

ATTACHMENT
TIER I CHECKLIST

Feb 19, 2009 Version

Data Validation Plan Review Form Tier I

This Data Validation Form is #	one	of	one	Forms completed in the review of this semiannual data submittal
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Facility Name		Validator/DO	
ID Number		Date of Plan	
Date Review of Plan Completed		Plan is: New	
Document Title:			
Lab Name:	Media Type(s):	Analyses Requested (Method Number) :	Notes:

Note: The criteria used in the Tier I Data Validation checklist are derived primarily from SW-846 methods and U.S. EPA's National Functional Guidelines (NFGs). Criteria from methods are considered preferable as they are specific to that procedure. Where the method is silent, criteria from the NFGs, or other sources when necessary, are adopted. For flashpoint (which uses ASTM methods dictated by the OAC rules), ASTM method criteria are used.

The Tier I data validation manual is the primary reference for this checklist. It explains and gives examples for the questions in this checklist. The Tier II methodology and terminology builds on that established in the Tier I checklist and its associated data validation manual. There is no Tier II manual, only the checklist and completed example checklists. Additional information is also available by referring to the specific methods.

Data Qualifiers and their meanings used throughout the Tier I Checklist	
J	Estimated
J+	Estimated High (results are likely reported higher than the true value)
J-	Estimated Low (results are likely reported lower than the true value)
R	Rejected
UJ	Undetected Estimated
NJ	Tentatively Identified, Quantitation Estimated

Section 1.0
Report Completeness and Technical Holding Times

1.1 Sample Package Completeness and Deliverables	
Completeness	
This section provides a checklist of important components of data reports. If the report is incomplete, it may be necessary to halt data validation procedures until all the missing information is provided. Please, refer to the Tier I data validation manual for additional assistance in completing the checklist.	
1.1.1	<p>Are COC forms present for all samples?</p> <p><i>Action: If not contact the facility for replacement of missing or illegible copies</i></p>
1.1.2	<p>Is a signed statement from the laboratory present that attests to the validity of the data?</p> <p><i>Action: Take no further action and contact the facility and have the lab submit a valid data report. If no response, qualify all data as unusable.</i></p>
1.1.3	<p>Is a case narrative present that summarizes QA/QC discrepancies and/or other problems?</p> <p><i>Action: No action is necessary, but this information is useful to focus data validation efforts.</i></p>
1.1.4	<p>Are all the requested analyses accounted for in the data report? Describe any omissions between the Chain Of Custody (COC) record and submitted sampling data.</p> <p><i>Action: If there are discrepancies, contact the laboratory for any missing deliverables and/or an explanation.</i></p>

1.1 Sample Package Completeness and Deliverables	
<p>1.1.5 Is a sample receipt form present? If so, does it contain information on the condition of sample containers, proper preservatives used (cross-check with COC) and temperature of the cooler? Note any comments or abnormal conditions: Action may be taken for the following special conditions:</p> <p>A. For samples analyzed for volatiles that were not properly cooled (temperature more than 6 - 10°C), all positive results should be qualified as "J-" and all non-detects qualified as "UJ."</p> <p>B. For all liquid Volatile Organic Compound (VOC) samples or vials with air bubbles (>2 mm), positive results should be qualified as "J-" and non-detects as "UJ" or "R" depending on professional judgment (taking into account other quality control information such as sample cooler temperature and other site specific data quality objectives).</p> <p>C. If aqueous samples for VOCs were not preserved, check that technical holding times were met (see Technical Holding Times, Table 1). If not, qualify all associated sample results.</p> <p>D. If liquid TCLP samples were preserved, qualify all associated results as rejected and flag the data with an "R."</p>	
<p>1.1.6 Do the COC forms, sample receipt form, or the case narrative indicate any problems with the sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? List any problems that were found.</p> <p><i>Action: Use the information to focus data validation efforts.</i></p>	
<p>1.1.7 Optional: Are custody seals present and intact?</p>	

1.2 Technical Holding Times

Table 1

Technical Holding Times for Volatile, Semi-Volatile, Metals and pH Samples

Technical holding time is the time, in days, from sample acquisition in the field to either laboratory preparation or analysis. Technical holding times are established from information contained in the laboratory report, chain of custody, and raw analytical bench sheets (if available). Technical holding times also depend upon whether samples were preserved. The recommended technical holding times for volatile compounds, semi-volatile compounds, metals, and TCLP analyses are listed below.

