



State of Ohio  
Environmental Protection Agency

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**Division of Emergency and Remedial Response**

# **LABORATORY AND FIELD SCREENING DATA REVIEW**

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**DERR-DI-00-034**

**August 19, 2005**

## Laboratory Data Review

### I) PURPOSE:

This guidance and checklist are designed to assist project coordinators, site assessors and voluntary action program reviewers to evaluate whether data provided by a laboratory may contain deficiencies which could result in a determination that the data quality is questionable for use in a project. It is designed to evaluate laboratory quality control processes which could effect the data processing within the laboratory. Each question has a guidance step to guide a reviewer to corrective action steps required if a deficiency is noted during the review. Please note this is not an in depth data validation process, but an initial review of data to determine if errors exist (*i.e.*, data verification).

### II) INTRODUCTION:

This checklist is a part of the larger Data Quality Objective (DQO) and Data Quality Assessment (DQA) processes. Therefore, additional evaluation may be necessary to fully determine the usability of data. The reviewer will need to consider the process leading up to receipt of data, such as the data objectives, sampling techniques, sampling locations, etc. The full DQO/DQA process should be adhered to in order to make well informed and appropriate decisions on data usability and accuracy required for a project. Additionally, the procedures described in this document do not provide a full data validation. This procedure provides a verification that basic quality control procedures were followed by a laboratory. It is an initial evaluation, that suggests whether or not the reviewer should conduct further evaluation to ensure that data is acceptable for its intended use.

This guidance has been developed so that each item on the laboratory checklist has a corresponding guidance action section using the same numbering system. It is meant to guide the reviewer through the checklist and note the type of action expected if items are missing or a potential concern exists. However, a review of the checklist and data should be fully completed prior to implementing any action set forth in this guidance to ensure the entire situation is addressed by the actions taken. For field measurements (*e.g.*, pH, turbidity), only portions of the checklist may be needed. If a data package is submitted by Ohio EPA's contract laboratory, the district laboratory coordinator should be informed and participate in any contact with the laboratory since this person is, by contract, the primary contact with the laboratory. The district laboratory coordinator will coordinate problem resolution with DERR's contract laboratory manager.

Section II.A. Laboratory Data Review will be used for data received from a fixed laboratory. For reviewing laboratory data packages that include quality control parameters and are used for a more detailed review of site conditions (*e.g.* risk assessment, confirmation samples, or site investigation and scoring). Section II.B. Field Screening Data Review will be used to review data packages when the objective of sampling was field screening and

the data is from a field and/or mobile laboratory.

You are encouraged to consult with your supervisor, district laboratory or QA coordinator(s), or the DERR contract laboratory coordinator for assistance contacting a laboratory or if issues are not satisfactorily addressed by the PRP/consultant or laboratory or the appropriate follow up action is unclear. Because you are determining whether a data package will be acceptable for its intended use, it is important that all anomalies be evaluated collectively.

The attached checklists and guidance should apply to most data packages received by DERR's programs. However, these procedures were developed to cover a variety of analytical procedures and some procedures may be applied differently in some programs. Therefore, not all checklist items may apply to a particular data package or application without using it in conjunction with other program guidance. Reviewers should consider the data quality objectives for the data received to appropriately assess when and how the attached procedures should be used. Specifically, VAP reviewers should use this procedure as directed in the VAP audit guidance. Due to the privatized nature of the VAP, all issues should be addressed to the Certified Professional and resolved through communications between the lead reviewer and CP. If it is clear that issues exist with data from a certified laboratory, the reviewer should contact the VAP certified laboratory coordinator for her to address in future certification processes.

Key Terms:

field screening  
laboratory data review  
data review  
quality assurance  
data validation  
analytical data  
analytical results  
data checklist

### III) CHECKLIST(S)/RECOMMENDED ACTION(S):

#### A. Laboratory Data Review

*DIRECTIONS: The reviewer must use this checklist in conjunction with the attached guidance. Circle the appropriate response and include clarifying comments.*

