

CLP Organics Data Review  
and Preliminary Review  
(CLP/SOW OLM0 4.3)



Prepared by: George Karras Date: 12/05/06  
George Karras, Chemist HWSS

Peer Reviewed by: Russell Arrone Date: 12/05/06  
Russell Arrone, Chemist HWSS

Concurred by: Linda Maue Date: 12/15/06  
Linda Maue, Chief HWSS

Approved by: Robert Runyon Date: 12/11/06  
Robert Runyon, Chief HWSB

Annual Review

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_  
Name

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_  
Name

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CLP Data Assessment Attachment 1

BFB ION ABUNDANCE CRITERIA Attachment 2

DFTPP ION ABUNDANCE CRITERIA Attachment 3

## INTRODUCTION

### Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the methods in the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis OLM04.2," May 1999. The validation methods and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999. This document attempts to cover technical as well as contractual problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

In addition to technical requirements, contractual requirements are also covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

### Summary of Method

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 3.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance. This information is further summarized on the Organic Regional Data Assessment Summary and Data Rejection Summary forms (see attached).

CADRE reports, when available, may be incorporated into the Data Assessment.

### Reviewer Qualifications

Data reviewers must possess a working knowledge of the USEPA Statement of Work and National Functional Guidelines mentioned above.

## DEFINITIONS

### Acronyms

BFB - bromofluorobenzene  
BHC - benzene hexachloride  
BNA - base neutral acid (another name for Semi Volatiles)  
CADRE - Computer Aided Data Review and Evaluation  
CARD - CLP Analytical Results Database  
CCS - contract compliance screening  
CLASS - Contract Laboratory Analytical Services Support  
CLP - Contract Laboratory Program  
CRQL - Contract Required Quantitation Limit  
%D - percent difference  
DCB - decachlorobiphenyl  
DDD - dichlorodiphenyldichloroethane  
DDE - dichlorodiphenylethane  
DDT - dichlorodiphenyltrichloroethane  
DoC - Date of Collection  
GC - gas chromatography  
GC/ECD - gas chromatograph/electron capture detector  
GC/MS - gas chromatograph/mass spectrometer  
GPC - gel permeation chromatography  
IS - internal standard  
kg - kilogram  
µg - microgram  
MAGIC - Mainframe Access Graphical Interface with CARD  
MS - matrix spike  
MSD - matrix spike duplicate  
l - liter  
ml - milliliter  
PCB - Polychlorinated biphenyl  
PE - performance evaluation  
PEM - Performance Evaluation Mixture  
QC - quality control  
RAS - Routine Analytical Services  
RIC - reconstructed ion chromatogram  
RPD - relative percent difference  
RRF - relative response factor  
RRF - average relative response factor (from initial calibration)  
RRT - relative retention time  
RSD - relative standard deviation  
RT - retention time  
RSCC - Regional Sample Control Center  
SDG - sample delivery group  
SMC - system monitoring compound  
SOP - standard operating procedure  
SOW - Statement of Work  
SVOA - semivolatile organic acid  
TCL - Target Compound List  
TCLP - Toxicity Characteristics Leachate Procedure  
TCX - tetrachloro-m-xylene  
TIC - tentatively identified compound

**Acronyms (cont'd.)**

TOPO - Task Order Project Officer  
TPO - Technical Project Officer  
VOA - Volatile organic  
VTSR - Validated Time of Sample Receipt

**Data Qualifiers**

- U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
- JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

**LAB QUALIFIERS:**

- D - The positive value is the result of an analysis at a secondary dilution factor.
- B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.
- E - The concentration of this analyte exceeds the calibration range of the instrument.
- P - Pesticide/Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.
- A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.
- C - Applies to Pesticide results where the identification of the analyte has been confirmed by GC/MS.

X,Y,Z Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

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YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: LABORATORY:

SITE NAME: SDG Number(s):

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ]

ACTION: If no, contact RSCC, or contact the WAM to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples and all fractions? [ ]

ACTION: If no, contact either RSCC or ask the WAM to obtain this information from the prime contractor.

2.0 Data Completeness and Deliverables

2.1 Have any missing deliverables been received and added to the data package? [ ]

NOTE: The lab is required to submit data for only two analyses, for each fraction. (i.e., the original sample and one dilution, or the most concentrated dilution analyzed and one further dilution.)

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the package in the Contract Problems/Non-compliance section of the Data Assessment.

2.2 Was CLASS CCS checklist included with package?

2.3 Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, Sampling Report and Sample Tags? [ ]

ACTION: If yes, contact the WAM to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 Cover Letter SDG Narrative

3.1 Is the Narrative or Cover Letter Present? [ ]

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		YES	NO	N/A
3.2	Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.6.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken?			
3.3	Does the narrative contain the following information:  VOA: description of trap and columns used for sample analyses?  VOA: a NOTE stating whether Volatile low level soil samples prepared according to the modified SW-846 Method 5035?(p. B-9/VOA, sec 2.6.1)  VOA: any discrepancies between low level soil weights determined in the field and in the Laboratory? (p. B-10/VOA, sec. 2.6.1)  BNA: description of columns used for sample analyses?  Pest: description of columns used for sample analyses?	[ ]	___	___
NOTE:	As per section 6.23.3.1 SOW/p. D-11/Pest, Packed columns are not permitted.	___	[ ]	___
3.4	Does the narrative, VOA and BNA sections, contain a list of all TIC's identified as alkanes and their estimated concentrations?	[ ]	___	___
3.5	Is the temperature indicator bottle present in the cooler? If not, did the Laboratory document in the SDG Narrative the alternative technique used to determine the cooler temperature?(Exhibit A/ p. A-5 sec. 4.2.1.2.3.3)	[ ]	___	___
3.6	Does the narrative contain a record of all cooler temperatures? If the temperature of a cooler was exceeded, > 10° C, the lab must list by fraction and sample number, all affected samples.	[ ]	___	___
3.7	Does the Narrative contain a list of sample reanalyses submitted? Did the Lab distinguish whether the reanalysis is billable, and if so why?			
3.8	Does the narrative contain a list of the pH values determined for each water sample submitted for volatile analysis (SOW Exhibit B, section 2.6.1.2)?	[ ]	___	___
3.9	Does the Case Narrative contain the statement, "verbatim", as required in Section B of the SOW?	[ ]	___	___
		[ ]	___	___
		[ ]	___	___

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		YES	NO	N/A
		[ ]	—	—
<p>ACTION: If "No", to any question in this section, contact the TOPO to obtain all necessary resubmittals. If information is not available, document in the Data Assessment under Contract Problems/Non-Compliance section.</p>				
<b>4.0 Data Validation Checklist</b>				
4.1	Check the package for the following discrepancies:			
	a. Is the package paginated in ascending order starting from the SDG narrative?	[ ]	—	—
	b. Are all forms and copies legible?	[ ]	—	—
	c. Is each fraction assembled in the order set forth in the SOW?	[ ]	—	—
	The following checklist is divided into three parts. Part A is for any VOA analyses, Part B is for BNA's and Part C is Pesticide/PCB's.	[ ]	—	—
	Does this package contain:			
	VOA Data?			
	BNA Data?			
	Pesticide/PCB data?	—	—	
	ACTION: Complete corresponding parts of checklist.	—	—	
		—	—	

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YES NO N/A

PART A: VOA ANALYSES

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50% - 90% water, all data shall be flagged as estimated (J). If a soil sample other than TCLP contains more than 90% water, then qualify positive results "J", and non-detects "R".

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (> 10° C), then flag all positive results with a "J" and all non-detects "UJ".

ACTION: If both VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: The smallest soil size permitted is 0.5g. If any soil sample is smaller than 0.5g, document in the Data Assessment under Contract Problems/Non-Compliance.

\_\_\_ [ ] \_\_\_

2.0 Holding Times

2.1 Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?

Technical Holding Times for AQUEOUS AND SOIL NON-ENCORE SAMPLES: If unpreserved, aqueous samples, maintained at 4° C for aromatic hydrocarbons analysis must be analyzed within 7 days of collection. If preserved with HCl (pH < 2) and stored at 4° C, then aqueous samples must be analyzed within 14 days of collection. If uncertain about preservation, contact sampler to determine whether or not samples were preserved. The holding time for non-Encore soils is 10 days from date of collection.

ACTION: If technical holding times for aqueous samples and soil non-Encore samples are exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ", and document in the Data Assessment that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results must be qualified "J", but the reviewer may determine that non-detect data are unusable "R". If holding times are exceeded by more than 28 days, all non detect data are unusable "R".

\_\_\_ [ ] \_\_\_

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NOTE: Contractual Holding Times: Analysis of water samples must be completed within 10 days of Validated Time of Sample Receipt (VTSR). This requirement does not apply to Performance Evaluation (PE) samples.

**Technical Holding Times for soils Encore samples:**

- i) If sample was preserved  $\leq$  2 days of VTSR:
  - 1. and analyzed  $\leq$  14 days from DoC, NO action needed.
  - 2. and analyzed  $>$  14 days from DoC, qualify positive results "J" and non-detects "UJ".
  - 3. and analyzed  $>$  28 days from DoC, qualify positive results "J" and non-detects "R".
  
- ii) If sample was NOT preserved, or preserved  $>$  2 days of VTSR
  - 1. and analyzed  $\leq$  7 days from DoC, No action needed.
  - 2. and analyzed  $>$  7 days from DoC, qualify AROMATIC analytes only, both positive and non-detects, as estimated "J".
  - 3. and analyzed  $>$  10 days from DoC, qualify ALL positive analytes "J" and ALL non-detects as "UJ".
  - 4. and analyzed  $\geq$  20 days from DoC, qualify positive results "J" and non-detects "R".

Note: CONTRACT holding times for soil Encore samples are:

- 1. Samples must be preserved within two (2) days of VTSR and must be analyzed within ten (10) days of VTSR.
- 2. Samples NOT preserved within two (2) days of VTSR must be analyzed within two (2) days of VTSR.

ACTION: If contractual holding times are exceeded, document in the Data Assessment.

NOTE: The data reviewer must note in the Data Assessment whether or not technical and contractual holding times were met.

