor		3510B/3620 A	8081A	60-140	<30%	75-130	< 50%	98%	0.02/0.1
Heptachlor Epoxide		3510B/3620 A	8081A	-	<30%	-	<50%	98%	0.02/0.1
Methoxychlor		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
Toxaphene		3510B/3620 A	8081A	-	<30%	-	<50%	98%	0.02/0.1
Endrin Ketone		3510B/3620 A	8081A	-	<30%	-	< 50%	98%	0.02/0.1
<b>Surrogates:</b>									
DCBP		3510B/3620 A	8081A	65-135	<30%	65-135	<50%	98%	
TCMX		3510B/3620 A	8081A	65-135	<30%	65-135	<50%	98%	
<u>OP Pesticides</u>									ug/L

**Table 4 (Continued)**  
**Data Quality Objectives and Analytical Methods**

Parameter	Cleanup Std. or Regulatory Limit	Extraction /Cleanup Method**	Analytical Method	Measurement Accuracy (% Recovery)	Measurement Precision (% RPD)	LCS Accuracy (% Recovery)	Field Duplicate Precision (% RPD)	Completeness (%)	Detection Limit/Quantitation Limit
Azinphos Methyl		3510B	8141	-	<30%	-	<50%	98%	1/5
Diazinon		3510B	8141	-	<30%	-	< 50%	98%	1/5
Dichlorovos		3510B	8141	-	<30%	-	< 50%	98%	1/5
Disulfoton		3510B	8141	60-140	<30%	75-130	<50%	98%	1/5
Parathion Methyl		3510B	8141	60-140	<30%	75-130	< 50%	98%	1/5
Parathion		3510B	8141	60-140	<30%	75-130	< 50%	98%	1/5
Paraoxon-ethyl *		3510B	8141	60-140	<30%	75-130	<50%	98%	1/5
Paraoxon-methyl *		3510B	8141	60-140	<30%	75-130	< 50%	98%	1/5
dimethoate		3510B	8141	60-140	<30%	75-130	<50%	98%	1/5
ethion *		3510B	8141	-	<30%	-	< 50%	98%	1/5
malathion		3510B	8141	60-140	<30%	75-130	<50%	98%	1/5
<b>Surrogate:</b> Tributyl Phosphate or Triphenyl Phosphate		3510B	8141	65-135	<30%	65-135	<50%	98%	

\* Compound not on the standard analyte list of Method 8141 or 8081.

\*\* Any other cleanup procedure required by interferences encountered will be used.

- Indicates a compound not included in the spike solution.

α This estimated waste designation threshold (20 times the TCLP limit) is lower than the MTCA Method B limit.

## **4. SAMPLING LOCATIONS AND PROCEDURES**

### **4.1 SAMPLING LOCATIONS**

Section 3 of the Field Sampling Plan describes the rationale for the sampling locations.

### **4.2 SAMPLING PROCEDURES**

Sampling procedures are specified in Section 4 of the Field Sampling Plan.

### **4.3 SAMPLING DURATION**

The project sampling activities will occur over a period of 20 - 30 days. Expedited analysis will be utilized to compress the decision cycle for moving the project forward.

## **5. SAMPLE CUSTODY AND HOLDING TIMES**

The sample identifiers are described in Section 5.3 of the Field Sampling Plan. Project phase designation, sampling location, and sampling sequence are encoded in the sample numbers. These identifiers are recorded in field notebook or sampling sheets and on sample tags/labels and Chain-of-Custody forms. Identifiers on sample labels include: site name, sampling location (station), date of collection and time of day, name of sampler, analysis requested, sample type (matrix), and preservation requirements.

Field sample custody is described in Section 5.6 of the Field Sampling Plan. The laboratory sample receipt, custody, sample identification, sequencing, and tracking procedures are described in detail in the Laboratory QA/QC Plans. Any problems found with samples at the time of receipt or during analysis will be documented and resolution sought from the Chemical Data Quality Manager. See "Corrective Action" for documentation of problems. Example Chain-of-Custody forms, seals, and corrective action forms are provided in Appendix A.

The holding times that affect the laboratories are provided in Table 5.

**Table 5**  
**Summary of Sampling Containers, Preservation and Holding Time Criteria**

Analyte	Sample Type	Preservation	Holding Time	Turn-around Time	Container Type
IAA	Soil	4°C	14 days to extraction	24 hrs.	4 oz wide mouth
OP and OC Pesticides	Soil	4°C	14 days to extraction/40 days until analysis	72 hrs	8 oz wide mouth
Carbamate and Paraquat	Soil	4°C	14 days to extraction/40 days until analysis	72 hrs	8 oz wide mouth
TCLP OC Pesticides/	Soil	4°C	14 days to TCLP leaching, see water methods for definitive analytical holding times	72 hrs	8 oz wide mouth
TCLP Metals	Soil	4°C	28 days TCLP leaching; 28 days to analysis.	72 hrs	8 oz wide mouth
OP and OC Pesticides	Water	4°C	7 days to extraction/ 40 days to analysis	72 hrs	1 L, amber glass jar.
Metals	Water	4°C, pre-preserved with HNO <sub>3</sub> pH<2	6 months to analysis	72 hrs	250 mL HDPE
TSS	Water	4°C	7 days	72 hrs	500 mL HDPE or glass

## 6. ANALYTICAL PROCEDURES

Fixed-laboratory procedures and IAA techniques have been established by the EPA that are capable of meeting the project objectives. Refer to Table 4 for method references to the sample preparation and analytical methods. Refer to Appendix B for IAA and analytical SOPs to be used for this project.