Item	Please circle	Comments
<b>1.0</b> Did the Laboratory use the approved SOPs from the OEPA approved QAPP for project or other appropriate protocols?	<b>Y N</b>	
<b>2.0</b> Is there a case narrative included? 2.1 Were any issues noted in the case narrative? 2.2 Did the case narrative note any corrective action that was implemented?	<b>Y N</b> <b>Y N</b> <b>Y N</b>	
<b>3.0</b> Was the Chain of Custody included?	<b>Y N</b>	
<b>4.0</b> Does the laboratory report indicate: 4.1 Sample reference numbers 4.2 Date sampled 4.3 Date extracted, if needed 4.4 Date analyzed 4.5 Matrix 4.6 Method used 4.7 Units 4.8 Reporting limit 4.9 Qualifiers, if needed	<b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b>	
<b>5.0</b> Were results from all samples sent to the laboratory provided?	<b>Y N</b>	
<b>6.0</b> Were sample results denoted as either dry or wet weight?	<b>Y N</b>	
<b>7.0</b> Was a sample receipt form included and did it indicate: 7.1 Custody seals, if applicable 7.2 Temperature at receipt 7.3 Bottle condition 7.4 Preservative 7.5 Problems	<b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b>	
<b>8.0</b> Were holding times for each of the samples met?	<b>Y N</b>	

<b>9.0</b> Was blank data from the following included: 9.1 Trip blanks (VOC only) 9.2 Equipment blanks 9.3 Method blanks 9.4 Was contamination noted in any blank?	<b>Y N</b> <b>Y N</b> <b>Y N</b> <b>Y N</b>	
<b>10.0</b> Were the appropriate number of laboratory duplicates completed pursuant to the laboratory's approved QAPP or SOPs?	<b>Y N</b>	
<b>11.0</b> Were the appropriate number of laboratory matrix spikes completed pursuant to the laboratory's approved QAPP or SOPs?	<b>Y N</b>	
<b>12.0</b> Were all of the laboratory duplicates or matrix spikes within QA limits?	<b>Y N</b>	
<b>13.0</b> Were method surrogates (organic analysis only) within QA limits?	<b>Y N</b>	
<b>14.0</b> Were laboratory control samples included: 14.1 Was the data within QA limits?	<b>Y N</b> <b>Y N</b>	

**NOTES:**

Data Quality Assessment (DQA) Issues:

- \*\* Reviewer should evaluate whether data appears similar to past data if continuing project or monitoring to ensure data is consistent in nature. If no, additional review may be necessary.
- \*\* DQA is an iterative process, data should be evaluated also on merits of sampling technique and matrix involved. If inconsistencies are noted, additional review may be necessary.

**1.0:** Use the approved Laboratory QAPP or appropriate SOPs to determine if the laboratory used the correct analytical methods, and reporting limits. Determine if results from all appropriate chemicals of concern were included. Please note that the laboratory sets the reporting limits and analytes analyzed for in a specific method. SW-846 is a guidance to follow in defining these parameters.

If no, contact the PRP/consultant or laboratory to ensure that the data is not missing or misreported.

**2.0** If no, the reviewer should contact the laboratory or PRP/consultant to obtain a copy of the case narrative for the data report.

**2.1** If yes, the reviewer should note if corrective action was performed or if the laboratory qualified the data as “J” (estimated). This may influence the use of the data for your project.

**2.2** If no, the reviewer should note if the laboratory or consultant/PRP has qualified the data as “J” (see above). If the laboratory has already determined the data should be “rejected”, the reviewer will need to determine if a data gap now exists. If no corrective action was completed and the data is not qualified, the reviewer should contact the laboratory for an explanation.

**3.0** If no, the reviewer will need to contact the laboratory to obtain the missing chain of custody. The chain of custody is what the reviewer will use to confirm the samples were analyzed within the appropriate holding times (Attachment A - Holding Times Reference).

**4.0** This section deals with the laboratory report sheets for each sample. All items should be on the data sheet or in some location within the report. A full review of the laboratory report may be necessary to locate some items since different laboratories report data differently.

**4.1** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. Sample reference numbers are the cross reference from the sample identification number to the laboratory’s reference number. These should be included as either a separate sheet at the front of the report or on the analysis data sheet itself. This becomes more complicated if the main laboratory has subcontracted. Be sure that the analyses can be cross referenced back to the original sample numbers.

**4.2** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. This information may be either on the analytical sheet or in the chain of custody.

**4.3** If no and required, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. The extraction date only refers to sample analyses which requires an extraction (e.g., SVOCs, PCBs, and pesticides/herbicides) and the extract can be held for a specific period (Attachment A - Holding Times Reference).