**Table of Holding Time Violations**

(See Chain-of-Custody Records)

Sample ID	Sample Matrix	Was Sample Preserved?	Date Sampled	Date Lab Received	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

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		YES	NO	N/A
<hr/>				
<b>3.0 System Monitoring Compound (SMC) Recovery (Form II)</b>				
3.1	Are the VOA SMC Recovery Summaries (Form II) present for each of the following matrices:			
	a. Low Water?	<input type="checkbox"/>	___	___
	b. Low Soil?	<input type="checkbox"/>	___	___
	c. Med Soil?	<input type="checkbox"/>	___	___
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water?			
	b. Low Soil?	<input type="checkbox"/>	___	___
	c. Med Soil?	<input type="checkbox"/>	___	___
ACTION:	Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory. If missing deliverables are unavailable, document the effect in the Data Assessment.	<input type="checkbox"/>	___	___
3.3	Were outliers marked correctly with an asterisk?			
ACTION:	Circle all outliers with red <u>pencil</u> .			
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?	<input type="checkbox"/>	___	___
	If yes, were samples re-analyzed?			
	Were method blanks re-analyzed?			
ACTION:	If recoveries are $\geq 10\%$ , but 1 or more compounds fail to meet SOW specifications:	___	<input type="checkbox"/>	___
	1. All positive results are qualified as estimated "J".	<input type="checkbox"/>	___	___
	2. Flag all non-detects as estimated detection limits "UJ" where recovery is less than the lower acceptance limit.			
	3. If SMC recoveries are above allowable levels, qualify positive results "J" and do not qualify non-detects.			
ACTION:	If any system monitoring compound recovery is $< 10\%$ :			
	1. Flag all positive results as estimated "J".			
	2. Flag all non-detects as unusable "R".			

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YES    NO    N/A

Professional judgement should be used to qualify data that only have method blank SMC recoveries out of specification in both original and re-analyses. Check the internal standard areas.

NOTE: Contractual requirements state that if any SMC fails the acceptance criteria, the sample must be re-analyzed. If the affected sample was not re-analyzed, document in the Data Assessment under Contract Problems/Non-Compliance.

NOTE: The laboratory must submit the following data:

1. If SMC recoveries and internal standard responses meet the acceptance criteria in the re-analyzed sample, then the laboratory must submit only the re-analysis.
2. If an SMC recovery and/or internal standard response fails to meet the acceptance criteria upon re-analysis, then submit data from both analyses.

(Refer to section 11.4.3.2, page D-45/VOA of the SOW for more information.)

3.5 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, contact the TOPO to obtain an explanation or resubmittal of corrected deliverables from the laboratory. Make any necessary corrections and note the effect in the Data Assessment.

\_\_\_        \_\_\_

**4.0 Matrix Spikes (Form III)**

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?

   \_\_\_    \_\_\_

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water?

   \_\_\_    \_\_\_

b. Low Soil?

   \_\_\_    \_\_\_

c. Med Soil?

   \_\_\_    \_\_\_

ACTION: If any matrix spike data are missing, take the action specified in section 3.2 above.

ACTION: No action is taken based upon MS/MSD data alone. However, using informed professional judgement, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data.

ACTION: Circle all outliers with red pencil.

**5.0 Blanks (Form IV)**

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	YES	NO	N/A
5.1 Is the Method Blank Summary (Form IV) present?	<input type="checkbox"/>	___	___
5.2 Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed during every 12-hour time period on each GC/MS system, before any samples, and for each matrix?(water, low soil or medium soil)	<input type="checkbox"/>	___	___
5.3 Has a VOA method blank been analyzed at least once every twelve hours for each matrix/concentration and GC/MS system used?	<input type="checkbox"/>	___	___
5.4 Was a VOA instrument blank analyzed after each sample/dilution which contained a target compound that exceeded the initial calibration range?	<input type="checkbox"/>	___	___
5.5 Was a VOA storage blank analyzed at the end of all samples for each SDG in a case?	<input type="checkbox"/>	___	___
ACTION: If any method/instrument blank data are missing, contact the TOPO to obtain any missing deliverables from the laboratory. If method blank data are not available, reject "R" all associated positive data. However, using professional judgement, the data reviewer may substitute field blank or trip blank data for missing method blank data.	<input type="checkbox"/>	___	___
<p>If the instrument blank was not analyzed after a sample with high concentration of reported values, inspect the chromatogram of the sample analyzed immediately after this analysis for possible carryover. Use professional judgement to determine if any contamination occurred and qualify analyte(s) accordingly.</p> <p>If storage blank data is missing, contact the TOPO to obtain any missing deliverables from the laboratory. If unavailable, note in the Contract Problems/Non-Compliance section of the Data Assessment.</p>			
Note: A storage blank shall be analyzed and	<p>reported as a water sample unless the SDG contains only soil samples. Then, the storage blank may be analyzed and reported as a soil sample. (p. D-49/VOA sec. 12.1.3.5)</p>		
5.6 The validator should verify that the correct identification scheme for the EPA Blank samples were used. See page <u>B-30</u> , section 3.3.7.3 of the SOW for further information.			
Was the correct identification scheme used for all VOA blanks?			
ACTION: Contact the TOPO to obtain missing deliverables from the lab, or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-compliance if corrections were made by the validator.	<input type="checkbox"/>	___	___

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	YES	NO	N/A
<p>5.7 Chromatography: review the blank raw data- chromatograms (RICs), quant. reports or data system printouts and spectra. Is the chromatographic performance (baseline stability) for each instrument acceptable for VOA's?</p> <p>ACTION: Use professional judgement to determine the effect on the data.</p>			
<p>5.8 Are all detected hits for target compounds in method, instrument and storage blanks less than the CRQL for that analyte?</p> <p><u>Exception:</u> Acetone and 2-butanone must be less than 5 times the CRQL, and methylene chloride and Cyclohexane must be less than 2.5 times its CRQL. (p. D-50/VOA sec. 12.1.4.6)</p> <p>ACTION: If no, an explanation and laboratory's corrective actions must be addressed in the case narrative. If the narrative contains no explanation, then make a note in the Contract Problems/Non-Compliance section of the Data Assessment.</p>	[ ]	___	___
<p>6.0 <u>Contamination</u></p> <p>NOTE: "Water blanks", "drill blanks", and "distilled water blanks" are validated like any other sample, and are <u>not</u> used to qualify data. Do not confuse them with the other QC blanks discussed below.</p> <p>6.1 Do any method/instrument/reagent/storage blanks have positive results (TCL and/or TIC) for VOA's?</p> <p>NOTE: When applied as directed in the table below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for %moisture when necessary.</p> <p>NOTE: A contaminated instrument blank is not allowable under this SOW. The instrument blank must meet the technical acceptance criteria for blank analyses(sec. 12.1.4). See page D-48/VOA, section 12.1.2.4 for additional information. Document in the Data Assessment under Contract Problems/Non-Compliance if contaminated instrument blank was submitted.</p> <p>6.2 Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?</p> <p>ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)</p> <p>NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped and are not required for non-aqueous matrices. Blanks may not be qualified because of contamination in another blank. Field Blanks &amp; Trip Blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration, and Internal standard QC problems.</p> <p>ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated data should be qualified as unusable "R".</p>	___	[ ]	___

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YES    NO    N/A

NOTE: Analytes qualified "U" for blank contamination are till considered as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R".

For:	Flag sample result with a "U"	Report CRQL & when: qualify "U"	No qualification when: is needed when:
------	----------------------------------	------------------------------------	---

<u>TCL COMPOUNDS</u>			
Methylene Chloride	Sample conc. is > CRQL, but ≤ 10× blank value.	Sample conc. is < CRQL and ≤ 10× blank value.	Sample conc. is > CRQL and > 10× blank value.
Acetone	blank value.	blank value.	blank value.
Toluene			
2-Butanone			
<u>Cyclohexane</u>			

Other Conta- minants	Sample conc. is > CRQL, but ≤ 5× blank value.	Sample conc. is < CRQL and ≤ 5× blank value.	Sample conc. is > CRQL and > 5× blank value.
----------------------	---	--	--

6.3 Are there field/rinse/equipment blanks associated with every sample?

[ ]    —    —

ACTION: For low level samples, note in the Data Assessment that there is no associated field/rinse/equipment blank. For samples with high concentrations of suspected blank contaminants, use professional judgement to qualify these values and make a note in the Data Assessment.

Exception: samples taken from a drinking water tap do not have associated field blanks.

**7.0 GC/MS Instrument Performance Check (Form V)**

7.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?

[ ]    —    —

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?

[ ]    —    —

7.3 Is the mass spectrum of BFB acquired according to sec. 9.2.4.1 D-23/VOA?

[ ]    —    —

Note: Sec. 9.2.4.1 states that "the mass spectrum of BFB MUST be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and MUST be accomplished using a single scan no more than 20 scans prior to the elution of BFB. DO NOT background subtract part of the BFB peak." See Attachment 2 for BFB criteria.

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YES    NO    N/A

Action: If not, reject "R" all samples associated with \_\_\_\_\_ that particular BFB.

7.4        Has an instrument performance check been analyzed for every analytical sequence on each instrument?

ACTION: List date, time, instrument ID, and sample numbers for which associated GC/MS tuning data are unavailable.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____

[ ]    \_\_\_    \_\_\_

ACTION: Notify the TOPO to obtain missing data, if possible. If the lab cannot provide the missing data, reject, "R", all data generated outside an acceptable twelve hour calibration interval.

7.5        Have the ion abundances been normalized to m/z 95 as specified in Exhibit D, page D-56/VOA?

NOTE: All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

ACTION: If mass assignment is in error, qualify all associated data as unusable "R".

[ ]    \_\_\_    \_\_\_

7.6        Have the ion abundance criteria been met for each instrument used?

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TPO must be notified.

[ ]    \_\_\_    \_\_\_

7.7        Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values, but if errors are found check more.)

7.8        Is the number of significant figures for the reported relative abundances consistent with the number given for each ion in the ion abundance criteria column?

\_\_\_    [ ]    \_\_\_

ACTION: If large errors exist, take action as specified in section 3.5 above.