In addition to the specific methods referenced, various sections of SW-846 contain specifications that apply to the methods for this project. General gas chromatography method requirements are outlined in Method 8000. Chapters 3 and 4 of SW-846 describe specific sample handling requirements for metals and organics, respectively.

The IAA tests have been modified slightly to make a single soil extraction serve for both tests. The GC/MS instrumentation will be used as the primary quantitative and qualitative technique for Methods 8081A and 8141. All modifications to the EPA reference methods are outlined in Table 6.

Each laboratory applies modifications to methods to improve performance or meet project-specific requirements. A summary of the modifications noted by the laboratories for each method is outlined in Table 6.

The organophosphorus analysis is going to be analyzed by GC/MS by the primary laboratory. The SOP describes the method as a modification of EPA 8141. The organochlorine pesticides will be analyzed by GC so that quantitation for multicomponent pesticides (technical chlordane and toxaphene) can be provided. All of the other target list organochlorine pesticides can be confirmed by the GC/MS results as well as the second column analysis specified in Method 8081.

The carbamates will be analyzed by GC with an NPD detector rather than the HPLC method recommended.

The detection limits and/or quantitation limits and analytical QC criteria are given in Table 4. More stringent criteria based on statistical evaluation or laboratory practice may be used by the laboratory. In such instances the laboratory-specific criteria will be used for data validation purposes as long as the criteria are more stringent than the targets set for this project.

Liquid standards are prepared by dilution of commercial standards. Standards and sample extracts are stored at 4° C in separate refrigerators/freezers. Other QA/QC performance options used by the laboratory, including use of QC charts and system audits, are described in detail in the Laboratory SOPs (Appendix C) and the Laboratory QA plans (submitted separately as validation documents).

**Table 6**  
**Modifications to Standard Methods**

<b>SOP</b>	<b>Method/Sec. #</b>	<b>Modification/Justification</b>
Cyclodiene IAA Test	EPA 4041	Extraction fluid will be pure methanol rather than water/methanol mix. This makes the test compatible with the DDT test, allowing for a single sample extraction for both tests.
DDT and Cyclodiene IAA tests.	EPA 4042 and 4041	The extractant volume will be doubled to 20 mL to better bracket the action levels for these tests based on the pilot study cross-sensitivity results. If anything, increasing the volume will improve extraction efficiency. Specific changes to the procedures provided by Strategic Diagnostics Inc. are outlined in a cover sheet to the IAA SOPs in the Appendix.
OP Pesticides	EPA Method 8141	GC/MS rather than GC/NPD is used. The surrogates and calibration requirements appropriate for this method are utilized from the source method (8141). The modification improves selectivity and maintains low enough quantitation limits to meet the project DQOs. The Continuing Calibration control limit will be 80-120%. The broader limits compensates for the GC/MS variability relative to GC.
Organophosphorus Compounds by GC modified for Carbamates (CARBNPD.DOC)	EPA 8141, mod.	GC/NPD is used as directed in EPA Method 632, modified for a soil matrix according to the SW-846 methods. The moderate project detection limit requirements and restricted analyte list allows the less sensitive but more selective GC/NPD technique to be used instead of HPLC (EPA Method 8321). The benefits will primarily be improved performance due to reduction of interferences.
Carbamates	EPA 8141, mod.	Surrogate selection is sulprofos. This pesticide has been chosen as a surrogate since the compound is rarely used in this geographical area.
Carbamates	EPA 8141, mod.	Column selection of DB35 and XLB. We have found that this selection of columns results in a reduced number of coeluting compounds.
Paraquat	RM-8-10.	This spectrophotometric method accommodates paraquat in a soil matrix according to procedures developed by Chevron Oil.

## 7. CALIBRATION PROCEDURES AND FREQUENCY

### 7.1 CALIBRATION FOR IMMUNOASSAY ANALYSES (IAA)

Calibration for the IAA tests is established by the manufacturer of the test kit. Calibration verification is performed with each batch of 12 samples. This frequent verification is necessary to provide quality control over the variables (e.g., temperature, reagents, and analyst technique) that affect the rate of reactions in the tests. Each test batch will be preceded with a set of three standards (“calibrators”), in addition to a blank (‘negative control’). The calibrator concentrations will be as follows:

Cyclodiene Test: Equivalent to 18, 86, and 510 ug/kg of soil (brackets the 100 ug/kg action level).

DDT Test: Equivalent to 800, 4000, and 40000 ug/kg of soil (brackets the 5000 ug/kg action level).

These equivalent concentrations are achieved by diluting all sample extracts by a factor of 2 (i.e., the extraction volume has been doubled as compared to the standard test procedure).

If consistency of the duplicate calibrators and duplicate samples appears to be adequate and the analytical conditions prove to be predictable, the duplicate standard pairs will be reduced to single standards, thereby increasing the sample throughput to 15 samples per batch.

Acceptable calibration is determined by the following:

1. The absorbance of the calibrators is within 0.3 absorbance units of the predicted curve.
2. The absorbance of the middle calibrator is one-half of the absorbance of the negative control ( $0.5 \pm 0.2$ ).
3. The duplicates are within 0.3 absorbance units of each other. Duplicate calibrator control may also be evaluated on the basis of relative percent difference (RPD), with an acceptance level at 30%.