- 4.4** If no, the reviewer should contact the laboratory to request the data that is missing or misreported.
- 4.5** If no, the reviewer should contact the laboratory to request the data that is missing or misreported. This should be included to determine what type of analysis was conducted (*i.e.*, water, extract, or solid).
- 4.6** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. Evaluate this in conjunction with item 1.0.
- 4.7** If no or inconsistent, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. The reviewer should include comments if the data does not appear consistent with the laboratory method, (*e.g.*, solid data reported in mg/L).
- 4.8** If no or inconsistent, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. The reviewer should also review data to ensure it meets the requirements for the specific project (*e.g.*, is low enough to determine if it meets remediation goals). If the reporting limits do not meet the needs of the data objectives, the reviewer will need to further evaluate this issue.
- 4.9** If the laboratory case narrative identifies issues with the data and uses qualifiers notations, a definition of the qualifiers used should be included and the appropriate data qualified. If the data is not qualified or the definition sheet is missing, the reviewer will need to contact the laboratory or PRP/consultant to request the missing information or clarification on the misreported data. (See Attachment B - General Qualifier Definitions).
- 5.0** If no, the reviewer should contact the laboratory or PRP/consultant to request the missing information.
- 6.0** If no or inconsistent with project DQOs, the reviewer should contact the laboratory or PRP/consultant to request the missing information or clarify the analysis procedure. Screening standards and published values are reported in a specific weight and must be comparable for further evaluations.
- 7.0** The laboratory should include a sample receipt form. However, it may be included as part of the case narrative only. If the information provided in the case narrative is acceptable for your project, a separate form is not necessary. If not, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported.
- 7.1** Custody seals may not be used for certain projects. However, if samples were sealed during the project, this should be noted on the sample receipt form. If no, the reviewer will need to contact the laboratory or PRP/consultant to request the data that is missing or misreported.
- 7.2** If no, the reviewer should contact the laboratory or PRP/consultant to request

the data that is missing or misreported. Temperature at receipt for organics, per SW-846, should be 4C +/- 2C. If you are using a different methodology, please ensure the temperature is appropriate for that method. If the temperature is above appropriate levels, additional review is necessary and may require additional technical assistance to determine the usability of the data.

- 7.3** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. If bottle condition is found to be a concern by the laboratory, the reviewer may need to decide if a data gap exists without the specific sample in question.
  - 7.4** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported. If the preservation is incorrect or omitted, the sample may be rejected or the holding time may be reduced (e.g., VOCs (1:1 HCL @ 4C, 14 days vs. without preservative @ 4C, 7 days).
  - 7.5** If yes, the reviewer should contact the laboratory or PRP/consultant to request additional information on the problem and its effect on the analysis. Further evaluation may be necessary with additional technical assistance.
- 8.0** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported.

For evaluation of holding times, see Attachment A. If the samples were not analyzed within the appropriate holding times, the data may not be usable for your project. Also, if the data is from Ohio EPA's contract laboratory, the reviewer must notify the laboratory coordinator since this may be a payment issue.

- 9.0** Any blank contamination will need further evaluation if identified in the laboratory report. See Attachment C - Analytical Blank Flow Chart.
- 9.1** Trip blanks are only included for VOC sampling events. However, if the trip blank was included in the sample set but there is no data in the report, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported.
  - 9.2** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported.
  - 9.3** If no, the reviewer should contact the laboratory or PRP/consultant to request the data that is missing or misreported.
  - 9.4** If yes, the reviewer should follow the guidance on determining the usability of data pursuant to their project. The reviewer may wish to ask for additional technical assistance. (See Attachment C on blank contamination and actions).

For the following sections, see Attachment D for definitions of QA samples used by the laboratory and their purpose in determining usability.

**10.0** Identify at what percentage the laboratory has committed to run laboratory matrix duplicates within a sample batch in their approved Laboratory QAPP or the SOP for the analytical method (*e.g.*, if special service or CLP) normal frequency is 10 to 30 percent of the batch amount or a minimum of one if it is a small batch. If no, the reviewer should contact the laboratory or PRP/consultant to request an explanation for the inconsistency.

**11.0** Identify at what percentage the laboratory has committed to run laboratory matrix spikes within a sample batch in their approved Laboratory QAPP or the SOP for the analytical method (*i.e.*, if special service or CLP) Normal frequency is 10 to 30 % of the batch amount or a minimum of one if it is a small batch.

If no, the reviewer should contact the laboratory or PRP/consultant to request an explanation for the inconsistency.

**12.0** If no, the reviewer should review the case narrative to determine if corrective action for any QA issues were resolved satisfactorily. If no corrective action or inappropriate corrective action was conducted, the reviewer should contact the laboratory or PRP/consultant to request an explanation for the inconsistency. Based on collected information, the reviewer may need to further evaluate the usability of the data provided.