[ ]    \_\_\_    \_\_\_

7.9        Are the spectra of the mass calibration compound acceptable?

ACTION: Use professional judgement to determine whether associated data should be

[ ]    \_\_\_    \_\_\_

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		YES	NO	N/A
<p>accepted, qualified, or rejected.</p>				
<p>8.0 <u>Target Compound List (TCL) Analytes (FORM I VOA)</u></p>				
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Samples and/or fractions as appropriate?			
	b. Matrix spikes and matrix spike duplicates?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	c. Blanks?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (quant. reports) included in the sample package for each of the following:	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	a. Samples and/or fractions as appropriate?			
	b. Matrix spikes and matrix spike duplicates (mass spectra not required)?			
	c. Blanks?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION:	If any data are missing, take action specified in 3.2 above.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8.3	Is chromatographic performance acceptable with respect to:			
	a. Baseline stability?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	b. Resolution?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	c. Peak shape?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	d. Full-scale graph (attenuation)?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	e. Other: _____?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION:	Use professional judgement to determine the acceptability of the data.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8.4	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?			
ACTION:	If any mass spectra are missing, take action as specified in 3.2 above. If the lab does not generate its own standard spectra, document in the Contract Problems/Non-compliance section of the Data Assessment.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8.5	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?			
8.6	Are all ions present in the standard mass spectrum at a relative			

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		YES	NO	N/A
	intensity greater than 10% also present in the sample mass spectrum?			
8.7	Do sample and standard relative ion intensities agree within ±20%?	[ ]	___	___
ACTION:	Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected "R", flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected "U" at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 8.5, 8.6, and 8.7.	[ ]	___	___
ACTION:	When sample carry-over is suspected, use professional judgement determine if instrument cross-contamination has affected positive compound identifications.	[ ]	___	___
<b>9.0 <u>Tentatively Identified Compounds (TIC)</u></b>				
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TIC's include scan number or retention time, estimated concentration and "JN" qualifier?			
9.2	Are the mass spectra for the TIC's and associated "best match" spectra included in the sample package for each of the following:	[ ]	___	___
	a. Samples and/or fractions as appropriate?			
	b. Blanks?			
	c. Are Alkanes listed in/or part of the Case Narrative?	[ ]	___	___
ACTION:	If any TIC data are missing, take action specified in 3.2 above.	[ ]	___	___
ACTION:	Add "JN" qualifier to all chemically named TIC's, if missing.	[ ]	___	___
9.3	Are any TCL compounds (from any fraction including all PCB congeners) listed as TIC compounds? (Example: 1,2- dimethylbenzene is xylene, a VOA TCL analyte, and should not be reported as a TIC.)			
ACTION:	Flag with "R" only TCL compound detected in another fraction. (Except blank contamination)			
9.4	Are any TIC's reported earlier than 30 sec before the first purgeable compound, or three (3) min. after the last purgeable compound listed in Exhibit C (Volatiles)?			
ACTION:	Flag with "R" any TIC compound reported. (p. D38-VOA, sec. 11.1.2.2)	___	[ ]	___
9.5	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?			
9.6	Do TIC and "best match" standard relative ion intensities agree within ±20%?	___	[ ]	___

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YES    NO    N/A

[ ]    —    —

[ ]    —    —

ACTION: Use professional judgement to determine the acceptability of TIC identifications. If it is determined an incorrect identification was made, change the identification to "unknown," or to some less specific identification as appropriate. (Example: "C3 substituted benzene.")

Also, when a compound is not found in any blank, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable "R". (E.g., Common Lab Contaminants: CO<sub>2</sub> (M/E 44), Siloxanes (M/E 73) hexane, aldol condensation products, solvent preservatives, and related by-products.

9.7 Are TIC's with responses < 10% of the internal standard (as determined by inspection of the peak areas or height) reported?

ACTION: If yes, cross out questionable TIC's.

**10.0 Compound Quantitation and Reported Detection Limits**

10.1 Are there any transcription/calculation errors in Form I results? (Check at least two positive values. Verify that the correct internal standards, quantitation ions, and RRF were used to calculate Form I results.)

—    [ ]    —

10.2 Are the CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture?

ACTION: If errors are large, take action as specified in section 3.2 above.

—    [ ]    —

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQL's are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample). Replace concentrations that exceeded the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form Is not to be used, including any in the data summary package.

[ ]    —    —

**11.0 Standards Data (GC/MS)**

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts

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	YES	NO	N/A
(quant. reports) present for each initial and continuing calibration?			
ACTION: If any calibration standard data are missing, take action specified in 3.2 above.	[ ]	—	—
<b>12.0 GC/MS Initial Calibration (Form VI)</b>			
12.1 Are the Initial Calibration Forms (Form VI) present and complete at concentrations of 10, 20, 50, 100, 200ng for separate calibrations of low water/med soils (unheated purge) and low soils (heated purge)?			
ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.			
12.2 Were all low level soil standards, blanks and samples analyzed by heated purge?	[ ]	—	—
ACTION: If low level soil samples were not heated during purge, qualify positive hits "J" (estimated) and non-detects "R".			
12.3 Are the % relative standard deviation (%RSD) values for VOA's ≤ 30% over the concentration range of the calibration?	[ ]	—	—
NOTE: Although 23 VOA compounds have a contractual minimum RRF and no maximum %RSD, the technical acceptance criteria are the same for all analytes.			
ACTION: Circle all outliers with red <u>pencil</u> .			
	[ ]	—	—
ACTION: If %RSD is > 30.0%, qualify associated positive results for that analyte "J" (estimated). Do not qualify non-detects. When %RSD is > 90%, flag all non-detects for that analyte "R" (unusable) and positive hits "J" .			
NOTE: Analytes previously qualified "U" for blank contamination are still considered as "hits" when qualifying for initial calibration criteria.			
12.4 Are any average RRFs < 0.05?			
ACTION: Circle all outliers with red <u>pencil</u> .			
ACTION: If the average RRF is < 0.05, then qualify associated non-detects with an "R" and flag associated positive data as estimated "J".	—	[ ]	—
NOTE: <u>Contract Requirement</u> : The SOW allows up to two of the <u>required</u> analytes to fail contractual %RSD or RRF criteria, provided the %RSD is ≤ 40% and RRF is ≥ 0.010. (See Table 5, page D-61/VOA and analytes marked with a "*" on Form VI for required analytes and contractual criteria.) Technical criteria, however, are the same for all analytes.			
ACTION: If more than two analytes failed %RSD or RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.			
12.5 Are there any transcription/calculation errors in the reporting of average relative response factors (RRF) or %RSD? (Check at least 2 values, but if errors are found, check more.)			

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YES NO N/A

ACTION: Circle errors with red pencil.

ACTION: If errors are large, contact the TOPO to obtain an explanation/resubmittal from the lab, document in the Data Assessment under Contract Problems/Non-Compliance.

13.0 GC/MS Continuing Calibration (Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for separate calibration of low water/med soil and low soil samples?

—  —

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?

— —

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, contact the TOPO to request an explanation/resubmittal from the lab. If continuing calibration data are not available, flag all associated sample data as unusable "R".

— —

ACTION: List below all sample(s) that were not analyzed within twelve hours of the previous continuing calibration.

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

13.3 Do any volatile compounds have a percent difference (%D) between the initial and continuing RRF which exceeds the ±25% criteria?

NOTE: Although 23 VOA compounds have a contractual minimum RRF and no maximum %D, the technical acceptance criteria are the same for all analytes.

ACTION: Circle all outliers with red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated. When %D is > 90%, qualify all non-detects for that analyte unusable (R) and positive results estimated (J) .

—  —

13.4 Are any continuing calibration RRFs < 0.05?

ACTION: Circle all outliers with red pencil.

ACTION: If the RRF is < 0.05, qualify the associated non-detects as unusable "R"

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YES    NO    N/A

and the associated positive values "J".

NOTE: Contract Requirement: The SOW allows up to two of the required analytes to fail contractual %D and RRF criteria, provided that the %D is  $\leq 40\%$  and the RRF is  $\geq 0.010$ . (See Table 5 pg. D-61/VOA or analytes marked with a "\*" on Form VI for required analytes.) Technical criteria, however, are the same for all analytes. \_\_\_ [ ] \_\_\_

ACTION: If more than two analytes failed %D and RRF, criteria document in the Data Assessment under contract Problems/Non-Compliance.

13.5 Are there any transcription/calculation errors in the reporting of RRF or %D between initial and continuing RRFs? (Check at least two values, but if errors are found, check more.)

ACTION: Circle errors with red pencil. \_\_\_ [ ] \_\_\_

ACTION: If errors are large, contact the TOPO to obtain an explanation/resubmittal from the lab, document in the Data Assessment under Contract Problems/Non-Compliance.

**14.0 Internal Standard (Form VIII)**

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

If no, was the sample re-analyzed?

ACTION: 1. Circle all outliers with red pencil. [ ] \_\_\_ \_\_\_  
2. List all the outliers below.

Sample #	Internal Std.	Area	Lower/Upper Limit		
_____	_____	_____	/	_____	
_____	_____	_____	/	_____	
_____	_____	_____	/	_____	

(Attach additional sheets if necessary,  
or attach copies of Form VIIIs.)

ACTION: If any sample was not re-analyzed, document in the Data Assessment under Contract Problems/Non-Compliance.

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when associated IS area counts are > 100%.

3. If the IS area in the sample is below the "lower limit," < 50%,

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qualify all analytes associated with that IS estimated, "J". If the area counts are extremely low, < 25% of the area in the 12 hour standard, or if performance exhibits a major abrupt drop-off, flag all associated non-detects as unusable, "R", and positive hits estimated, "J".

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

[ ] \_ \_

NOTE: Contractual requirements state that if any internal standard fails the acceptance criteria, the sample must be re-analyzed. If the affected sample was not re-analyzed, document in the Data Assessment under Contract Problems/Non-Compliance.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for VOA analysis?