	Preserved	From field collection to extraction	from extraction to preparation	From extraction to analysis	Max Holding Times	Common preservative
VOCs (8260) (aqueous)	Yes	NA	NA	14 days	14 days	Cool to 4° C, HCl
VOCs (8260) (aqueous)	No	NA	NA	7 days	7 days	Cool to 4° C
VOCs (8260) (liquid waste)	No	NA	NA	14 days	14 days	Cool to 4° C
VOCs (8260) (solid/soil/waste)	No	NA	NA	NA	14 days	Cool to 4° C or no preservative
VOCs (EnCore) (5035/8260) (solid/soil/waste)	Yes	2 days	NA	12 days	14 days	Encore Sampler
SVOC(8270)	Yes	7 days	NA	40 days	47 days	Cool to 4° C
Total Metals (6010B/7000)	Yes	NA	NA	180 days	180 days	Nitric Acid (pH<2- aqueous); cool to 4° C - solid samples
Mercury (7470A)	Yes	NA	NA	28 days	28 days	Nitric Acid (pH<2- aqueous); cool to 4° C - solid samples
TCLP VOCs (1311/8260)	No	14 days	NA	14 days	28 days	no preservative
TCLP SVOCs (1311/8270)	No	14 days	7 days	40 days	61 days	no preservative
TCLP Metals (except mercury) (1311/6010B)	No	180 days	NA	180 days	360 days	no preservative

	Preserved	From field collection to extraction	from extraction to preparation	From extraction to analysis	Max Holding Times	Common preservative
TCLP mercury (1311/7470A)	No	28 days	NA	28 days	56 days	no preservative
pH (9040B)	No	24 hours	NA	NA	1 day	no preservative
Ammonia (Liquid, SM 4500-N)	No	NA	NA	7 days	7 days	Cool to 4° C
Ammonia (Liquid, SM 4500-N)	Yes	NA	NA	28 days	28 days	Cool to 4° C; H ₂ SO ₄ to pH <2
Cyanide (Liquid)	Yes	NA	NA	14 days	14 days	Cool to 4° C; NaOH >10

1.2 Technical Holding Times

Technical Holding Times

Technical holding time evaluation is important to assure the data is valid and not biased from inappropriate handling procedures. Technical holding times are judged by assessing the lapsed time from field sampling to extraction and then to analysis. There are specific technical holding time requirements for specific classes of compounds. In addition, holding times may vary due to the presence or absence of preservatives. The validator should refer to specific criteria for holding times listed in Table 1 and in the Tier I Data Validation Manual. Use information on sampling found on the chain-of-custody, and extraction and analysis dates (found in the data report, examined in section 1.0) to determine whether technical holding times are in compliance with criteria listed in Table 1. Complete the following table to determine if any violations of technical holding time exist, and qualify all associated sampling data.

Technical Holding Times - Volatile Organic Compounds

1.2.1	Are samples properly preserved? Check preservation requirements, chain-of-custody, and sample receipt form for discrepancies. <i>Action: Note any problems and use the information to qualify results.</i>	List any problems:
1.2.2	If samples were improperly preserved, or unpreserved, and the technical holding times were exceeded, qualify all positive results for affected samples as "J-" and all non-detected results as "UJ."	List sample ID(s):
1.2.3	If technical holding times are greatly exceeded (> 2x the time requirement) upon analysis or re-analysis then the validator may use professional judgment to qualify all non-detected compounds as "UJ" or "R" based upon professional judgment and on DQOs.	List sample ID(s):