### 7.2 CALIBRATION FOR GC OR GC/MS ANALYSIS

For GC and GC/MS analysis, five point calibration (Initial Calibration) is performed periodically based on method performance and must meet the method linearity criteria for the analytes of interest for this project. GC/MS methods for this project will utilize internal standards. The quantitation limits set for this project (Table 4) define the lowest standard in the calibration (i.e., all calibration curves will include a standard at or below the required quantitation limit). Analytical results below the lowest standard will be flagged by the laboratory as estimates.

The calibrations for all GC methods for this project are checked daily with a continuing calibration verification (CCV) sample prior to running samples for each batch. The CCV results must be within the criteria established for the corresponding method SOP (equal to or more stringent than the source method).

The calibration of the GC/MS is outlined in detail in the SOPs and summarized in the table below. The GC/MS detector is tuned every twelve hours with DFTPP as per EPA Method 8270. Three internal standard compounds are added to samples and all calibration standards. A five point calibration (Initial Calibration) is performed and must meet linearity criteria for the Calibration Check Compounds (relative standard deviation (RSD) for relative response factors across the calibration concentration range). The Calibration Check Compound (CCC) list for this project includes the entire project target list.

On the day of analysis, a continuing calibration verification (CCV) is performed at the beginning of the 12-hour period during which the instrument tune is valid. For this CCV sample, the performance for

problem-sensitive compounds (SPCC) are checked against the SW-846 Method criteria. Also, the CCC compounds are evaluated to determine if the calibration is still valid. The CCV results must be within the percent difference (%D) criteria established for the corresponding method SOP (equal to or more stringent than the source method) for analytes of interest. If any of the analytes that are found in the samples do not meet the continuing calibration criteria at the start of the analytical batch, the Chemical Data Quality Manager will be notified immediately. If appropriate, the samples containing the non-compliant analytes will be reanalyzed at no cost to GSA or the Government.

Method	Tune	%RSD of RRF	%D for CCV
GC/MS	As per SW-846	<20% RSD	<20%
GC Methods	NA	<20% RSD or correlation coefficient >0.995	<15%

Samples will be re-analyzed after problems have been corrected when a CCV fails for analytes found in the samples or when originally analyzed outside of the 12 hour tune window. If the %D for an analyte in the CCV indicates abnormally low response for a compound not found in the samples, the case narrative will note the deficiency. If SPCC criteria cannot be met or CCV criteria repeatedly cannot be met, the initial calibration will be performed and affected samples will be reanalyzed.

### 7.3 CALIBRATION FOR METALS

For ICP, calibration on a given day is established with a single high level standard containing all elements of interest (“mixed standard”) and a blank. Once every three months the linear range for individual elements is established, allowing a much higher calibration range than can be established with the mixed standards. Mercury analysis calibration is performed on the day of analysis utilizing the same reagent batches as the samples analyzed. If the interference check sample, calibration verification, or calibration blank is outside of control limits, the entire analytical batch will be re-analyzed.

### 7.4 CALIBRATION FOR SPECTROPHOTOMETRIC METHODS

Calibration is established with a minimum of 3 standards over the range of interest and a blank. On the day of analysis a midrange standard is run to verify the curve. The calibration verification sample is repeated every 10 samples in the analytical sequence with an acceptance limit of 80-120%.

## 8. INTERNAL QA/QC CHECKS

Both IAA and laboratory QC samples are outlined in Table 7. The QC Duplicates are split field samples used to evaluate laboratory performance. The QC samples will be taken for every 10 samples collected per matrix and method.

Equipment blanks will be utilized for characterization and confirmation sampling at a minimum rate of one per sampling event to confirm adequate equipment decontamination.

**Table 7**  
**Summary of QC**

Analytical Parameter	Method	Sample Type	No. Field Samples	No. Field Duplicates	No. Equip. Blank	Matrix Spike (Lab)	Matrix Spike Duplicates (Lab)	Lab Control Sample (Lab)	Blanks (Lab)
<b>Focused Removal</b>									
OC and OP Pesticides	GC/MS and GC	Soil	6	1	1 per day	1 per batch	1 per batch	1 per batch	1 per batch
<b>Characterization</b>									
IAA	4041/4042	Soil	162	16	0	NA	1 dup./batch	1 per batch	1 per batch
OC and OP Pesticides,	GC/MS and GC	Soil	162 (36 analyzed)	16 (4)	1 per day	1 per batch	1 per batch	1 per batch	1 per batch
<b>Final Confirmation</b>									
IAA	4041/4042	Soil	27	3	0	NA	1 dup./batch	1 per batch	1 per batch
OC and OP Pesticides, Carbamate pesticides,	GC/MS and GC	Soil	27 (9 analyzed)	3 (1)	1 per day	1 per batch	1 per batch	1 per batch	1 per batch
Paraquat	Spectrometric	Soil	27 (9 analyzed)	3 (1)	1 per day	1 per batch	1 per batch	1 per batch	1 per batch
<b>Waste Profile</b>									
Prelim OC, OP	GC and GC/MS	Soil	6	1	0	1 per batch	1 per batch	1 per batch	1 per batch
Final OC, OP	GC and GC/MS	Soil	3 (est)	(taken above)	0	1 per batch	1 per batch	1 per batch	1 per batch
Carbamate Pesticides	GC	Soil	1	1	0	1 per batch	1 per batch	1 per batch	1 per batch
Paraquat	Spectrometric	Soil	1	1	0	1 per batch	1 per batch	1 per batch	1 per batch
OC Pesticides	GC	TCLP extract	3	1	0	1 per batch	1 per batch	1 per batch	1 per batch
Metals	3010/6010	TCLP extract	5	1	0	1 per batch	1 per batch	1 per batch	1 per batch
<b>Wastewater</b>									
OC and OP Pesticides,	GC/MS and GC	Water	1	1	0	1 per batch	1 per batch	1 per batch	1 per batch
Metals	ICP/MS and	Water	1	1	0	1 per batch	1 per batch	1 per batch	1 per batch
Total Suspended Solids (TSS)	Gravimetric	Water	1	1	0	NA	1 dup. per batch	1 per batch	1 per batch