[ ] \_ \_

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

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PART B: BNA ANALYSES

**1.0 Sample Conditions/Problems**

1.1 Do the Traffic Reports/Chain-of-Custody records or laboratory SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

\_\_\_ [ ] \_\_\_

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50% - 90% water, all data should be flagged as estimated "J". If a soil sample, other than TCLP, contains more than 90% water, qualify positive hits "J" and non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon arrival at the laboratory and the temperature of the cooler was elevated (> 10° C), flag all positive results "J" and all non-detects "UJ".

**2.0 Holding Times**

2.1 Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?

NOTE: Technical Holding Time: Continuous extraction of water samples for BNA analysis must be started within fourteen days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within 40 days of the date of extraction.

\_\_\_ [ ] \_\_\_

Table of Holding Time Violations  
(See Chain-of-Custody Records)

Sample Analyzed	Sample Matrix	Date Sampled	Date Lab Received	Date Lab Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If technical holding times were exceeded, flag all positive results as estimated (J) and sample quantitation limits as estimated (UJ), and document in the Data Assessment that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable "R". If holding times were exceeded by more than 28 days, all non-detect data must be qualified "R", unusable.

NOTE: Contractual Holding Times: Extraction of water samples must be started within 5 days VTSR. Soil/sediment samples must be extracted within 10 days of VTSR. This requirement does not apply to Performance Evaluation (PE) samples. Water and soil/sediment extracts must be analyzed within 40 days following

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YES    NO    N/A

extraction.

ACTION: If contractual holding times are exceeded, document in the Data Assessment.

NOTE: The data reviewer must note in the Data Assessment whether or not technical and contractual holding times were met.

**3.0 Surrogate Recovery (Form II)**

3.1 Are BNA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water?

[ ]    —    —

b. Low Soil?

[ ]    —    —

c. Med Soil?

[ ]    —    —

3.2 Are all the BNA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

a. Low Water?

[ ]    —    —

b. Low Soil?

[ ]    —    —

c. Med Soil?

ACTION: Contact the TOPO to request an explanation or resubmittal of any missing deliverables from the laboratory. If missing deliverables are unavailable, document the effect in the Data Assessment.

[ ]    —    —

3.3 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with red pencil.

3.4 Were two or more base-neutral OR acid surrogate recoveries out of specification for any sample or method blank?

[ ]    —    —

If yes, were samples reanalysed?

Were method blanks reanalysed?

—    [ ]    —

ACTION: If all BNA surrogate recoveries are  $\geq 10\%$ , but two within the base-neutral or acid fraction do not meet SOW specifications, for the affected fraction only (i.e. acid or base-neutral compounds):

[ ]    —    —

1. Flag all positive results as estimated (J).

[ ]    —    —

2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.

3. Do not qualify non-detects if recoveries are greater than the upper acceptance limit.

ACTION: If any base-neutral or acid surrogate has a recovery of  $< 10\%$ :

1. Qualify positive results for that fraction as estimated (J).

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YES NO N/A

2. Qualify non-detects for that fraction as unusable (R).

Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Also check the internal standard areas.

NOTE: Contractual requirements state that if two surrogate fails acceptance criteria, within the same fraction, i.e. Acid or BN, the sample must be re-analyzed. If sample was not re-analyzed, document in the Data Assessment under Contract Problems/Non-Compliance.

NOTE: The laboratory must submit the following data:

1. If surrogate recoveries and internal standard responses meet the acceptance criteria in the re-analyzed sample, then the laboratory must submit only the re-analysis.

2. If surrogate recoveries and/or internal standard responses fail to meet the acceptance criteria upon re-analysis, then submit data from both analyses.

3.5 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, contact the TOPO to request an explanation or resubmittal of corrected deliverables from the laboratory. Make necessary corrections and note errors in the Data Assessment.

**4.0 Matrix Spikes (Form III)**

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water?

b. Low Soil?

c. Med Soil?

\_\_\_ [ ] \_\_\_

[ ] \_\_\_ \_\_\_

[ ] \_\_\_ \_\_\_

[ ] \_\_\_ \_\_\_

[ ] \_\_\_ \_\_\_

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

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YES NO N/A

ACTION: No action is taken based upon MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

ACTION: Circle all outliers with red pencil.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present?

5.2 Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each time samples are extracted?

5.3 Has a BNA method blank been analyzed for each GC/MS system used? (See SOW pg. D-55/SVOA, Section 12.1.2.)

ACTION: If any method blank data are missing, contact the TOPO to obtain an explanation/resubmittal from the lab. If resubmittals are unavailable, use professional judgement to determine if the associated sample data should be qualified.

5.4 The validator should verify that the correct identification scheme for the EPA Blank samples were used. See page B-30, sec. 3.3.7.3 of the SOW for further information.

Was the correct identification scheme used for all BNA blanks?

ACTION: Contact the TOPO to obtain resubmittals from the lab or make the required corrections on the forms. Document all corrections made by the validator in the Data Assessment under Contract Problems/Non-Compliance.

5.5 Chromatography: review the blank raw data - chromatograms (RICs), quant. reports or data system printouts and spectra. Is the chromatographic performance (baseline stability) acceptable for each instrument?

ACTION: Use professional judgement to determine the effect on the data.

5.6 Are all detected hits for target compounds less than the CRQL for that analyte in all method blanks?

Exception: Phthalate esters must be less than five times (5x) the CRQL.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/reagent blanks have positive results (TCL and/or TIC)?

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YES    NO    N/A

NOTE:    Water: When applied as directed in the table below (page 33), the contaminant concentration in method/ instrument/reagent blanks is multiplied by the sample dilution factor, where necessary.

Soil: If the lab has not already done so, the contaminant concentration in soil blanks is multiplied by 33 times the sample dilution factor and corrected for %moisture (fraction of solid) where necessary. 30 grams of sodium sulfate (1 gram for medium level soils) are used to prepare the soil reagent/method blank as instructed on page D-54/SVOA, section 12.1.3. Contact the TOPO to obtain resubmittals if the soil blanks are not reported in soil units (µg/kg).

6.2    Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?

ACTION: Prepare a list of samples associated with each contaminated blank. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify sample data. Do not convert field blank results to account for the difference in soil CRQLs. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, spectral, instrument performance, calibration and Internal standard QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable "R".

<b>For:</b>	<b>Flag sample result with a "U" when:</b>	<b>Report CRQL &amp; qualify "U" when:</b>	<b>No qualification is needed when:</b>
-------------	--	--	---

Common Phthalate-Esters	Sample conc. is > CRQL, but ≤ 10× blank value.	Sample conc. is < CRQL and ≤ 10× blank value.	Sample conc. is > CRQL and > 10× blank value.
-------------------------	--	---	---

<b>For:</b>	<b>Flag sample result with a "U" when:</b>	<b>Report CRQL &amp; qualify "U" when:</b>	<b>No qualification is needed when:</b>
-------------	--	--	---

Other Contaminants	Sample conc. is > CRQL, but ≤ 5× blank value.	Sample conc. is < CRQL and ≤ 5× blank value.	Sample conc. is > CRQL and > 5× blank value.
--------------------	---	--	--

NOTE: Analytes qualified "U" for blank contamination are still treated as "hits" when qualifying for calibration criteria.

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YES    NO    N/A

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample?

ACTION: For low level samples, note in the Data Assessment that there is no associated field/rinse/equipment blank. For analytes with high concentration, use professional judgement on qualification of these values and make a note in the Data Assessment.

[ ]    —    —

Exception: samples taken from a drinking water tap do not have associated field blanks.

**7.0 GC/MS Instrument Performance Check**

7.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift?

7.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?

[ ]    —    —

ACTION: List date, time, instrument ID, and sample number for which no associated GC/MS tuning data are valid.

[ ]    —    —

[ ]    —    —

SAMPLE NUMBERS	DATE	TIME	INSTRUMENT ID
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If the TOPO cannot obtain missing data from the lab, reject "R" all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 198 (see SOW, page D-62/SVOA)?

[ ]    —    —

NOTE: All ion abundance ratios must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may up to 110% that of m/z 198.

ACTION: If mass assignment is in error, flag all associated sample data as unusable "R".

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		YES	NO	N/A
7.5	Have the ion abundance criteria been met for each instrument used?			
ACTION:	List all data which do not meet ion abundance criteria (attach a separate sheet).	[ ]	___	___
ACTION:	If ion abundance criteria are not met, the Region II TPO must be notified.			
7.6	Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values, but if errors are found check more.)			
7.7	Is the number of significant figures for the reported relative abundances consistent with the number given for each ion in the ion abundance criteria column?	___	[ ]	___
ACTION:	If large errors exist, take action as specified in section 3.5 above.			
7.8	Is the mass spectrum of DFTPP acquired according to sec. 9.2.4.2 p. D-20/SVOA ?	[ ]	___	___
NOTE:	Sec. 9.2.4.2 states that "the mass spectrum of DFTPP MUST be acquired in the following manner: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and MUST be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. Do NOT subtract part of the DFTPP peak." See Attachment 3 for DFTPP criteria.	[ ]	___	___
ACTION:	If not, then reject "R" all data analyzed under that particular tune.			
7.9	Are the spectra of the mass calibration compound acceptable?			
ACTION:	Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.			
		[ ]	___	___
<b>8.0 Target Compound List (TCL) Analytes (FORM I SV)</b>				
8.1	Are the Organic Analysis Data Sheets (Form I SV) present with required header information on each page, for each of the following:			
	a. Samples and/or fractions as appropriate?			
	b. Matrix spikes and matrix spike duplicates?	[ ]	___	___
	c. Blanks?	[ ]	___	___
8.2	Has GPC cleanup been performed on all soil/ sediment sample extracts?	[ ]	___	___
	Has the Laboratory provided the TWO most recent			
	UV traces of the	[ ]	___	___