Technical Holding Times - Semi-Volatile Organic Compounds

1.2.4	If technical holding times are exceeded (Table 1), qualify all positive results for affected samples as "J-" and all non-detected results as "UJ."	List sample ID(s):
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<p>1.2.5 If technical holding times are greatly exceeded (> 2x the time requirement), based on the project's DQOs, qualify all positive results as estimated (J-). The validator may use professional judgment to qualify all non-detected compounds as "R" or "UJ".</p>	<p>List sample ID(s):</p>
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Technical Holding Times - Inorganic Compounds

<p>1.2.6 Are samples properly preserved (4°C for solids; acid preservation for aqueous samples or unpreserved)? Check preservation requirements, chain-of-custody, and sample receipt form for discrepancies.</p> <p><i>Action: Note any problem, and use the information to qualify results in the next step.</i></p>	<p>List problems:</p>
<p>1.2.7 If samples were improperly preserved or properly preserved and the technical holding times were exceeded (Table 1), qualify all positive results for affected samples as estimated ("J-") and all non-detected results as "UJ." or rejected ("R") depending on DQOs.</p>	<p>List sample ID(s):</p>
<p>1.2.8 If technical holding times are greatly exceeded (> 2x the time requirement), the validator may use professional judgment and the project's DQOs to qualify all non-detected compounds as "R" and all positive results as "J-" or "R" depending on DQOs.</p>	<p>List sample ID(s):</p>

Technical Holding Times - pH

<p>1.2.9 If technical holding times are exceeded, the data validator may use professional judgment and DQOs to qualify data as "R" or "J-."</p> <p>Note: For ground water samples, pH should be evaluated in the field within 15 minutes of sampling. For waste samples, the technical holding time is more flexible and requires an examination of the type of waste and the project's DQOs. If technical holding times exceed 24 hours, consider qualification. If wastes exhibit the characteristic of corrosivity (i.e., <pH 2 or > pH of 12.5), samples should not be qualified.</p>	<p>List sample ID(s):</p>
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Section 2.0
VOC Data Validation

2.0 VOC Analysis Data Validation	
2.1 Blank Data Summary Review - Volatile Organic Compounds	
Blank Data	
Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.	
<p>2.1.1 Is the method blank data present for each batch (matrix and sample number dependent), including TCLP?</p> <p><i>Action: If not present, request information from the facility. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J," for positive results and "UJ," for non-detected compounds) based upon the validator's judgment. Additional qualification may result based upon other QA/QC information.</i></p>	
<p>2.1.2 Is there an indication that the samples associated with the method blank were diluted?</p> <p>Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).</p>	List the dilution factor(s):
<p>2.1.3 Do any method/field/trip/rinsate blanks have any positive results for any volatile target analytes? Were the same target compounds found in the samples? List those analytes and the results that are both found in the blanks and samples. These analytes are subject to qualification.</p> <p>Note: A list of samples associated with each of the contaminated blanks should be prepared. Trip blanks are used to qualify samples based on potential contamination during shipment, and are not required for non-aqueous matrices.</p> <p><i>Action: Follow the directions in question 2.1.4 using the criteria in the table below to qualify sample results due to blank contamination. Use the largest value from all of the associated blanks. If any blanks are grossly contaminated, all associated data may be qualified as "R", based upon professional and the project's DQOs.</i></p>	
<p>2.1.4 For those analytes identified in question 2.1.3 , follow the directions in the following table.</p> <p>Note: If analytes are detected in a blank but not in the sample of interest, then qualification of those analytes are not necessary. Use the information from 2.1.2 to determine whether a dilution factor should be used to determine qualification. When a dilution is applied to samples, the contaminant concentration in the samples are divided by the dilution factor, then use the criteria listed in the following table to qualify blanks and sample data.</p>	

For Common Volatile Contaminants: methylene chloride, acetone, 2- butanone, cyclohexane	For Other Contaminants:	Action:
Sample Conc. > Detection Limit but < 10x Blank Result	Sample Conc. > Detection Limit but < 5x Blank Result	Qualify result as undetected and flag the result with an "U".
Sample Conc. > Detection Limit & > 10x Blank Result	Sample Conc. > Detection Limit & > 5x Blank Result	No qualification is necessary