**13.0** If no, the reviewer should review the case narrative to determine if corrective action for any QA issues were resolved satisfactorily. If no corrective action or inappropriate corrective action was conducted, contact the laboratory or PRP/consultant to request an explanation for the inconsistency. Based on collected information, the reviewer may need further evaluate the usability of the data provided.

**14.0** If no, the reviewer should contact the laboratory or PRP/consultant to request the missing data, if necessary.

**14.1** If no, the reviewer should review the case narrative to determine if corrective action for any QA issues were resolved satisfactorily. If no corrective action or inappropriate corrective action was conducted, the reviewer should contact the laboratory or PRP/consultant to request an explanation for the inconsistency. If issues are not satisfactorily resolved, the reviewer may need to further evaluate the usability of the data provided.

B. Field Screening Data Review

*DIRECTIONS: The reviewer must use this checklist in conjunction with the attached guidance. Circle the appropriate response and include clarifying comments.*

Item	Please circle	Comments
<b>1.0</b> Did the field analysis provider use the approved SOPs from the OEPA approved QAPP for project?	<b>Y</b> <b>N</b>	
<b>2.0</b> Is there a case narrative included? 2.1 Were any issues noted in the case narrative? 2.2 Did the case narrative note any corrective action that was implemented?	<b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b>	
<b>3.0</b> Does the laboratory report indicate: 3.1 Sample ID 3.2 Date sampled 3.3 Date analyzed 3.4 Matrix 3.5 Method analysis type 3.6 Units 3.7 Reporting limit 3.8 Summary of sample preparation, if any 3.9 Notation of wet or dry weight	<b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b>	
<b>4.0</b> Were field/analysis login sheets included? 4.1 Do the sheets correspond to field sample names?	<b>Y</b> <b>N</b> <b>Y</b> <b>N</b>	
<b>5.0</b> Does the laboratory analysis report indicate: 5.1 Initial calibration standard 5.2 Blanks 5.3 Laboratory/field duplicates 5.4 Laboratory control sample, if used 5.5 Laboratory/field continuing calibration check(s)	<b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b> <b>Y</b> <b>N</b>	

NOTES:

Data Quality Assessment (DQA) Issues:

\*\* Reviewer should evaluate whether data appears similar to past data if continuing project or monitoring to ensure data is consistent in nature. If no, additional review may be necessary.

\*\* DQA is an iterative process, data should be evaluated also on merits of sampling technique and matrix involved. If inconsistencies are noted, additional review may be necessary.

- 1.0:** Use the approved Laboratory QAPP or SOPs, to determine if the laboratory used the correct analytical methods, and reporting limits. Determine if results from all appropriate chemicals of concern were included. Please note that the laboratory sets the reporting limits and analytes analyzed for in a specific method. SW-846 is a guidance to follow in defining these parameters.

If no, contact the PRP/consultant or laboratory to ensure that the data is not missing or misreported

- 2.0** If no, the reviewer should contact the laboratory or PRP/consultant to obtain a copy of the case narrative for the data report.

**2.1** If yes, the reviewer should note if corrective action was performed or if the laboratory qualified the data such as “J” (estimated). This may influence the use of the data for your project.

**2.2** If no, the reviewer should note if the laboratory has qualified the data as “J” (see above). If the laboratory has already determined the data should be “rejected”, the reviewer will need to determine if a data gap now exists. If no corrective action was completed and the data is not qualified, the reviewer should contact the laboratory for an explanation.

- 3.0** This section deals with the field laboratory report sheets for each sample. Please note that all items should be on the data sheet or in some location within the report. A full review of the field laboratory report may be necessary to locate some items. Different laboratories report data differently; therefore, there could be various locations where the items may be located. (e.g., cross references to sample identification numbers may be at the front of a report while some laboratories will report on the data sheet)

**3.1** If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request the data that is missing or misreported. Sample reference numbers are the cross reference from the sample identification number to the field laboratory service’s reference number. These should either be included as a separate sheet at the front of the report or on the analysis data sheet itself.

**3.2** If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request the data that is missing or misreported.

**3.3** If no, the reviewer will need to contact the field laboratory service to request the data that is missing or misreported.

**3.5** If no, the reviewer will need to contact the field laboratory service to request the data that is missing or misreported. This should be included to determine what type of analysis was conducted (*i.e.*, if on a water, extract, or solid).