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		YES	NO	N/A
calibration solution? GPC 39/SVOA,sec.10.3.4.4)	calibration and GPC CCV?(D-	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION:	If data suggests that GPC was not performed, use professional judgement. Make note in Contract Problems/Non-Compliance section of the Data Assessment.			
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (quant. reports) included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?			
	b. Matrix spikes and matrix spike duplicates (mass spectra not required)?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	c. Blanks?			
ACTION:	If any data are missing, take action specified in 3.2 above.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8.4	Is chromatographic performance acceptable with respect to:	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	Baseline stability?			
	Resolution?			
	Peak shape?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	Full-scale graph (attenuation)?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	Other: _____?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION:	Use professional judgement to determine the acceptability of the data.			
8.5	Are lab-generated standard mass spectra of identified BNA compounds present for each sample?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION:	If any mass spectra are missing, take action specified in 3.2 above. Note under Contract Non-compliance if lab does not generate their own standard spectra. If spectra are missing, reject all positive data.			
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?			
8.7	Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?			
8.8	Do sample and standard relative ion intensities agree within ±20%?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION:	Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected "R", flagged "N" (presumptive evidence of the presence of the	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

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		YES	NO	N/A
	compound) or changed to not detected "U" at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 8.6, 8.7, and 8.8.	[ ]	—	—
	ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.			
	NOTE : The maximum dilution factor permitted for low level soils is 30. If a low level soil sample requires a dilution greater than 30, then the medium level method shall be utilized.(p. D-43) SVOA, sec. 10.6.5.4 . Document in the Data Assessment under Contract Problems/Non compliance if any low level soil was analyzed at a dilution factor greater than 30.			
<b>9.0 Tentatively Identified Compounds (TIC)</b>				
9.1	Are all Tentatively Identified Compound Forms (Form I, Part B) present? and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?			
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?			
	b. Blanks?			
	c. Are Alkanes listed in/or part of the Case Narrative?			
	ACTION: If any TIC data are missing, take action specified in 3.2 above.			
	ACTION: Add "N" qualifier to all chemically named TIC's, if missing.	[ ]	—	—
		[ ]	—	—
		[ ]	—	—
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds? (Example: 1,2-dimethylbenzene is xylene - a VOA TCL - and should not be reported as a TIC.)	[ ]	—	—
	ACTION: Flag with "R" any TCL compound detected in another fraction. (Except blank contamination)	[ ]	—	—
9.4	Are any TICs reported earlier than 30 sec. before the first semivolatle compound, or three (3) minutes after the last semivolatle compound listed in Exhibit C SVOA?(p.D-45/SVOA,11.1.2.2)			
	ACTION: <u>Flag with "R" any TIC compound reported.</u>	—	[ ]	—

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		YES	NO	N/A
9.5	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?			
9.6	Do TIC and "best match" standard relative ion intensities agree within ±20%?	[ ]	___	___
		[ ]	___	___
ACTION:	Use professional judgement to determine the acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown," or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R".			
9.7	Are any TICs with responses < 10% of the internal standard (as determined by inspection of the peak areas or height) reported?			
ACTION:	If yes, cross out questionable TIC(s).	___	[ ]	___
<b>10.0 <u>Compound Quantitation and Reported Detection Limits</u></b>				
10.1	Are there any transcription/calculation errors in Form I results? (Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result.)			
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	___	[ ]	___
ACTION:	If errors are large, take action as specified in section 3.5 above.	[ ]	___	___
ACTION:	When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form Is that should not be used, including any in the summary package.			
<b>11.0 <u>Standards Data (GC/MS)</u></b>				
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts (quant. reports) present for initial and continuing calibration?			
ACTION:	If any calibration standard data are missing, take action specified in 3.2 above.			
<b>12.0 <u>GC/MS Initial Calibration (Form VI)</u></b>				
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the			

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		YES	NO	N/A
	BNA fraction?	<input type="checkbox"/>	___	___
		<input type="checkbox"/>	___	___
ACTION:	If any calibration standard forms are missing, take action specified in 3.2 above.			
12.2	Are the % relative standard deviation (%RSD) values for BNAs $\leq$ 30% over the concentration range of the calibration?	<input type="checkbox"/>	___	___
ACTION:	Circle all outliers with red <u>pencil</u> .			
NOTE:	Although <u>25</u> BNA compounds have a contractual minimum RRF and no maximum %RSD, the technical criteria are the same for all analytes.			
NOTE:	Eight BNA compounds do not require a 20ng standard. They are 2,4-Dinitrophenol, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol, and Pentachlorophenol.			
ACTION:	If the %RSD is > 30.0%, qualify only positive results for that analyte "J" and do not qualify non-detects. When %RSD is > 90%, flag all non-detect results for that analyte "R" (unusable) and all positive results "J" (estimated) for ALL samples analyzed under that particular Initial Calibration.			
NOTE:	Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.			
12.3	Are any average RRFs < 0.05?			
ACTION:	Circle all outliers with red <u>pencil</u> .			
ACTION:	If the average RRF is < 0.05 then:			
	1. "R" all non-detects for ALL samples analyzed under that particular Initial Calibration.	___	<input type="checkbox"/>	___
	2. "J" all positive results for ALL samples analyzed under that particular Initial Calibration.			
12.4	Are there any transcription/calculation errors in the reporting of RRFs and/or %RSDs? (Check at least two values; if errors are found check more.)			
ACTION:	Circle errors with red <u>pencil</u> .			
ACTION:	If errors are large, take action as specified in section 3.5 above.			

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YES    NO    N/A

\_\_\_    [ ]    \_\_\_

NOTE: Contract Requirement: The SOW allows up to four of the required analytes to fail contractual %RSD or RRF criteria provided the %RSD is  $\leq$  40% or RRF is  $\geq$  0.010. (See Table 5, page D-67/SVOA and analytes marked with a "\*" on Form VI for a list of required analytes and contractual criteria.) Technical criteria, however, are the same for all analytes.

ACTION: If more than four analytes fail %RSD or RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.

**13.0 GC/MS Continuing Calibration (Form VII)**

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?

[ ]    \_\_\_    \_\_\_

ACTION: List below all sample analyses that were not analyzed within twelve hours of a continuing calibration standard for each instrument used.

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

[ ]    \_\_\_    \_\_\_

ACTION: If any forms are missing, or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, contact the TOPO to obtain an explanation/resubmittal from the lab. If continuing calibration data are unavailable, flag all associated sample data as unusable "R".

13.3 Does any BNA compound have a percent difference (%D) between the initial and continuing calibration RRFs which exceeds the  $\pm 25.0\%$  criteria?

ACTION: Circle all outliers with red pencil.

\_\_\_    [ ]    \_\_\_

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated "J". When %D is  $> 90\%$ , reject all non-detects for that analyte, "R", and qualify positive results "J" (estimated) for ALL samples analyzed under that particular Continuing Calibration.

13.4 Are any continuing RRFs  $< 0.05$ ?

ACTION: Circle all outliers with red pencil.

\_\_\_    [ ]    \_\_\_

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YES    NO    N/A

ACTION: If the RRF is < 0.05, qualify as unusable (R) associated non-detects and "J" associated positive values for ALL samples analyzed under that particular Continuing Calibration.

NOTE: Contract Requirement: The SOW allows up to four of the required analytes to fail contractual %D and RRF criteria, provided that the %D is ≤ 40% and the RRF is ≥ 0.010. (See Table 5 page D-67/SVOA or analytes marked with a "\*" on Form VI for a list of the required analytes.) Technical criteria, however, are the same for all analytes.

ACTION: If more than four analytes failed %D and RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.

13.5 Are there any transcription/calculation errors in the reporting of average relative response factors (RRF) or %difference (%D) between initial and continuing RRFs? (Check at least two values, but if errors are found, check more.)

ACTION: Circle errors with red pencil.

ACTION: If errors are large, take action as specified in section 3.5 above.

**14.0 Internal Standards (Form VIII)**

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?

—    [ ]    —

If no, was sample re-analyzed?

ACTION: 1. Circle all outliers with red pencil.

2. List all the outliers below.

[ ]    —    —

[ ]    —    —

ACTION: If sample was not reanalysed, document in Data Assessment in Contract Problems/Non-Compliance.

Sample #	Internal Std.	Area	Lower/Upper Limit
_____	_____	_____	_____ / _____
_____	_____	_____	_____ / _____
_____	_____	_____	_____ / _____
_____	_____	_____	_____ / _____

(Attach additional sheets if necessary.)  
(or attach copies of Form VIIIs)

ACTION: 1. If the internal standard area count is outside the "upper" or "lower" limit, flag with "J" all positive results and non-detects quantitated with this

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YES NO N/A

internal standard with the following exceptions:

A. Do not qualify non-detects associated with IS area > 100%.

B. If the IS area in the sample is < 50%, qualify all analytes associated with that IS estimated (J). If area counts are extremely low (< 25% of the area in the 12 hour standard), or if performance exhibits a major abrupt drop-off, flag all associated non-detects as unusable (R) and positive hits estimated (J).

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

NOTE: Contractual requirements state that if any internal standard fails the acceptance criteria, the sample must be re-analyzed. If the affected sample was not re-analyzed, document in the Data Assessment under Contract Problems/Non-Compliance.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for BNA analysis?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

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YES    NO    N/A

PART C: PESTICIDE/PCB ANALYSIS

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody Records or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50% - 90% water, all data should be qualified as estimated "J". If a soil sample, other than TCLP, contains more than 90% water, qualify positive results "J" and non-detects "R". \_\_\_ [ ] \_\_\_

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory, and the temperature of the cooler was elevated > 10° C, flag all positive results "J" and all non-detects "UJ".

ACTION: Check aqueous extraction log for sample pH, if adjustment was needed, it should have been noted in the SDG Narrative. If more information is needed, notify the TOPO to contact the lab.

2.0 Holding Times

2.1 Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?

NOTE: Technical Holding Times: Water and soil samples for PEST/PCB analysis must be extracted within 14 days of the date of collection. Extracts must be analyzed within 40 days of the date extraction.

\_\_\_ [ ] \_\_\_

ACTION: If technical holding times are exceeded, flag all positive results as estimated "J" and sample quantitation limits "UJ" and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be qualified "J", but the reviewer may determine that non-detects are unusable "R".

Table of Holding Time Violations  
(See Chain-of-Custody Records)

Sample Analyzed	Sample Matrix	Date Sampled	Date Lab Received	Date Lab Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

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YES    NO    N/A

NOTE: Contractual Holding Times: Extraction of water samples must be completed within 5 days VTSR. Soil/sediment samples must be extracted within 10 days of VTSR. This requirement does not apply to Performance Evaluation (PE) samples. Extracts of water and soil/sediment samples must be analyzed within 40 days following start of extraction.