2.2 Volatile Organic Data Review - Laboratory Control Sample (LCS)	
Laboratory Control Sample	
<p>An LCS should be included with each batch of samples (approximately 20). The LCS consists of an aliquot of a clean (control) matrix similar to the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix (Method 8260B).</p>	
<p>2.2.1 Was an LCS prepared, extracted, analyzed and reported once per group of 20 samples?</p> <p>Note: This information should be included in the QA package provided by the lab. If not, contact the laboratory and request that the information be submitted to the agency. This information should be found in the injection log.</p> <p><i>Action:</i> If LCS information cannot be found, consult the facility for re-submittal of the data package. If LCS information is not present, qualify all positive results as "J." If warranted, the Data Validator may reject all results as unacceptable.</p>	
<p>2.2.2 Does the LCS contain the following volatile target compounds in addition to the required surrogates?</p> <p>1,1-Dichloroethene Toluene Trichloroethene Benzene Chlorobenzene</p> <p>Note: Method 8260B calls for the LCS to be spiked at the same level as the matrix spike. When the results of the matrix spike indicate a problem due to sample matrix, the LCS should be checked to determine whether the laboratory can perform the analysis on a clean matrix.</p>	

2.2 Volatile Organic Data Review - Laboratory Control Sample (LCS)	
<p>2.2.3 Do the percent recoveries (%R) meet the QC limits provided by the lab?</p> <p><i>Action: If the LCS recovery is greater than the upper acceptance limit, then positive sample results for the affected compound(s) should be qualified as estimated "J+."</i></p> <p><i>If the LCS recovery is less than the lower acceptance limit, then the associated detected target compounds should be qualified as "J-," and the associated non-detected target compounds should be qualified as rejected and data flagged with an "R."</i></p> <p><i>If more than half of the compounds in the LCS are not within the recovery criteria, then all of the associated detected target compounds should be qualified as "J," and all associated non-detected compounds should be qualified as "R."</i></p>	<p>List compounds and sample IDs that do not meet QC limits</p>
<p>2.2.4 Verify the calculations for at least one %R. $\%R = \text{found/true} \times 100$</p> <p><i>Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submittal or if the data should be flagged.</i></p>	

2.3 Quality Assurance Summary Review - Matrix Spike/Matrix Spike Duplicates, VOC	
<p>Matrix Spike/Matrix Spike Duplicates</p> <p>Matrix spike and matrix spike duplicates are performed to assess method precision for VOC and SVOC analyses. Matrix spikes and duplicates are required for every batch of samples (every 20 - 30 samples). The validator should be aware that the MS/MSD are batch specific, not sample specific. For example, the MS/MSD information may be any sample in the batch, but not necessarily a sample being validated. Because of this, matrix spike and matrix spike duplicate data alone usually aren't used to qualify results, but the information is used with other QA/QC data to qualify data.</p>	
<p>2.3.1 Is matrix spike/matrix spike duplicate recovery data present?</p> <p><i>Action: If the matrix spike data is missing, the laboratory should be contacted for a re-submittal.</i></p>	
<p>2.3.2 How many VOC spike recoveries are outside the QC limits?</p>	<p>Record the spike recovery and control limits:</p>
<p>2.3.3 How many RPDs for matrix spike and matrix spike duplicate recoveries are outside the QC limits for VOCs?</p> <p>Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification. Outliers should be identified.</p>	<p>Record the recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualification is necessary:</p>

2.4 VOC Surrogate Recovery

VOC Surrogate Compound Recovery

Surrogate compounds are spiked compounds of known composition that are added to samples and blanks. The recovery of surrogate compounds allows an assessment of matrix interference. VOC surrogate recoveries are used with other QA/QC data to qualify sample results and to justify laboratory re-analysis. Specific examples are listed in the data validation guidance document.