## 8.1 GENERAL

The quality control procedures for the preparation of soil samples for all methods include preparation blanks, matrix spikes, laboratory duplicates or matrix spike duplicates, laboratory control samples, and surrogate spiking (for organics analysis). The recovery requirements for these data are provided in Table 4. The analyses by all methods will be accompanied by method quality control including a batch laboratory control spike (LCS) with representative compounds. The compounds and limits used are given in Table 4. LCS standards must be from a source independent of the calibration standards.

## 8.2 GC AND GC/MS ANALYSES

The QC frequency for the GC analyses is based on a daily (24 hour) sequence or twenty samples, whichever comes first. The QC frequency for GC/MS is based on a 12 hour instrument tune sequence. The QC frequency for metals analysis is based on the preparation batch for project QC and a 20-sample analytical batch for the instrument.

The analytical QC for GC and GC/MS following calibration of each instrument includes performance of a method blank (one per instrument per day minimum) as well as the calibration verification noted in the section above. In addition, the DDT breakdown standards will be as specified in EPA Method 8081.

### 8.2.1 Other Method 8081 QC Parameters

- Confirmation for all single component analytes: %RPD < or = 40% for second column.
- Laboratory Blanks: < MDLs for all analytes.
- Initial Calibration: %RSD  $\leq$  20% for all analytes or  $R^2 >$  or = 0.995.
- Continuing Calibration (CCV): %D < +/- 15% for all analytes.
- Laboratory Control Sample (Second Source Standard) - analytes per Table 4, limits 75-130%.
- At least two surrogates must be added to all environmental and QC samples. The maximum acceptable recovery range for surrogates is 65%-135%.
- Perform % Breakdown checks as per Method 8081 at the beginning of the analytical shift and every 12 hours of analyses. The % Breakdown checks must be performed before the initial calibration or any CCV. The % Breakdown for either DDT or Endrin must be  $\leq$  15%.

### 8.2.2 Other Method 8141 QC Parameters

- Confirmation for all single component analytes : %RPD < or = 40% for second column if GC is used.
- Laboratory Blanks: < MDLs for all analytes.
- Initial Calibration: %RSD  $\leq$  20% for all analytes or  $r^2 >$  or = 0.995.
- Continuing Calibration: CCV %R: < +/- 20% for all analytes for GC/MS or < +/- 15% for GC.
- Laboratory Control Sample (Second Source Standard) - analytes per Table 4, limits 75-130%.

### 8.2.3 Other QC Parameters for Carbamates

- Laboratory Blanks: < QL for all analytes.
- Initial Calibration: : %RSD  $\leq$  20% for all analytes or  $R^2 >$  or = 0.995 for all analytes.
- Continuing Calibration: %D < +/- 15% for all analytes.
- Laboratory Control Sample (Second Source Standard): Recovery % 75-130.

### 8.3 METALS

Analytical QC specific to ICP analysis includes Interference Check Samples at the beginning and end of the analytical run and serial dilutions as needed to verify freedom from matrix effects.

### 8.4 SPECTROPHOTOMETRIC METHODS (PARAQUAT)

The extraction procedure and overall analysis will be verified with a method blank and laboratory control sample for each analytical batch of 20 or fewer samples, with recovery limits of 75-130%. Other QC parameters for paraquat are as follows:

- Laboratory Blanks: < QL for all analytes.
- Initial Calibration: : %RSD ≤20% for all analytes or  $R^2 > \text{or} = 0.995$  for all analytes.
- Continuing Calibration: %D < +/- 20% for all analytes.
- Laboratory Control Sample (Second Source Standard): Recovery % 75-130.

### 8.5 PERFORMANCE EVALUATION SAMPLES

Performance evaluation (PE) samples will be purchased from Environmental Resource Associates, Arvada, Colorado, that can provide statistically derived certified soil reference samples. The PE samples should contain the site-specific primary POCs. The organochlorine PE sample will include: DDE, DDT, DDD, Dieldrin, and Endrin. The organophosphorus PE sample will include: Disulfoton, Dimethoate, Guthion, Parathion. These analytes should be contained in the PE samples at the site-specific health based action levels for each target compound. All PE samples will be containerized and labeled so that they are blind to the laboratory. Throughout the duration of the project, 4 PE samples per analysis type will be submitted to the fixed laboratory. At least 2 of the PE samples will be submitted during the first week of analysis so that GSA can assess whether the laboratories are functioning within acceptable control limits early in the sampling effort. PE sample results will immediately be compared against the vendor's documented acceptable control limits. If the laboratory does not meet certified PE sample acceptance limits, project investigative sample analysis will not continue until an evaluation of whether required corrective action has been completed and any actions deemed necessary have been implemented. GSA will supply the Corps QA Representative with PE sample results and PE sample vendor control limit documentation within 12 hours following receipt of the PE sample results from the fixed laboratory. The results of the PE sample analyses and any data quality issues associated with the results will be summarized in the summary chemical data quality reports.