ACTION: If contractual holding times are exceeded, document in the Data Assessment.

NOTE: The data reviewer must note in the Data Assessment whether or not technical and contractual holding times were met.

**3.0 Surrogate Recovery (Form II)**

3.1 Are the PEST/PCB Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water?

b. Soil?

[ ]    —    —  
[ ]    —    —

3.2 Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:

a. Low Water?

b. Soil?

[ ]    —    —  
[ ]    —    —

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.3 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with red pencil.

[ ]    —    —

3.4 Were surrogate recoveries of TCX or DCB outside of the contract specification for any sample, method blank or sulfur clean-up blank (30-150%)?

—    [ ]    —

ACTION: In the absence of matrix interference, qualification of the data is not required in the following three situations:

1. When surrogates on both columns are diluted out.
2. When one surrogate on one column was outside (either above or below)

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YES    NO    N/A

the contract limits but above 10%.

3. When the same surrogate on both columns is above the contract limit.

ACTION: If the same surrogate on both columns is below the contract limit but above 10%, check chromatograms for interference. The reviewer may use professional judgement, and qualify only those analytes which elute in the region of the GC chromatogram where interference was observed.

ACTION: If the same surrogate on both columns is below the contract limit but above 10% (with no interference), qualify non-detects and positive hits "J" (estimated).

ACTION: If recoveries for both surrogates on both columns are below the contract limit but above 10%, flag positive results and non-detects for that sample "J".

ACTION: If recoveries are above the contract limit for both surrogates on both columns, then qualify positive values "J".

ACTION: If both surrogates on one column are below the contract limit but above 10%, then use the data from the other column, providing both surrogates on that column are within contract limits. The validator must check from which column the concentration is reported for each analyte. If the value is reported from the failed column, then cross it out and use the value from the other column. Document this change in the Data Assessment.

ACTION: If recovery is below 10% for either surrogate on any column, qualify positive results "J" and flag non-detects "R".

3.5 Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A (see Form VI Pest-1)?

ACTION: If the RT limits are not met, positive results and non-detects for that sample may be qualified unusable, "R", based on professional judgement. [ ]    —    —

3.6 Are there any transcription/calculation errors between raw data and Form II?

ACTION: If large errors exist, contact the TOPO to obtain an explanation or resubmittal of corrected deliverables from the laboratory. Make any necessary corrections and document the effect in the Data Assessment. —    [ ]    —

**4.0 Matrix Spikes (Form III)**

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices (one MS/MSD must be performed for every 20 samples of similar matrix or concentration level):

- a. Low Water? [ ]    —    —
- b. Soil?

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		YES	NO	N/A
ACTION:	If any matrix spike data are missing, take the action specified in 3.2 above.	[ ]	___	___
ACTION:	Circle all outliers with red <u>pencil</u> .	[ ]	___	___
ACTION:	No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.			
<b>5.0 <u>Blanks (Form IV)</u></b>				
5.1	Is the Method Blank Summary (Form IV) present?			
5.2	<u>Frequency of Analysis</u> : Has a reagent/method blank been analyzed for each SDG, every 20 samples of similar matrix and concentration level or each extraction batch, whichever is more frequent?	[ ]	___	___
ACTION:	If any blank data are missing, take action as specified above in section 3.2. If blank data is not available, reject "R" all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.			
5.3	A separate Form IV should be present if part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV submitted when required?			
ACTION:	If sulfur clean-up blank data and Form IV are missing, take action as specified in 3.2 above.			
5.4	Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)?	[ ]	___	___
ACTION:	If any blank data are missing, take action as specified in section 3.2 above.	[ ]	___	___
5.5	Was the correct identification scheme used for all Pest/PCB blanks? (See page B-30, sec. 3.3.7.3 of the SOW for further information.)	[ ]	___	___
ACTION:	Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.			
5.6	<u>Chromatography</u> : review the blank raw data - chromatograms, quant. reports			

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and data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable?				
ACTION: Use professional judgement to determine the effect on the data.				
6.0	<u>Contamination</u>	<input type="checkbox"/>	___	___
NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are <u>not</u> used to qualify the data. Do not confuse them with the other QC blanks discussed below.				
6.1	Do any method/reagent, instrument, or cleanup blanks show positive hits for pest/PCBs?			
6.2	If any method blanks and/or sulfur clean-up blanks contain "hits" for target compounds, are these hits greater than the CRQL for that analyte?	___	<input type="checkbox"/>	___
6.3	In any instrument blanks, is the concentration of any target hit > 0.5 times CRQL for that analyte (see SOW, section <u>12.1.4.3.3</u> , page <u>D-73</u> /PEST)?	___	<input type="checkbox"/>	___
NOTE: Most labs will report 0.5 times CRQLs on the instrument blank Form I instead of the actual method CRQLs. If the lab reported the actual CRQLs, then check if any detected hits are above 0.5 times the CRQLs reported on the Form I.				
ACTION: If yes to any of the above questions: note in the Data Assessment under Contract Problems/Non-Compliance if any method or clean-up blanks contain hits > the CRQL, or of instrument blank contained hits > 0.5 times CRQL for that analyte.				
6.4	Do any field/rinse blanks have positive pest/PCB results?	___	<input type="checkbox"/>	___
ACTION: Prepare a list of the samples associated with each contaminated blank. (Attach a separate sheet)				
NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Do not convert <u>field blank</u> results to account for the difference in soil CRQLs. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, and/or calibration QC problems.				
ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.				
NOTE: When applied as directed in the table below, the contaminant concentration in method/instrument/ reagent/cleanup blanks is multiplied by the sample dilution factor, where necessary.				

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YES    NO    N/A

If the laboratory has not already done so, the contaminant concentration in soil blanks is multiplied by 33 times the sample dilution factor and corrected for %moisture (fraction of solid) where necessary. 30 grams of sodium sulfate are used to prepare each soil reagent/method blank as instructed on page D-69/PEST, section 12.1.2.3.1. Ask the **TOPO** to contact the laboratory if the soil blanks are not reported in soil units (µg/kg).

<b>Flag sample result with a "U":</b>	<b>Report CRQL &amp; qualify "U":</b>	<b>No qualification is needed:</b>
---------------------------------------	---------------------------------------	------------------------------------

Sample conc. > CRQL, but ≤ 5× blank.	Sample conc. < CRQL & is ≤ 5× blank value.	Sample conc. > CRQL & > 5× blank value.
--------------------------------------	--	---

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as "R", unusable.

6.5 Are there field/rinse/equipment blanks associated with every sample? [ ]    \_\_\_    \_\_\_

ACTION: For low level samples, note in the Data Assessment that there is no associated field/rinse/equipment blank. For analytes with high concentrations, use professional judgement to qualify these values and document in the Data Assessment.

Exception: samples taken from a drinking water tap do not have associated field blanks.

**7.0 Calibration and GC Performance**

- |     |  |     |     |     |
|-----|--|-----|-----|-----|
| 7.1 | Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks and MS/MSD:              |     |     |     |
|     | a. Peak resolution check?  |     |     |     |
|     | b. Performance evaluation mixtures?  |     |     |     |
|     | c. Aroclor 1016/1260?  | [ ] | ___ | ___ |
|     | d. Aroclors 1221, 1232, 1242, 1248, 1254?  | [ ] | ___ | ___ |
|     | e. Toxaphene?  | [ ] | ___ | ___ |
|     | f. Low points individual mixtures A & B?   | [ ] | ___ | ___ |
|     | g. Med points individual mixtures A & B?   | [ ] | ___ | ___ |
|     | h. High points individual mixtures A & B?  | [ ] | ___ | ___ |
|     | i. Instrument blanks?  | [ ] | ___ | ___ |
|     | j. Were the appropriate GC columns used as specified on pg. <u>D-10</u> /PEST, sections 6.23.3 to 6.23.3.7, in the SOW?                  | [ ] | ___ | ___ |
| 7.2 | Do the chromatograms for all Individual Standard Mixtures and PEM analyses display single component analytes at > 10% but < 100% of full |     |     |     |

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		YES	NO	N/A
	scale (see sections 9.3.5.8.1 thru 9.3.5.8.4, pages <u>D-30 &amp; 31</u> /PEST)?	[ ]	___	___
	Have chromatograms for Individual Standard Mixtures and PEM analyses been replotted, showing scaling factor(s), to meet the above requirements when necessary?			
NOTE:	All standard chromatograms must clearly display all peaks at > 10% but < 100% of full scale, and replotted if necessary to accommodate peaks not properly scaled in the initial chromatogram(s). Both the initial and replotted chromatograms must be submitted with the data package.	[ ]	___	___
ACTION:	If all single component peaks are not clearly displayed on chromatograms for all Individual Standard Mixtures and PEM analyses, notify the <b>TOPO</b> to obtain resubmittal of the necessary data.	[ ]	___	___
7.3	Are Forms VI PEST 1-7 present and complete for each column and each analytical sequence?			
ACTION:	If no, take action as specified in 3.2 above.			
		[ ]	___	___
7.4	Are there any transcription/ calculation errors between raw data and Forms VI?	___	[ ]	___
ACTION:	If large errors exist, take action as specified in section 3.2 above.			
7.5	Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the Initial Calibration (see Form VI PEST-1)?			
ACTION:	If no, all samples in the entire analytical sequence are potentially affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results "JN" and non-detects as unusable (R). For Aroclors, the RT may be outside the window, but the Aroclor may still be identified from its distinctive pattern.	[ ]	___	___
7.6	Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns? (%RSD must be ≤ 25.0 for alpha and delta BHC, ≤ 30.0 for the two surrogates and ≤ 20% for all other analytes.)			
NOTE:	Contractual requirements allow up to two single component TCL compounds, but not surrogates, on each column to exceed the criteria provided the %RSD is ≤ 30%. (See page <u>D-26</u> /Pest, sec. 9.2.5.7 in the SOW.) Technical			