**3.6** If no or inconsistent, the reviewer will need to contact the field laboratory service or PRP/consultant to request the data that is missing or misreported. The field laboratory service may report data incorrectly. The reviewer should note if the data does not appear consistent with the field laboratory service method, such as

solid data reported in mg/L.

- 3.7** If no or inconsistent, the reviewer will need to contact the field laboratory service or PRP/consultant to request the data that is missing or misreported. The reviewer should also review data to ensure it meets the requirements for use under the project (*i.e.*, is low enough to determine if meets remediation goals). If the reporting limits does not meet the needs of the data objectives, the reviewer will need to further evaluate this issue.
- 3.8** If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request the missing information or clarification on the misreported data. Please note that specific clean up techniques may be required for samples or preparation kits for immunoassay kits.
- 3.9** If no or inconsistent with needs, the reviewer will need to contact the field laboratory service or PRP/consultant to request the missing information or clarify the analysis procedure. Please be advised that screening standards and published values are reported in a specific weight and need to be comparable for further evaluations.
- 4.0** If no, the reviewer should review the package to ensure that the samples can be cross referenced to a field locations and sampling points. If they can not, the reviewer will need to contact the field laboratory service or PRP/consultant to request the missing information.
- 4.1** If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request information to determine which sample corresponds to the appropriate analysis results.
- 5.0** All laboratory work, even from mobile laboratories and field analysis (*e.g.*, immunoassay work), should include a field analysis report. For field measurements, portions of the checklist may be appropriate for data evaluation, but a formal report, in most cases, will not be generated.
- 5.1** If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request the missing information or clarify the analysis procedure. If there is no calibration standards or standards appear inappropriate, reviewer will need to request further review to determine usability of the data.
- 5.2** If no, please note the type of blank missing (*e.g.*, trip, equipment, method). The reviewer should note that trip blanks are only included for VOC sampling events. However, if the trip blank was included in the sample set but there is no data in the report, the reviewer will need to contact the field laboratory service or PRP/consultant to request the data that is missing or misreported.

If contamination is noted, the reviewer should follow the flow chart (Attachment A) on qualifying the data and determining the usability of data pursuant to their project.

**5.3** The reviewer must identify, from the approved Field laboratory service QAPP or SOP for the analytical method (e.g., special service or CLP), at what percentage the field laboratory service will run matrix duplicates and matrix spikes within a sample batch. Normal quantities are 10 to 30 % of the batch amount, or a minimum of one if a small batch.

If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request an explanation for the inconsistency.

**5.4** If no, the reviewer must review the case narrative to determine if Laboratory Control Samples were run and the reason for running the controls. If the control samples were run as a corrective action, the report should state if the QA issues was resolved satisfactorily . Please note the LCS is not a required sample and may not have been run.

If the LCS was not within QA limits, the reviewer will need to contact the field laboratory service or PRP/consultant to request an explanation for the inconsistency. Based on collected information, the reviewer may need to qualify all data for use or reject data.

**5.5** The reviewer must identify, from the approved field laboratory service QAPP or SOP for the analytical method (e.g., special service or CLP), at what percentage the field laboratory service will run continuing calibration checks and determine if the checks were completed correctly.

If no, the reviewer will need to contact the field laboratory service or PRP/consultant to request an explanation for the inconsistency.

**Attachments:**

- A** Holding Times Reference
- B** Qualifer Definitions (non-standardized)
- C** Analytical Blanks Flow Chart
- D** Definitions and Information on Calibration Samples
- E** District Laboratory and Quality Assurance Coordinators

## Attachment A:

### Sample Holding Times, Recommended Digestion Volumes and Recommended Collection Volumes for Inorganic Determinations in Aqueous and Solid Samples

Measurement	Digestion Volume (mL) <sup>a, c</sup>	Collection Volume (mL) <sup>a, c</sup>	Treatment/Preservative Holding Time <sup>b</sup>
<u>Inorganic Analytes</u> (except hexavalent chromium and mercury):			
Aqueous			
Total	100	600	HNO <sub>3</sub> to pH <2; 6 months
Dissolved	100	600	Filter on site; HNO <sub>3</sub> to pH <2; 6 months
Suspended	100	600	Filter on site; 6 months
Solid			
Total	2g	200g	6 months
<u>Hexavalent Chromium</u>			
Aqueous	100	400	24 hours; store at 4° ± 2°C until analyzed
Solid	2.5g	100g	One month to extraction; 4 days after extraction; store at 4° ± 2°C until analyzed
<u>Mercury</u>			
Aqueous			
Total	100	400	HNO <sub>3</sub> to pH <2; 28 days
Dissolved	100	400	Filter; HNO <sub>3</sub> to pH <2; 28 days
Solid			
Total	0.2g	200g	28 days; store at 4° ± 2°C until analyzed

a Unless otherwise stated.

b Either glass or plastic containers may be used.

c Any sample volume reduction from the reference method's instructions must be made in the exact proportion as described in the method and representative sampling must be maintained.