The PE samples will come fully prepared from the vendor. Standard sample labels and labeling that appear to be site samples will be applied to the PE samples by GSA.

## 9. CALCULATION OF DATA QUALITY INDICATORS

### 9.1 PRECISION

Analytical precision is measured by determining the relative percent difference (RPD) between duplicate samples. The equation for relative percent difference is shown below:

$$\% \text{ RPD} = \left( \frac{\text{Sample 1} - \text{Sample 2}}{(\text{Sample 1} + \text{Sample 2}) / 2} \right) \times 100$$

## 9.2 ACCURACY

Analytical accuracy is measured by determining the percent recovery of known method or matrix spikes. Surrogates are also evaluated for each sample to provide support for the accuracy of organics analysis. The equation for percent recovery is shown below:

$$\% \text{ Recovery} = \left( \frac{\text{Amount of spiked sample} - \text{Amount of sample before spike}}{\text{Amount of spike}} \right) \times 100$$

## 9.3 COMPLETENESS

Completeness is evaluated by dividing the number of unqualified analytical results by the total number of results attempted. A distinction is made between completeness goals for usable data as compared to unqualified data. A 98% completeness target for usable data would indicate that only 2% missing data, or data qualified as unusable, can be tolerated for the particular data use. An 80% completeness goal for unqualified data indicates that up to 20% of the results in a data set may be qualified with a data defect, but not qualified as unusable.

$$\% \text{ Complete} = \left( \frac{\text{Unqualified analytical results}}{\text{Total number of results attempted}} \right) \times 100$$

For usable data completeness, “unqualified analytical results” refers to the total of usable data. For quality data completeness, “unqualified analytical results” refers to all data not associated with a quality control defect.

## 10. CORRECTIVE ACTIONS

Corrective actions in the field relate to inspections of equipment, procedures, and problems found during data review, as outlined in Section 7 of the Field Sampling Plan. Appendix A contains standard forms used to transmit a record of corrective actions at various levels of the project.

Corrective actions will be taken in the laboratory if method-specific QC or project-specific Data Quality Objectives (DQOs) are not met and as the result of problems found during data review.

When corrective action is initiated, the source of the problem must be investigated and appropriate corrective measures taken and documented before further analysis proceeds. The laboratory manager must address problems and solutions in the analytical report narrative.

### 10.1 INCOMING SAMPLES

The laboratory will inspect incoming samples to make sure that sample identification, preservation and chain of custody were reliably carried out as outlined in the Field Sampling Plan. Documentation and sample condition are considered. The GSA Chemical Data Quality Manager will be consulted for any problems not meeting the requirements of Table 5 above or the laboratory SOP or QA plan. GSA may require resampling or reanalysis for critical samples.

### 10.2 SAMPLE HOLDING TIMES

The GSA Chemical Data Quality Manager will be contacted by the laboratory prior to analysis of any samples exceeding the holding times. Resampling will be performed if the Chemical Data Quality Manager deems the sample to be critical.

### **10.3 INSTRUMENT CALIBRATION**

All sample results associated with calibrations which do not meet the requirements of the method for the analytes of interest will not be reported by the laboratory. The instrument will be recalibrated and/or demonstrated in control prior to the samples being reanalyzed. The results for reanalysis will be reported.

### **10.4 METHOD QC**

All method QC, including blanks, matrix spikes, matrix spike duplicates or duplicates, surrogate recoveries, laboratory control samples and other method-specific QC will meet the method-specified requirements and project-specific DQOs, or else be subject to corrective action within the laboratory. Failure of method-required QC will result in the review by the laboratory of all potentially affected data. If no errors or assignable cause can be found, the sample or entire batch may need to be reanalyzed within holding times with appropriate QC to demonstrate method control. When matrix spikes or duplicates are out of control, laboratory control sample results will be reported to demonstrate method control. Assignment of a cause that does not result in re-extraction and/or re-analysis must be fully justified in the report narrative.

### **10.5 DETECTION, DILUTIONS, AND BLANK CONTAMINATION**

The GSA Chemical Data Quality Manager will be consulted by the laboratory if dilutions or blank contamination prevent the requirements of Table 4 from being met. If the blank exceeds the project quantitation limits, the analytical system is considered out of control. Blank results will not be subtracted from analytical results. Reanalysis or resampling may be performed depending on the nature of the problem encountered and the level of importance of the particular sample. Resampling may be performed if reanalysis cannot be performed within the holding times. During data validation/usability assessment, blank contamination (either laboratory or field) may be grounds for qualifying certain results as undetected, in accordance with the EPA Functional Guidelines.

### **10.6 DATA INTERPRETATION**

During data review, the GSA Chemical Data Quality Manager may encounter results which do not correlate well with expectations, with other results, and with results from other methods performed on the same samples. Such situations may trigger inquiries into raw data, such as chromatograms, that are not normally reported with the results. These data must be provided by the laboratory for review at no additional cost to GSA or the Government.

If field duplicates or laboratory duplicates do not show acceptable precision, problems with obtaining homogeneous or representative samples are suspected. Procedures and records will be reviewed. If the data are near the regulatory limit, resampling may be necessary.

### **10.7 NOTIFICATION OF NONCOMPLIANCE**

The Chemical Data Quality Manager will be notified of any detected noncompliance with the foregoing requirements. Data reports will include a narrative discussing all problems found and the course of resolution.

## **11. DATA REDUCTION, VALIDATION, AND REPORTING**

### **11.1 DATA REDUCTION**

The Laboratory QA Plan outlines data reduction and review procedures that provide a check on data transcription, calculations, and sensibility.