<u>Surrogate Compound</u>	<u>Water</u> ^a	<u>Soil/Sediment</u> ^a
4-Bromofluorobenzene	86-115	74-121
Dibromofluoromethane	86-118	80-120
Toluene-d ₈	88-110	81-117
1,2-Dichloroethane-d ₄	80-120	80-120

Other Common VOC Surrogates

1,2-Dichlorobenzene-d₄
Pentafluorobenzene
Fluorobenzene

^a SW-846 Method 8260B, Table 8. Acceptance criteria is guidance.

2.4.1 Are the surrogate recovery data present for each batch (method and matrix), including TCLP?

Note: Samples may be included in separate sample batches and separate surrogate recoveries should be provided.

Action: If no, contact the laboratory for explanation and re-submittal.

2.4.2 Were any outliers marked correctly (based upon the laboratory's criteria)?

Action: Mark, circle or highlight the suspected outliers.

List the sample ID(s), matrix(-ces) and parameter(s):

2.4.3 If any surrogate compound was out of compliance was re-analysis performed to confirm a matrix interference?

Note: Check the report narrative for an indication of re-analysis. Additionally, qualification may not be appropriate for TCLP data. Best professional judgment may be used to qualify data.

Action: If a surrogate is above the upper control limit, all positive results should be qualified as "J+". Results listed as non-detected should not be qualified.

If any surrogate recovery is less than the lower criteria, but greater than or equal to 10% recovery, all detected compounds should be qualified as "J-" and all non-detected compounds as "UJ."

If any surrogate recovery is less than 10%, all detected compounds should be qualified as "J-" and all non-detected compounds as "R."

List sample ID(s) for surrogate compounds out of compliance and criteria:

Section 3.0 SVOC Data Validation

3.0 SVOC Analysis Data Validation	
3.1 Blank Data Summary Review - Semi-Volatile Compounds	
Blank Data	
Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.	
3.1.1	<p>Is the method blank data present for each batch (matrix and sample number dependent), including TCLP?</p> <p><i>Action: If not present, request information from the facility. If the required method blank(s) was not analyzed, sample results may be qualified as estimated ("J," for positive results and "UJ," for non-detected compounds) based upon the validator's judgment. Additional qualification may result based upon other QA/QC information.</i></p>

3.0 SVOC Analysis Data Validation	
3.1 Blank Data Summary Review - Semi-Volatile Compounds	
<p>3.1.2 Is there an indication that samples, associated with the blank, were diluted?</p> <p>Note: The dilution factor can be found in the data report (a dilution factor of 1 indicates no dilution).</p>	<p>List the dilution factor(s):</p>
<p>3.1.3 Do any method/field/trip/rinsate blanks have any positive results for any semivolatile target analytes? Was the same target compounds found in the samples? List those analytes and the results that are both found in the blanks and samples. These analytes are subject to qualification.</p> <p>Note: A list of samples associated with each of the contaminated blanks should be prepared. Field blank results should be used to qualify data. Trip blanks are used to qualify samples based on potential contamination during shipment, and are not required for non-aqueous matrices.</p> <p><i>Action: Go to question 3.1.4 and follow the directions in the table below to qualify sample results due to blank contamination. Use the largest value from all of the associated blanks. If any blanks are grossly contaminated, all data associated may be qualified as "R", based upon professional and the project's DQOs.</i></p>	
<p>3.1.4 For those analytes identified in question 3.1.3, follow the directions in the table below.</p> <p>Note: If analytes are detected in a blank but not in the sample of interest, then no qualification is necessary. Use the information from 3.1.2 to determine whether a dilution factor should be used to determine qualification. When a dilution is applied to samples, the contaminant concentration in the samples are divided by the dilution factor, then use the criteria listed in the following table to qualify blanks and sample data.</p>	

For Common Semi-Volatile Contaminants: Phthalate esters	For Other Contaminants:	Action:
Sample Conc. > Detection Limit but < 10x Blank Result	Sample Conc. > Detection Limit but < 5x Blank Result	Qualify result as undetected and flag the result with an "U".
Sample Conc. > Detection Limit & > 10x Blank Result	Sample Conc. > Detection Limit & > 5x Blank Result	No qualification is necessary