Reference - SW-846 guidance document (<http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>)

## Sample Containers, Preservation, Techniques, and Holding Times

VOLATILE ORGANICS			
Sample Matrix	Container	Preservative	Holding Time
Concentrated Waste Samples	Method 5035: 40-mL vials with stirring bar. Method 5021: See method. Methods 5031 & 5032: 125-mL widemouth glass container. Use Teflon-lined lids for all procedures.	Cool to 4°C	14 days
Aqueous Samples With <b>No</b> Residual Chlorine Present	Methods 5030, 5031, & 5032: 2x40 mL vials with Teflon-lined septum caps	Cool to 4°C and adjust pH to <2 with H <sub>2</sub> SO <sub>4</sub> , HCl, or solid NaHSO <sub>4</sub> .	14 days
Aqueous Samples <b>WITH</b> Residual Chlorine Present	Methods 5030, 5031, & 5032: 2x40 mL vials with Teflon-lined septum caps	Collect sample in a 125-mL container that has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial. Cool to 4°C and adjust pH to <2 with H <sub>2</sub> SO <sub>4</sub> , HCl, or solid NaHSO <sub>4</sub> .	14 days
Acrolein and Acrylonitrile in aqueous Sample	Methods 5030, 5031, & 5032: 2x40 mL vials with Teflon-lined septum caps.	Adjust pH to 4-5. Cool to 4°C.	14 days
Solid Samples (e.g., soil, sediments, sludges, ash)	Method 5035: 40-mL vials with septum and stirring bar. Method 5021: See method. Methods 5031 & 5032: 125-mL widemouth glass container. Use Teflon-lined lids.	See the individual methods.	14 days
SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES/PCBs AND HERBICIDES			
Sample Matrix	Container	Preservative	Holding Time
Concentrated waste sample	125-mL widemouth glass with Teflon-lined lid	None	Samples extracted within 14 days and extracts analyzed within 40 days after extraction
Aqueous Samples With <b>No</b> Residual Chlorine Present	1-gal., 2x0.5-gal., or 4x1.0L amber glass container with Teflon-lined lid	Cool to 4°C.	Samples extracted within 7 days and extracts analyzed within 40 days after extraction
Aqueous Samples <b>WITH</b> Residual Chlorine Present	1-gal., 2x0.5-gal., or 4x1.0L, amber glass container with Teflon-lined lid	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4°C.	Samples extracted within 7 days and extracts analyzed within 40 days after extraction
Solid Samples (e.g., soils, sediments, sludges, ash)	250-mL widemouth glass container with Teflon-lined lid	Cool to 4°C.	Samples extracted within 14 days and extracts analyzed within 40 days after extraction

## Attachment B: Qualifier Definitions and Report Limits

### Basic Laboratory Qualifiers:

*\* Qualifier definitions vary by laboratory. The reviewer should ensure a definition sheet for laboratory qualifiers is included in the Laboratory Analytical Report.*