Data reduction for IAA analysis includes computation of concentration from the response curves provided by the kit manufacturer (verified with each batch)

Adjustment of IAA action levels may be applied after the correlation between IAA and fixed laboratory analysis is established. GSA will establish the action levels based on the slope of response ( $S_{IL}$ ) when IAA results are plotted against the concentrations for the reactivity group for the particular IAA test. The reactivity group is the pesticides to which a particular IAA test is sensitive (see Section 3.3.1 above for the reactivity groups). If a systematic bias is established (slope different from 1), then the particular IAA action level can be recalibrated multiplying the action level by  $S_{IL}$ .

### **11.2 DATA REVIEW**

#### **11.2.1 Laboratory Data**

The laboratory data review covers transcriptions, computations, dilutions, and QC results. The data is reviewed in the laboratory and at the project level. The full data package with QC results allows extensive review if deemed necessary. The review of data packages will include an evaluation of the information provided on the analytical data sheets and required support documentation for all sample analysis, and the supporting sample collection documentation, including chain-of-custody. The QA review will also examine adherence to the procedures as described in the requested analytical methods.

#### **11.2.2 On-site Measurement Data**

On-site data review follows the data entry process prompted by field sampling sheets and as required by the Field Sampling Plan. On-site data review covers transcriptions, computations, and QC results. The data is reviewed by the field chemist, Chemical Data Quality Control Manager. Data will not be released without approval by the Chemical Data Quality Manager and the site Quality Control Manager. The review of field data will include an evaluation of the information provided in the field notebook and required support documentation for all sample analysis documented on field sampling sheets, including chain-of-custody; and field instrument calibration and/or performance check documentation (if required by the SOP).

### **11.3 DATA REPORTING**

Data output for this analysis includes sample results calculated on a dry weight basis in mg/kg (ppm), percent moisture, and quality control data for blanks, spikes, duplicates, and surrogates.

Data reporting will be in a clear format and organized as follows (from EM 200-1-3, 1 Sept. 94):

- A general discussion of the sample types received, tests performed, problems encountered, and general comments, along with a table of sample data and any failed QC parameters.
- Analytical data, presented by sample number or by test.
- Calibration verification information.

- Laboratory performance and matrix-specific information including surrogates, matrix spike results, laboratory control samples, laboratory duplicate results, pesticide breakdown assessment, clean-up QC check sample results.
- Any other pertinent information, including cooler receipt forms and corrective action forms, for example.

The laboratory data output for GC and GC/MS and metals will be in a format comparable to that of routine analytical services for the EPA Contract Laboratory Program (CLP) without the raw data. The results reported include a case narrative; QC data for blanks, spikes and spike concentration, duplicates; tabulated calibration data and continuing calibration verification; quantitation for target analytes and surrogates; interference check sample results, internal standard area checks, and second column confirmation for GC analysis. The forms and control limits will be modified from the corresponding CLP forms as necessary to accommodate the unique characteristics of GC analyses and SW-846 methodology for GC/MS and ICP, and laboratory practice as documented in the SOPs. All GC/MS results will be supported by mass spectra evaluated and initialed by a qualified laboratory analyst.

Raw data will be stored in each laboratory according to their respective QA Manual. Raw data will be made available upon request by the QAR. It is likely that 100% of the confirmation samples will be required to undergo third party data validation as per the applicable standards in the EPA Functional Guidelines document for organics.

#### **11.4 DATA VALIDATION**

The Government may request that the entire excavation confirmation data set, or other soil results, be evaluated by third party data validation. These soil results from the fixed laboratory will be assessed using EPA CLP Functional Guidelines (EPA 1995) as a template and using analytical parameters and control limits as specified in the source methods and the SOPs documented in the Sampling and Analysis Plan. These parameters and limits differ in many ways from the CLP methodology. Hence, the individual topics listed below note areas where the project methods differ.

Other data types for this project are not designed for third party data validation and the raw data will be retained by the laboratories according to their records retention policies. However, 100% of all analytical data reported by the laboratories and field records project-wide will be reviewed by GSA to ensure the integrity of the data and records. Accuracy, precision and completeness will be computed and correlation between methods will be evaluated. Overall data representativeness and total measurement error will be evaluated. The chemical reports will include an assessment of measurement biases and a discussion of whether identified biases are significant to project decision making.

Analytical sensitivity (detection limits), preservation, holding times, PE sample results, calibration, and field and method blanks are included in the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) parameters evaluated.

All data will be reviewed and flagged as appropriate using project data quality criteria. Qualification of primary sample results and the basis for qualification will be discussed in the chemical reports.

All project records, from field sampling logs and notebooks to the completed Chain-of-Custody forms and laboratory reports, provide the basis for data validation. The Project Manager through the authority to assign tasks to the appropriate personnel and to set priorities, will verify that the whole data collection process has integrity, that the data quality objectives have been met, and that all records are retained. The data and sample handling requirements specified in Tables 1 and 2 must be verifiably met and all corrective actions brought to conclusion to the satisfaction of the Chemical Data Quality Manager. All

records will be retained for a minimum of five years, or as per state and local regulations, in case further review is necessary.