3.2 Semi-Volatile Data Review - Laboratory Control Sample (LCS)

3.2 Semi-Volatile Data Review - Laboratory Control Sample (LCS)			
Laboratory Control Sample			
<p>An LCS should be included with each batch of samples (approx. 20). The LCS consists of an aliquot of a clean (control) matrix similar to the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix (Method 8270C).</p>			
<p>3.2.1 Was an LCS prepared, extracted, analyzed and reported once per group of 20 samples (per batch)?</p> <p>Note: This information should be included in the QA/QC package provided by the lab. If not, contact the laboratory and request that the information be submitted to the Agency.</p> <p><i>Action: If LCS information is not present, consult the facility for re-submission of the data package. If LCS information is not available, qualify all positive results as "J." If warranted, the validator may reject all results.</i></p>			
<p>3.2.2 Does the LCS contain the following semi-volatile target compounds in addition to the required surrogates?</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%; vertical-align: top;"> <u>Base/Neutrals</u> 1,2,4-Trichlorobenzene Acenaphthene 2,4-Dinitrotoluene Pyrene N-Nitroso-di-n-propylamine 1,4-Dichlorobenzene </td> <td style="width: 50%; vertical-align: top;"> <u>Acids</u> Pentachlorophenol Phenol 2-Chlorophenol 4-Chloro-3-methylphenol 4-Nitrophenol </td> </tr> </table> <p>Note: Method 8270C calls for base/neutral compounds to be spiked at 100 µg/L and acid compounds to be spiked at 200 µg/L. However, for waste samples the concentration should be 5 times higher. Other compounds can be spiked into the LCS; however, these compounds should represent the entire range of target analytes. In addition, the compounds in the LCS should be consistent with the compounds included in the matrix spike/matrix spike duplicate.</p>	<u>Base/Neutrals</u> 1,2,4-Trichlorobenzene Acenaphthene 2,4-Dinitrotoluene Pyrene N-Nitroso-di-n-propylamine 1,4-Dichlorobenzene	<u>Acids</u> Pentachlorophenol Phenol 2-Chlorophenol 4-Chloro-3-methylphenol 4-Nitrophenol	
<u>Base/Neutrals</u> 1,2,4-Trichlorobenzene Acenaphthene 2,4-Dinitrotoluene Pyrene N-Nitroso-di-n-propylamine 1,4-Dichlorobenzene	<u>Acids</u> Pentachlorophenol Phenol 2-Chlorophenol 4-Chloro-3-methylphenol 4-Nitrophenol		

3.2 Semi-Volatile Data Review - Laboratory Control Sample (LCS)	
<p>3.2.3 Do the percent recoveries (%R) meet the QC limits provided by the lab?</p> <p><i>Action: If the LCS recovery is greater than the upper acceptance limit, then positive sample results for the affected compound(s) should be qualified as "J+."</i></p> <p><i>If the mass spectral criteria are met, but the LCS recovery is less than the lower acceptance limit, then the associated detected target compounds should be qualified as "J-," and the associated non-detected target compounds should be qualified as "R."</i></p> <p><i>If more than half of the compounds in the LCS are not within the recovery criteria, then all of the associated detected target compounds should be qualified as "J," and all associated non-detected compounds should be qualified as "R."</i></p>	<p>List compounds and sample IDs that do not meet QC limits:</p>
<p>3.2.4 Verify the calculations for at least one %R.</p> <p>$\%R = \text{found/true} \times 100$</p> <p><i>Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submission. If the recalculated %R values fall within the QC limits, the validator should use professional judgment to determine if the lab should be contacted for re-submission or the data should be flagged.</i></p>	