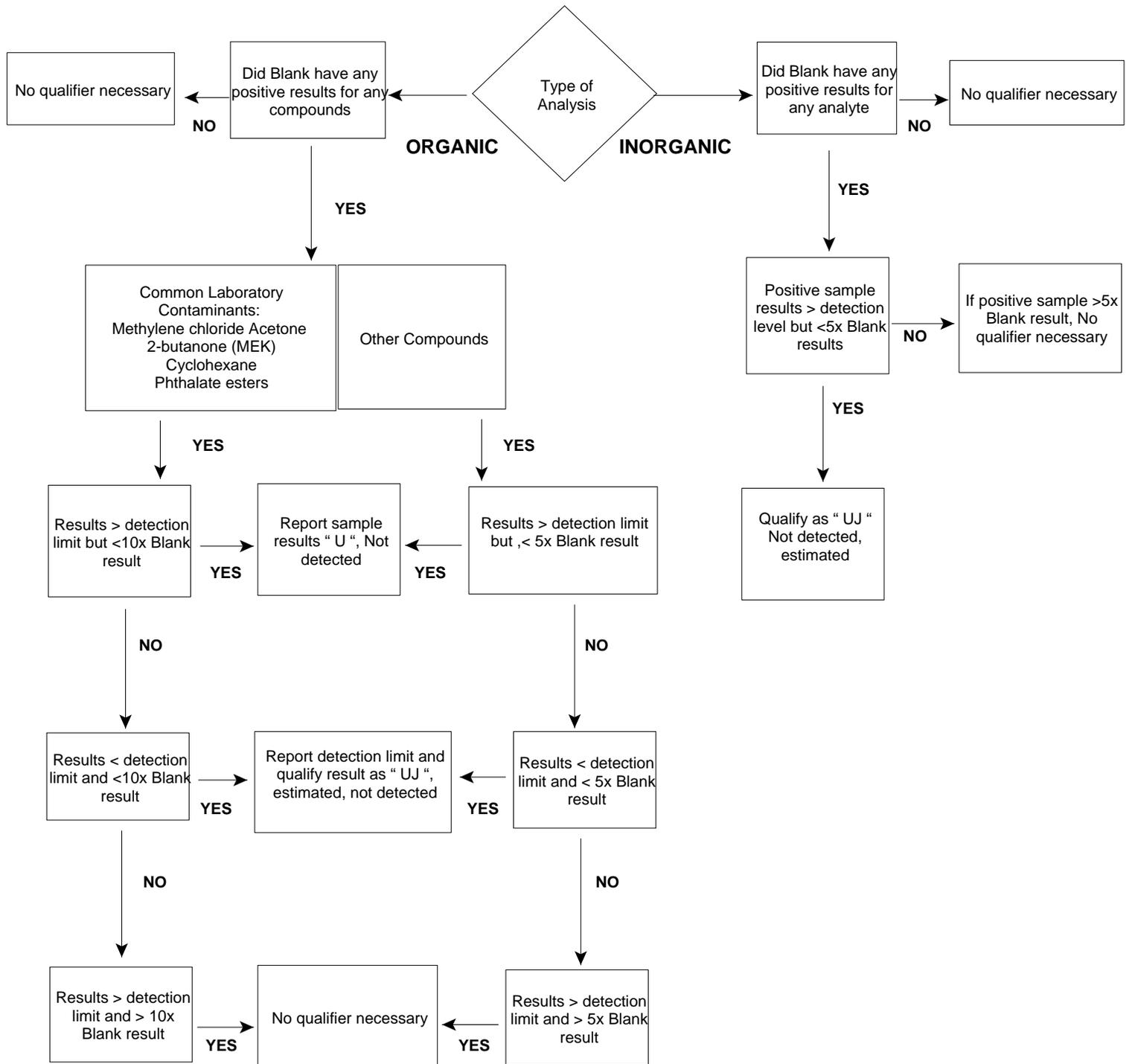
Qualifier	Meaning
B	Compound was detected in the associated blank
D	Result was obtained from a different dilution than other analytes.
E	Result is estimated. Usually, this qualifier indicates that the result is above the calibrated range of the instrument.
J	Result is estimated. Usually, this qualifier indicates the reported concentration is below the laboratory's reporting limit
N	Indicates a Tentatively Identified Compound
ND	Analyte was not detected
U	Analyte was not detected. (U and ND qualifiers are interchangeable.)

### Reporting Limits v.s. Method Detection Limits:

Laboratories will develop their own Reporting Limits (RL) for laboratory data. It is based upon the Method Detection Limits (MDLs) which are determined using detection limit studies for the instrumentation used; usually seven runs of spiked samples; plus a safety factor.

The MDLs are below the method calibration range (below the lowest calibration standard); consequently, results that fall between the low standard or Reporting Limits and the MDL are flagged "J" (estimated) to indicate that they have more uncertainty than the results within the calibration range of the method.

## Attachment C: Analytical Blanks Flow Chart



## Attachment D: Definitions and Information on Calibration Samples

**Analytical Batch.** Samples which are analyzed together with the same method sequence, the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition.

**Analytical Spike.** ( Inorganic Analysis) The post-digestion spike. The addition of a known amount of standard after digestion.

**4-bromofluorobenzene (BFB).** (Organic Analysis) Compound used to establish mass spectral instrument performance for volatile analyses. Also used as a surrogate for volatile organic analyses.