The Chemical Quality Control Manager is responsible for establishing the usability of all project data. The current project scope does not include third-party data validation except for those data sets determined by the Corps to need further evaluation. The laboratory and field record submittals for this project, however, are designed for third-party data validation at a future date, should such a review be needed. Under the current scope, the CDQCM will verify that the entire data collection process has integrity, that the data quality objectives have been met, and that all records are retained. All project records, from field sampling logs and notebooks to completed Chain-of-Custody forms and laboratory reports, provide the basis for data validation. The data quality and sample handling requirements specified in Tables 1 and 2 will be verified and all corrective actions will be responded to.

Spot checks of the data will be made to evaluate whether the data validation guidelines referenced below have been met. A review of the laboratory reports will be made in order to verify that all data needed for third party data validation are present. All records will be retained for a minimum of five years, or as per state and local regulations, in case further review is necessary.

The following criteria will be evaluated. Where they are different the criteria specified in the "Functional Guidelines," notation has been made. These unique criteria need to be considered for data acceptance by either the CDQCM or by third-party data review.

#### **11.4.1 Holding Times**

Holding time prior to analysis should meet the criteria in Table 5.

#### **11.4.2 GC/MS Tuning Criteria**

SW-846 tuning criteria used for this project are slightly more stringent than that for current CLP methodology. The SW-846 tuning criteria correlate with calibration criteria that are less stringent than CLP. Hence the criteria are not interchangeable. If the CLP tuning criteria are used, then the CLP calibration criteria must also be used. Some newer instruments have the recent CLP tuning criteria built into the software, hence caution must be used when running SW-846 methods because some of the instrument tunes may not meet SW-846 criteria. The time limit for valid data under a given instrument tune is 12 hours for SW-846, which is less stringent than CLP.

#### **11.4.3 Initial and Continuing Calibration Criteria**

The calibration criteria for SW-846 GC/MS methods are based on a subset of analytes identified as Calibration Check Compounds (CCC) and System Performance Check Compounds. The list of analytes specifically controlled in SW-846 methods is less than that for CLP. Hence, CLP criteria cannot be used for these methods. The calibration concentration range is also broader in SW-846. As noted above, the time limit for a valid tune and calibration is 12 hours for SW-846.

#### **11.4.4 Blank Contamination**

There are no allowable levels of background contamination specified in SW-846 methods, hence the guidelines in the EPA validation guidelines are not appropriate. Corrective action should be taken to reanalyze samples affected by blank contamination. As per the EPA validation guidelines, any result within a factor of ten of the concentration for analytes found in the batch control sample should be flagged if reanalysis after a clean blank was not performed.

#### **11.4.5 Matrix Spike and Surrogate Criteria**

The unique QC control limits for this project are specified in Table 4. The EPA CLP guidelines are based on different sample types, methods and concentration levels. Laboratory control samples are used to supplement evaluation of matrix spike nonconformances. The laboratory is expected to take internal action based on their statistically derived control limits, but must use the project specific control limits if the latter are more stringent.

#### **11.4.6 Pesticide Breakdown**

If the pesticide breakdown standard run with each instrumental batch shows the sum of DDE and DDD exceeding 15% of the DDT value, then results for all three analytes will be qualified for use with caution. The appropriate corrective action by the laboratory is to reanalyze samples without expense to the Government. If the breakdown exceeds 20%, the results will be rejected.

#### **11.4.7 Laboratory Turnaround Time**

The laboratory will notify the Chemical Data Quality Manager if results will take beyond three days to deliver.

### **11.5 DEFINITION OF USABLE VS. REJECTED DATA**

Data will be evaluated using SW-846 criteria and project specific DQOs. Data will be rejected if any part of these criteria have not been met and no documented corrective actions have been taken. Rejected data are not usable. If corrective actions have been taken but QC criteria still could not be met, data will be flagged for use with caution (for example, a “J” qualifier), provided that the corrective action provides evidence that use of the data will not result in the wrong decision for the project. Such data will be defined as usable.

## **12. PREVENTATIVE MAINTENANCE**

Preventative maintenance activities are performed in order to prevent loss of data due to malfunctions or delay. Critical functions are identified for field and laboratory and contingencies are accordingly established.

### **12.1 FIELD ACTIVITIES**

The critical functions in the field require that extra sampling containers and field-screening kit reagents be on hand in the field. Alternative sources (such as an instrument rental agency) for field screening or health and safety-related monitoring devices will be identified prior to going in the field. This contingency will prevent loss of data or delays.

### **12.2 LABORATORY ACTIVITIES**

The Laboratory QA/QC plan should outline a formal preventative maintenance program, including contingencies for sending samples to a Corps approved back-up laboratory if samples requiring analysis within regulatory holding times are going to be compromised. Major and critical equipment should be on a service contract or under a laboratory program staffed by equipment technicians capable of emergency service. Back-up instrumentation should be available for larger projects. Routine maintenance for equipment for this project are outlined below.

### 12.2.1 IAA Analysis

The enzyme conjugates will be refrigerated when not in use and allowed to adjust to room temperature prior to use.	Daily
Refrigerators will be checked for proper storage temperature ( $4 \pm 2^{\circ}\text{C}$ ).	Daily
Soil sample balance will be checked with a 100 g weight.	Daily or when disturbed.
Pipetter calibration will be checked with the volumetric calibration kit prior to use.	Weekly
Spectrometer calibration will be checked prior to use.	Daily

### 12.2.2 Gas Chromatography of Purge-and-Trap device (Mod. 8021 or 8260)

Septum replacement	Daily
Flow rate/purge rate check	Monthly or as required by EPA Method 8000 to meet retention time window criteria
Gases	New lots are analyzed before use

### 12.2.3 ICP for Metals

Sample pump tubing replacement	Weekly or as needed
Torch cleaning	Monthly, spare torch on hand

## 13. PERFORMANCE AND SYSTEM AUDITS

GSA will document inspections and audits to confirm the quality or orderly progression of a portion of the work by outlining procedures, acceptability of methods or personnel, qualifications, or other verifications of quality. Performance audits (performance samples) and system audits (site inspections) of the fixed laboratories are performed by the Corps during the validation described in EM 200-1-1. No additional audits for laboratories are scheduled for this project. GSA will perform audits of field sampling and analysis operations periodically throughout the project to document the implementation of the QA program. GSA will perform audits of the laboratory and field operations at the discretion of the Corps QA Representative and if deemed necessary as part of a corrective action for a problem encountered with sampling and analytical data.