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	YES	NO	N/A
criteria, however, are the same for all analytes.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION: If technical criteria were not met, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When %RSD > 90%, flag all non-detect results for that analyte "R" (unusable), and positive results as "J" estimated.			
ACTION: If more than two analytes failed %RSD, document in the Data Assessment Contract Problems/Non-Compliance section.			
7.7 Is the resolution between each pair of adjacent peaks in the Resolution Check Mixture ≥ 60.0% for both columns? (See Form VI PEST-4.)(D-25/Pest)			
ACTION: If no, qualify positive results for compounds that were not adequately resolved "J". Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" as presumptive evidence of presence or unusable (R).	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
7.8 Is Form VI PEST-5 present and complete for each Performance Evaluation Mixture (PEM) standard used for <u>both initial and continuing</u> calibrations (see SOW section 3.12.4.4, page <u>B-48</u> )?			
ACTION: If no, take action as specified in section 3.2 above.			
7.9 For each PEM standard, was the resolution between each pair of adjacent peaks ≥ 90.0% on both columns?			
ACTION: Qualify positive results for compounds not adequately resolved estimated (J). Qualify non-detects based on professional judgement.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
7.10 Have Forms VI PEST-6 & PEST-7 been completed for all midpoint Individual Standards A and B used for initial calibration?			
For each standard, was the resolution between each pair of adjacent peaks ≥ 90.0% on both columns?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
ACTION: If no, qualify positive results for compounds that were not adequately resolved estimated (J). Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" as presumptive evidence of presence or unusable "R".			
7.11 Is Form VII Pest-1 present and complete for each PEM standard analyzed during the analytical sequence for both columns?	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
NOTE: If a PEM or Individual std mixture does not meet technical criteria listed on sec. 9.3.5.8.1 through 9.3.5.8.4, it MUST be reinjected immediately. If the second injection meets the criteria, sample analysis may continue. Otherwise, ALL data collection MUST BE STOPPED. Document it in the Data Assessment under Contract Problems/Non compliance.(p. D-31/Pest, sec. 9.3.6.4).	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Was the %Breakdown of DDT and Endrin calculated using the equations given on page <u>D-24</u> /PEST, sec. 9.2.4.8 in the SOW?			
Were all pesticides and surrogates in each PEM standard within the RT			

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		YES	NO	N/A
	windows established during the Initial Calibration?	<input type="checkbox"/>	___	___
		<input type="checkbox"/>	___	___
		<input type="checkbox"/>	___	___
ACTION:	If no, take action as specified in 3.2 above.			
7.12	Has the individual percent breakdown for DDT/Endrin exceeded 20.0% in any PEM on either column? (See Form VII PEST-1.)			
	- for 4,4'-DDT?	___	<input type="checkbox"/>	___
	- for Endrin?	___	<input type="checkbox"/>	___
	Has the combined percent breakdown for DDT/Endrin exceeded 30.0% in any PEM on either column (required for all PEM analyses)?	___	<input type="checkbox"/>	___
ACTION:	1. If any percent breakdown has failed the QC criteria in either PEM in steps 2 and 17 in the <u>initial calibration</u> sequence (page D-21/Pest, sec. 9.2.3.4 in the SOW), qualify <u>all samples</u> in the entire analytical sequence as described in sections 2.a, b and c below.			
	2. If any percent breakdown failed the QC criteria in a PEM <u>calibration verification</u> analysis, review data beginning with the samples which followed the last in-control standard until the next acceptable PEM and qualify the data as described below.			
	a. <u>4,4'-DDT Breakdown</u> : If DDT breakdown was > 20.0%:			
	I. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT unusable, "R".			
	ii. Qualify positive results for DDD and/or DDE as presumptively present at an approximated quantity "JN".			
	b. <u>Endrin Breakdown</u> : If endrin breakdown was > 20.0%:			
	I. Qualify all positive results for endrin with "J". If endrin was			

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not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for Endrin as unusable "R".

ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity "JN".

c. Combined Breakdown: If the combined 4,4'-DDT and endrin breakdown is greater than 30.0%:

I. Qualify all positive results for DDT and Endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable "R". If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable "R".

ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity "JN". Qualify positive results for DDD and/or DDE as presumptively present at an approximated quantity "JN".

7.13 Are all percent difference (%D) values for PEM analytes and surrogates on both columns  $\geq -25\%$  and  $\leq +25.0\%$ ? (See Form VII PEST-1.)

ACTION: If no, qualify all associated positive results generated during the analytical sequence "J" and sample quantitation limits "UJ".

NOTE: If the failing PEM is part of the initial calibration, all samples are potentially affected. If the offending standard is a calibration verification, the associated samples are those which followed the last in-control standard until the next passing standard.

7.14 Is Form VII Pest-2 present and complete for each INDA and INDB calibration verification analyzed?

ACTION: If no, take action specified in 3.2 above.

7.15 Are there any transcription/calculation errors between raw data and Form VII Pest-2?

ACTION: If large errors exists, take action as specified in section 3.6 above.

7.16 Do all standard retention times for each INDA and INDB calibration verification fall within the RT windows established during the initial calibration sequence? (See Form VII PEST-2.)

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.17 Are all %D values for INDA and INDB calibration verification compounds  $\geq$

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-25.0% and ≤ +25.0%?

ACTION: If the %D is outside the ±25.0% range for any compound(s), qualify associated positive results for that compound "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte(s) in question. If the %D is > 90%, flag all non-detects for that analyte "R" (unusable).

8.0 Analytical Sequence Check (Form VIII-PEST)

8.1 Is Form VIII present and complete for each column and each period of analyses?

ACTION: If no, take action specified in 3.2 above.

8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses, and all standards analyzed at the required frequency for each GC/ECD instrument used.? (See SOW pages D-21 & D-55/PEST.)

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered and/or the calibration was out of QC limits.

8.3 Were all samples analyzed within a 12 hour time period beginning with the injection of an instrument blank and bracketed by acceptable analyses of the proper standards?

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly. Document in the Data Assessment under Contract Problems/Non-Compliance.

8.4 If a multi-component analyte was detected in a sample, was a matching multi-component standard analyzed within 72 hours of the injection of the sample and within a valid 12 hour sequence?

NOTE: This additional standard is for identification purposes only. Positive results for Aroclors and Toxaphene are quantitated from the initial calibration.

ACTION: If no, document in the Data Assessment under Contract Problems/Non-Compliance.

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	YES	NO	N/A
<b>9.0 <u>Cleanup Efficiency Verification (Form IX)</u></b>			
<p>9.1 Is Form IX PEST-1 present and complete for each lot of Florisil Cartridges used? (Florisil Cleanup is required for <u>all Pest/PCB extracts.</u>)</p> <p>Are all samples listed on the Pesticide Florisil Cartridge Check Form?</p>	<p><input type="checkbox"/></p> <p><input type="checkbox"/></p>	<p>___</p> <p>___</p>	<p>___</p> <p>___</p>
<p>ACTION: If no, take action specified in 3.2 above. If data suggests florisil clean-up was not performed, document in the Data Assessment under the Contract Non-compliance section.</p>			
<p>9.2 Are percent recoveries (%REC) of the pesticide and surrogate compounds used to check the efficiency of the florisil clean-up procedure within QC limits of 80 - 120%?</p>	<p><input type="checkbox"/></p>	<p>___</p>	<p>___</p>
<p>ACTION: Qualify only the analyte(s) which failed the recovery criteria as follows:</p> <p style="margin-left: 40px;">If %REC is &lt; 80%, qualify positive results "J" and non-detects "UJ".</p> <p style="margin-left: 40px;">If any pesticide %REC was zero, flag non-detects "R" for that compound.</p> <p style="margin-left: 40px;">Use professional judgement to qualify positive results if any recoveries are &gt; 120%.</p>			
<p>NOTE: Sample data should be evaluated for potential interferences if recovery of 2,4,5-trichlorophenol was &gt; 5% in the Florisil Cartridge Performance Check analysis. Document any problems found in the Data Assessment under the Contract Problems/Non-Compliance section.</p>			
<p>9.3 If GPC Cleanup was performed (mandatory for all soil sample extracts), is Form IX Pest-2 present?</p> <p>Are all soil samples listed on Form IX Pest-2?</p>	<p><input type="checkbox"/></p> <p><input type="checkbox"/></p>	<p>___</p> <p>___</p>	<p>___</p> <p>___</p>
<p>ACTION: If no, take action specified in 3.2 above. If data suggests GPC clean-up was not performed when required, document in the Data Assessment under the Contract Problems/Non-Compliance section.</p> <p>Are the %REC values for all pesticides in the GPC calibration solution between 80 - 110%?</p>			
<p>ACTION: Qualify only those analytes which failed the recovery criteria as follows:</p> <p style="margin-left: 40px;">If %REC are &lt; 80%, qualify positive results "J" and non-detects "UJ".</p> <p style="margin-left: 40px;">If any pesticide %REC was zero, flag non-detects "R" for that</p>	<p><input type="checkbox"/></p>	<p>___</p>	<p>___</p>

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YES    NO    N/A

compound.

Use professional judgement to qualify positive results if any recoveries are > 110%.

NOTE: An Aroclor mixture containing Aroclors 1016 and 1260 is also analyzed during GPC calibration; however, Aroclor data is not listed on Form IX PEST-2. The raw GPC data for Aroclors 1016/1260 must be evaluated for pattern similarity with previously analyzed Aroclor standards.

9.4 The validator should verify that the correct identification scheme for the EPA Blank samples were used. See page B-30, sec. 3.3.7.2 and 3.3.7.9 of the SOW for further information.

Was the correct identification scheme used for GPC and Florisil blanks?

**10.0 Pesticide/PCB Identification**

10.1 Is Form X complete for every sample in which a pesticide or PCB was detected? [ ]    \_\_\_    \_\_\_

ACTION: If no, take action specified in 3.2 above.

10.2 Are all sample chromatograms properly scaled, attenuated, etc. as required for proper identification of single and multi-component analytes? (Refer to SOW sections 11.3.7.1 thru 11.3.7.8, page D-67/Pest for specific details.) [ ]    \_\_\_    \_\_\_

NOTE: Proper verification of Pest/PCB results depends on clear, legible presentation of the raw data. Single component pesticides and all peaks chosen for quantitation of multi-component analytes must appear at less than full scale. Toxaphene and PCB patterns must be clearly visible to enable comparison with standard chromatograms. [ ]    \_\_\_    \_\_\_

ACTION: If retention times or apex of peaks cannot be verified, or if multi-component peak patterns cannot be discerned, contact the WAM to obtain rescaled chromatograms from the lab.