**Calibration Blank.** ( Inorganic Analysis) Usually an organic or aqueous solution that is free of analyte as possible and prepared with the same volume of chemical reagents used in the preparation of the calibration standards and diluted to the appropriate volume with the same solvent used in the preparation of the calibration standard. The calibration blank is used to give the null reading for the instrument response in generating calibration curves.

**Calibration Check.** Verification of the ration of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

**Calibration Check Standard.** Standard used to determine the state of calibration of an instrument between periodic recalibrations.

**Calibration Standards.** A series of known standard solutions used by the analyst for calibration of the instrument (i.e. generation of the analytical curve).

**Calibration Check Compound (CCC).** A set of compounds with known concentrations that are used to monitor the instrument calibration.

**Continuing Calibration.** ( Organic Analysis) Analytical standard run every 12 hours to verify the calibration of the GC/MS system.

**Continuing Calibration.** ( Inorganic Analysis) Analytical standard run every 10 analytical samples or every two hours, whichever is more frequent, to verify the calibration of the analytical system.

**Contract Required Detection Limit (CRDL).** Minimum level of detection acceptable under the contract Statement of Work.

**Control Limits.** A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

**Contract Required Quantitation Limit.** Minimum level of quantitation acceptable under the contract Statement of Work.

**Decafluorotriphenylphosphine (DFTPP).** ( Organic Analysis) Compound used to establish mass spectral instrument performance for semivolatile analysis.

**Duplicate.** A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

**Equipment Blank.** Usually an organic or aqueous solution that is as free of analyte as possible and is transported to the site, opened in the field, and poured over or through the sample collection device, collected in sample container, and returned to the laboratory. This serves as a check on the cleanliness of the sampling device.

**Field Blank.** Usually an organic or aqueous solution that is as free of analyte as possible and transferred from one vessel to another at the sampling site and preserved with the appropriate reagents. This serves as a check on reagent and environmental contamination.

**Initial Calibration.** Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

**Instrument Check Standard.** A multi-element standard of known concentration prepared by the analyst to monitor and verify instrument performance on a daily basis.

**Instrument Detection Limit (IDL).** Determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3 to 5 times IDL on three nonconsecutive days with seven consecutive measurements per day.

**Internal Standard(s).** Compound(s) added to every standard blank, matrix spike, matrix spike duplicate, sample ( for VOAs), sample digestates (for ICP-MS), and sample extracts (for semivolatiles) at a known concentration, prior to analysis. Internal standard(s) are used as the basis for quantitation of the target compounds.

**Laboratory Control Sample (LCS).** A control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same preparation, reagents, and analytical methods employed for the samples received.

**Matrix Spike (MS).** Aliquot of a sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

**Matrix Spike Duplicate (MSD).** A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method.

**Method Blank.** An analytical control consisting of all reagents, internal standards, and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

**Method Detection Limit (MDL).** The constituent concentration that, when processed through the complete method, produces a signal with 99% probability that it is different from the blank. For seven replicates of the sample, the mean must be 3.14 above the blank where  $\sigma$  is the standard deviation of the seven replicates.

**Practical Quantitation Limit (PQL).** The lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory conditions.

**Relative Percent Difference (RPD).** To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value.

**Relative Response Factor (RRF).** A measure of the relative mass spectral response

of an analyte compared to its internal standard. Relative response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

**Reporting Limit (RL).** The minimum concentration that can be readily achieved on a daily basis during routine laboratory conditions.

**System Performance Check Compound (SPCC).** A specified list of compounds at known concentrations that are used to monitor the performance of the analytical system.

**Spectral Interference Check Solution.** (Inorganic Analysis) A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

**Surrogate.** Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in the environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference samples) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate so that the overall efficiency of the method can be determined.

**Trip Blank.** Usually an organic or aqueous solution that is free of analyte as possible and is transported to the sampling site and returned to the laboratory without being opened. This serves as a check on sample contamination originating from sample transport, shipping, and from site conditions.

## **Attachment E: District Laboratory and Quality Assurance Coordinators**

SEDO - Chris Osborne, QA; Mark Stello, Laboratory

SWDO - Randy Watterworth, QA and Laboratory

NEDO - Nancy Zikmanis, QA and Laboratory

NWDO - Archie Lunsey, QA and Laboratory

CDO - Diana Bynum, QA; Doug Crandall, Laboratory

SIFU - Gavin Armstrong, QA; Victoria Sigler, Laboratory

CO - Tim Christman