3.3 Quality Assurance Summary Review - Matrix Spike/Matrix Spike Duplicates, SVOC	
<p>Matrix Spike/Matrix Spike Duplicates</p> <p>Matrix spike and matrix spike duplicates are performed to assess method precision for VOC and SVOC analyses. Matrix spikes and duplicates are required for every batch of samples (every 20 - 30 samples). The validator should be aware that the MS/MSD are batch specific, not sample specific. For example, the MS/MSD information may be any sample in the batch, but not necessarily a sample being validated. Because of this, matrix spike and matrix spike duplicate data alone usually aren't used to qualify results, but the information is used with other QA/QC data to qualify data.</p>	
3.3.1	<p>Is matrix spike/matrix spike duplicate recovery data present?</p> <p><i>Action: If any matrix spike data is missing, the laboratory should be contacted for a re-submittal.</i></p>
3.3.2	<p>How many SVOC spike recoveries are outside the QC limits?</p> <p>Record the compound(s) out of compliance, their spike recovery and the control limits:</p>
3.3.3	<p>How many RPDs for matrix spike and matrix spike duplicate recoveries are outside the QC limits for SVOCs?</p> <p>Note: The MS/MSD results may be used in conjunction with other QC criteria to determine the need for data qualification. Outliers should be identified.</p> <p>Record compound(s) with recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualification is necessary:</p>

3.4 SVOC Surrogate Recovery															
<p>SVOC Surrogate Compound Recovery</p> <p>Surrogate compounds are spiked compounds of known composition that are added to samples and blanks. The recovery of surrogate compounds allows an assessment of matrix interference. SVOC analyses include compounds that can be divided into two classes: acid compounds and base/neutral compounds. Each class has a specific assigned set of surrogate compounds. The list of compounds can be found in the data validation guidance manual or SW-846, Method 8270. Data validation is also based upon the type of compound being analyzed. SVOC surrogate recoveries also are used to justify re-analysis to confirm matrix interference, but the number of surrogate compounds out of compliance will justify qualification. Specific examples are listed in the data validation guidance document.</p> <table border="0" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th style="text-align: center;"><u>Surrogate Compound</u></th> <th style="text-align: center;"><u>Fraction</u></th> </tr> </thead> <tbody> <tr> <td style="text-align: center;">phenol-d₆</td> <td style="text-align: center;">Acid</td> </tr> <tr> <td style="text-align: center;">2-fluorophenol</td> <td style="text-align: center;">Acid</td> </tr> <tr> <td style="text-align: center;">2,4,6-tribromophenol</td> <td style="text-align: center;">Acid</td> </tr> <tr> <td style="text-align: center;">nitrobenzene-d₅</td> <td style="text-align: center;">Base/Neutral</td> </tr> <tr> <td style="text-align: center;">2-fluorobiphenyl</td> <td style="text-align: center;">Base/Neutral</td> </tr> <tr> <td style="text-align: center;">p-terphenyl-d₁₄</td> <td style="text-align: center;">Base/Neutral</td> </tr> </tbody> </table>		<u>Surrogate Compound</u>	<u>Fraction</u>	phenol-d ₆	Acid	2-fluorophenol	Acid	2,4,6-tribromophenol	Acid	nitrobenzene-d ₅	Base/Neutral	2-fluorobiphenyl	Base/Neutral	p-terphenyl-d ₁₄	Base/Neutral
<u>Surrogate Compound</u>	<u>Fraction</u>														
phenol-d ₆	Acid														
2-fluorophenol	Acid														
2,4,6-tribromophenol	Acid														
nitrobenzene-d ₅	Base/Neutral														
2-fluorobiphenyl	Base/Neutral														
p-terphenyl-d ₁₄	Base/Neutral														
3.4.1	<p>Are the surrogate recovery data present for each batch (method and matrix), including TCLP?</p> <p>Note: Samples may be included in separate sample batches and separate surrogate recoveries should be provided.</p> <p><i>Action: If no, contact the laboratory for explanation and re-submittals.</i></p>														
3.4.2	<p>Were any outliers marked correctly?</p> <p><i>Action: Mark, circle or highlight the suspected outliers.</i></p> <p>List the sample ID(s), matrix(-ces) and parameter(s):</p>														