10.3 Are there any transcription/calculation errors between raw data and Forms 10A and 10B?

ACTION: If large errors exist, take action as specified in section 3.2 above. \_\_\_    [ ]    \_\_\_

10.4 Are RTs of sample compounds within the established RT windows for analyses on both columns? [ ]    \_\_\_    \_\_\_  
Was GC/MS confirmation provided when required (when compound

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YES NO N/A

concentration is > 10 ug/ml in the final extract)?

ACTION: Use professional judgement to qualify positive results which were not confirmed by GC/MS analysis. Qualify as unusable (R) all positive results which were not confirmed on a second GC column. Also qualify as unusable (R) all positive results which do not meet RT window criteria, unless associated standard compounds are similarly biased. Use professional judgement to assign an appropriate quantitation limit.

10.5 Is the percent difference (%D) calculated for the positive sample results on both columns > 25.0%?

ACTION: If the reviewer finds neither column shows interference for the positive hits, the Pesticide data should be flagged as follows:

<u>% Difference</u>	<u>Qualifier</u>
0 - 25%	None
26 - 70%	"J"
71 - 100%	"JN"
100 - 200% (No Interference)	"R"
100 - 200% (Interference detected)*	"JN"
> 50% (Pesticide value is < CRQL)**	"U"
> 200%	"R"

\* When the reported %D is 100- 200%, but interference is detected on either column, qualify the data with "JN".

\*\* When the reported pesticide value is lower than the CRQL, and the %D is > 50%, raise the value to the CRQL and qualify "U", undetected.

NOTE: For Aroclors, if the %D is > 50%, but the pattern of GC peaks on both columns indicates a specific Aroclor is present, qualify that Aroclor "J".

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the Data Assessment.

10.6 Check chromatograms for false negatives, especially the multiple-peak compounds (Toxaphene and the PCBs). Were there any false negatives?

ACTION: Use professional judgement to decide if the compound should be reported. If the appropriate PCB standards were not analyzed within 72 hrs. of the sample(s) in question, qualify the data unusable "R".

Also note in Data Assessment under Contract Problems/Non-Compliance if the lab failed to analyze Aroclor standards when required.

11.0 Target Compound List (TCL) Analytes

11.1 Are the Organic Analysis Data Sheets (Form I Pest) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate?

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		YES	NO	N/A
	b. Matrix spikes and matrix spike duplicates?	[ ]	___	___
	c. Blanks?	[ ]	___	___
	d. Instrument Blanks (per column & analysis)?	[ ]	___	___
11.2	Are the Pest chromatograms and quant. reports included in the sample data package for each of the following:			
	a. Samples and/or fractions as appropriate?	[ ]	___	___
	b. Matrix spikes and matrix spike duplicates?	[ ]	___	___
	c. Blanks?	[ ]	___	___
	d. Instrument Blanks (per column & analysis)?	[ ]	___	___
ACTION:	If any data are missing, take action specified in 3.2 above.	[ ]	___	___
11.3	Is chromatographic performance acceptable with respect to:			
	a. Baseline stability?			
	b. Resolution?			
	c. Peak shape?	[ ]	___	___
	d. Full-scale graph attenuation?	[ ]	___	___
	e. Other: _____?	[ ]	___	___
		[ ]	___	___
11.4	Were any electropositive displacement (negative peaks) or unusual peaks seen?	___	[ ]	___
ACTION:	Use professional judgement to determine the acceptability of the data. Address comments under System Performance section of the Data Assessment.			

**12.0 Compound Quantitation and Reported Detection Limits**

12.1	Are there any transcription/calculation errors in Form I results? Check at least two positive results. Were any errors found?	___	[ ]	___
------	---	-----	-----	-----

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. Use professional judgement to decide whether a large discrepancy indicates the presence of an interfering compound. If an interfering compound is visible on the chromatogram, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity "JN". This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has interfered with the evaluation of the second column confirmation.

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YES NO N/A

12.2 Are the CRQLs adjusted to reflect sample dilutions?

ACTION: If large errors exist, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample). Replace concentrations which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer may offer an approximated quantitation limit (UJ) for each affected compound.

NOTE: If a sample required greater than a 10 times dilution, then a 10 times more concentrated analysis must also be performed and submitted (see SOW, page D-57/PEST, section 10.2.3.5).

ACTION: If a more concentrated analysis is unavailable, document in the Contract Problems/Non-Compliance section of the Data Assessment. Use professional judgement to qualify non-detects and positive hits below the CRQL.

13.0 Field Duplicates

13.1 Were any field duplicates submitted?

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

ATTACHMENT 1

CLP Data Assessment

CLP DATA ASSESSMENT

Functional Guidelines for Evaluating Organic Analysis

CASE No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_ LABORATORY: \_\_\_\_\_  
SITE: \_\_\_\_\_

DATA ASSESSMENT

The current SOP No. HW-6 (Revision 12), January 2000 for CLP Organics Review and Preliminary Review has been applied.

All data were found to be valid and acceptable except those analytes which have been rejected, "R" (unusable). Due to various QC problems some analytes may have been qualified with a "J" (estimated), "N" (presumptive evidence for the presence of the material), "U" (non-detect), or "JN" (presumptive evidence for the presence of the material at an estimated value) flag. All action is detailed on the attached sheets.

The "R" flag means that the associated value is unusable. In other words, significant data bias is evident and the reported analyte concentration is unreliable.

Reviewer's  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Verified By: \_\_\_\_\_ Date: \_\_\_\_\_

## CLP DATA ASSESSMENT

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

2. SURROGATES:

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

3. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for additional qualification of data.

4. BLANK CONTAMINATION:

## CLP DATA ASSESSMENT

Quality assurance (QA) blanks, i.e., method, trip, field, or rinse blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field and rinse blanks measure cross-contamination of samples during field operations. If the concentration of the analyte is less than 5 times the blank contaminant level (10 times for common contaminants), the analytes are qualified as non-detects, "U". The following analytes in the sample shown were qualified with "U" for these reasons:

A) Method blank contamination:

B) Field or rinse blank contamination:

C) Trip blank contamination:

### 5. MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for volatile organics is (BFB) Bromofluorobenzene and for semi-volatiles Decafluorotriphenyl-phosphine (DFTPP).

If the mass calibration is in error, all associated data will be classified as unusable "R".

### 6. CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) Response Factor GC/MS:

The response factor measures the instrument's response to specific chemical compounds. The response factor for the Target Compound List (TCL) must be  $\geq 0.05$  in both initial and continuing calibrations. A value  $< 0.05$  indicates a serious detection and quantitation problem (poor sensitivity). Analytes detected in the sample will be qualified as estimated, "J". All non-detects for that compound will be rejected "R".

## CLP DATA ASSESSMENT

### 7. CALIBRATION:

#### B) Percent Relative Standard Deviation (%RSD) and Percent Difference (%D):

Percent RSD is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent D compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent D is a measure of the instrument's daily performance. Percent RSD must be < 30% and %D must be < 25%. A value outside of these limits indicates potential detection and quantitation errors. For these reasons, all positive results are flagged as estimated, "J" and non-detects are flagged "UJ". If %RSD and %D grossly exceed QC criteria, non-detects data may be qualified "R".

For the PEST/PCB fraction, if %RSD exceeds 20% for all analytes, for alpha and delta BHC 25%, and for the two surrogates (which must not exceed 30% RSD), qualify all associated positive results "J" and non-detects "UJ".

The following analytes in the sample shown were qualified for %RSD and %D:

### 8. INTERNAL STANDARDS PERFORMANCE GC/MS:

Internal standards (IS) performance criteria ensure that the GC/MS sensitivity and response are stable during every experimental run. The internal standard area count must not vary by more than a factor of 2 (-50% to +100%) from the associated continuing calibration standard. The retention time of the internal standard must not vary more than ±30 seconds from the associated continuing calibration standard. If the area count is outside the (-50% to +100%) range of the associated standard, all of the positive results for compounds quantitated using that IS are qualified as estimated, "J", and all non-detects as "UJ", or "R" if there is a severe loss of sensitivity.

If an internal standard retention time varies by more than 30 seconds, the reviewer will use professional judgement to determine either partial or total rejection of the data for that sample fraction.

### 9. COMPOUND IDENTIFICATION:

#### A) Volatile and Semi-Volatile Fractions:

### CLP DATA ASSESSMENT

TCL compounds are identified on the GC/MS by using the analyte's relative retention time (RRT) and by comparison to the ion spectra obtained from known standards. For the results to be a positive hit, the sample peak must be within  $\pm 0.06$  RRT units of the standard compound and have an ion spectra which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. For the tentatively identified compounds (TIC) the ion spectra must match accurately. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.

B) Pesticide Fraction:

The retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns and a GC/MS confirmation is required if the concentration exceeds 10ng/ml in the final sample extract.

10. CONTRACT PROBLEMS NON-COMPLIANCE:

11. FIELD DOCUMENTATION:

12. OTHER PROBLEMS:

13. This package contains reextractions, reanalyses or dilutions. Upon reviewing the QA results, the following Form 1(s) are identified not to be used.

CLP DATA ASSESSMENT

ATTACHMENT 2

BFB ION ABUNDANCE CRITERIA

BFB KEY IONS AND ION ABUNDANCE CRITERIA

MASS ION	ABUNDANCE CRITERIA
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95 (see note)
173	less than 2.0 percent of mass 95
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

**NOTE:** All ion abundance must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120.0 percent that of m/z 95.

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

MASS ION	ABUNDANCE CRITERIA
51	30.0 to 80.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Mass 69 relative abundance
70	Less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100% of relative abundance
199	5.0 to 9.0 percent of mass 198
275	10.0 to 30.0 of mass 198
365	Greater than 0.75% of mass 198
441	Present, but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

**NOTE:** All ion abundance must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 120.0 percent that of m/z 198.