## **14. CHEMICAL QUALITY CONTROL REPORTS**

Daily activities and decisions that affect the quality of the results will be documented on the Daily Quality Control Reports, along with field conditions, samples taken, and decisions made. This includes activities during field operations and for each day data are collected. Observations of site conditions which could affect performance of chemical tests will be documented. Deviations from procedures or expected results will be addressed in the daily report, along with corrective actions. An example report form is provided in Appendix A. Chemical QC reports need to be provided daily (within 24 hours) and have IAA results attached.

### **14.1 SUMMARY CHEMICAL QC REPORTS**

Summary Chemical QC Reports covering the observations and conclusions of data review of each sample delivery group by GSA will be submitted with laboratory reports within 1 week of receipt of results. Summary Chemical QC Reports will include the following areas:

1. Summary of DQOs Affected
2. Sample Handling, Holding Time and Chain of Custody
3. Methodology, Comparability, and Performance Sample Results
4. Analytical Sensitivity
5. Accuracy - Calibration, Matrix Spikes, Laboratory Control Samples, Surrogates and Blanks
6. Precision - Overall Measurement and Laboratory
7. Data Integrity - Consistency of Field and Laboratory Documentation
8. Representativeness
9. Completeness
10. Conclusions and Corrective Actions

### **14.2 THIRD-PARTY DATA VALIDATION**

For selected results, upon request of the Corps, GSA will provide validation for the complete raw data record according to guidelines based on the EPA Functional Guidelines, as modified to meet the project methodology needs and DQOs (described in Part 11.4). These Data Validation Reports will be delivered to the Corps within 30 days of the validation request. The following areas will be covered:

#### **14.2.1 Organics**

1. Holding Times and Preservation
2. GC/MS Tune and Instrument Performance Checks
3. Initial and Continuing Calibration - SW-846 Criteria
4. Internal Standard Area Checks
5. Blanks and Associated Samples
6. System Monitoring Compounds
7. Surrogates
8. Matrix Spikes and Laboratory Control Samples

9. Duplicate Matrix Spikes and Laboratory Control Sample Duplicates (if performed)
10. Sample Duplicates
11. Target Compound Identification
12. Compound Quantitation and Project Quantitation Limits
13. System Performance
14. Overall Assessment

#### **14.2.2 Inorganics**

1. Holding Times and Preservation
2. Interference Check Sample Results
3. Linear Range
4. Detection Limits
5. Initial and Continuing Calibration
6. Calibration Blanks
7. Blank
8. Laboratory Control Sample
9. Spike Sample Analysis
10. Serial Dilutions
11. Duplicate Spike Precision
12. Field Duplicate Precision
13. Overall Assessment

**APPENDIX A - SAMPLING AND ANALYSIS FORMS**

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**Chain-of-Custody Form**  
**Chain-of-Custody Seal**  
**Daily Chemical Quality Control Report**  
**Laboratory Cooler Receipt**  
**Corrective Action Report**  
**Field Sampling Log Sheet**

## **APPENDIX B - ANALYTICAL SOPS**

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### **SOUND ANALYTICAL:**

- 1. ORGANOPHOSPHORUS PESTICIDES BY GC/MS (8141 MOD)**
- 2. ORGANOCHLORINE PESTICIDES (8081)**
- 3. ICP/MS FOR METALS (6020)**
- 4. ICP FOR METALS (6010)**
- 5. EXTRACTION OF SEMIVOA IN SOIL BY SOXHLET (3540)**
- 6. EXTRACTION OF SEMIVOA IN WATER (3510)**
- 7. DIGESTION OF SEDIMENTS FOR ICP (3050)**
- 8. DIGESTION OF WATER FOR ICP AND ICPMS (3010)**
- 9. DIGESTION OF TCLP EXTRACT FOR ICP (3010)**
- 10. TCLP (1311)**

### **CASCADE ANALYTICAL**

- 1. GC/NPD FOR CARBAMATE PESTICIDES (8141 MOD)**

### **NORTH COAST ANALYTICAL**

- 1. PARAQUAT IN SOIL**

### **IAA/FIELD LABORATORY**

- 1. COVER SHEET WITH CUSTOM PROCEDURES FOR DDT AND CHLORDANE IAA TESTS**
- 2. STANDARD INSTRUCTIONS FOR DDT AND CHLORDANE IAA TESTS**

## APPENDIX C - REFERENCES

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EPA 1994. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, EPA540/R-94/012, EPA Office of Emergency Response and Remedial Response, Washington, D.C., February 1994.

EPA 1994. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA540/R-94/013, EPA Office of Emergency Response and Remedial Response, Washington, D.C., February 1994.

EPA 1995. Test Methods for Evaluating Solid Waste. SW-846. Third Edition. OSW. September, 1986, with Updates through 1995.