3.4 SVOC Surrogate Recovery	
<p>3.4.3 If any TWO surrogate compounds in either the acid <u>or</u> base/neutral fractions were out of compliance, was re-analysis performed to confirm a matrix interference?</p> <p>Note: Check the report narrative for an indication of re-analysis.</p> <p><i>Action: If no information is present, request information from the facility.</i></p>	<p>List sample ID(s) for surrogate compounds out of compliance and criteria:</p>
<p>3.4.4 If any ONE surrogate compound has a recovery of less than 10% in either the acid <u>or</u> base/neutral fractions, check for indications that re-analysis was performed to confirm a matrix interference?</p> <p>Note: Check the report narrative for an indication of re-analysis.</p>	<p>List sample ID(s) for surrogate compounds out of compliance and criteria:</p>
<p>3.4.5 Based on the findings, qualify data in either the acid or base/neutral fractions with the following criteria:</p> <p>Note: Qualification may not be appropriate for TCLP data. Best professional judgment may be used to qualify data.</p> <p><i>Action: If TWO surrogates in a particular class are above the upper control limit, all positive results, for that fraction, in that fraction should be qualified as "J+" Results listed as non-detected should not be qualified.</i></p> <p><i>If any TWO surrogates in a particular fraction have recoveries less than the lower criteria, but the recovery is greater than or equal to 10%, all detected compounds, for that fraction, should be qualified as "J-" and all non-detected compounds as "UJ."</i></p> <p><i>If any surrogates in a particular fraction have recoveries less than 10%, all detected compounds, for that fraction, should be qualified as "J-" and all non-detected compounds as "R."</i></p>	<p>List the ID(s) of the affected sample(s):</p>

Section 4.0

Metals Data Validation

4.0 Metals Analysis Data Validation	
4.1 Blank Data Summary Review - Metals Data	
Blank Data	
Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.	
<p>4.1.1 Is the method/prep blank summary data present for each batch (generally separated by method and matrix), including TCLP?</p> <p><i>Action: If not present, request information from the facility. If the required method blanks were not analyzed, sample results may be qualified as "J" for positive results and "UJ" for non-detected compounds. Qualification should take into account other QA/QC information and the DQOs.</i></p>	
<p>4.1.2 Were any samples diluted?</p> <p><i>Action: Record the sample ID and dilution factor(s).</i></p>	
<p>4.1.3 If metals are detected in the blank, check the sample results and record all analytes and the results detected in both the blank and sample.</p> <p>Note: Use the information from 4.1.2 to determine whether a dilution factor should be used to determine qualification. When a dilution factor is applied to samples, the contaminant concentration in the samples are divided by the dilution factor, then the criteria discussed below is used to qualify sample results.</p> <p><i>Action: Positive sample results that are greater than the detection limit but less than 5 X the blank results (after dilution is accounted for) should be qualified as estimated and flagged with a "U." Sample results greater than 5X the blank results (after accounting for dilution) should not be qualified.</i></p>	
Blanks- Mercury	
Mercury is analyzed using SW-846 Method 7470A for solid samples and Method 7471A for liquid samples. These methods utilize a manual cold vapor atomic adsorption (AA) technique to quantify mercury. These methods have slightly different acceptance criteria than other AA methods and therefore are separated in the checklist.	
<p>4.1.4 Was a method/preparation blank included with each batch of samples.</p> <p><i>Action: Consult the lab and if possible have the data submitted. If the data is not available, the data validator may apply best professional judgment to qualify the sample results.</i></p>	

Blanks- Mercury

Mercury is analyzed using SW-846 Method 7470A for solid samples and Method 7471A for liquid samples. These methods utilize a manual cold vapor atomic adsorption (AA) technique to quantify mercury. These methods have slightly different acceptance criteria than other AA methods and therefore are separated in the checklist.

<p>4.1.5 Did the method blank contain mercury above detectable levels? Was mercury also detected in the sample results? If so, these results are subject to qualification.</p> <p>Note: If mercury is discovered in the method blank above the detection limit, the lowest concentration of any sample in that batch must be 10 times the method blank concentration (after dilution is accounted for). If this is not the case, all samples in that batch should have been re-digested and re-analyzed.</p> <p><i>Action: Review the blank data. If the sample results are positive but less than 10 times the concentration in the blank, the results should be qualified as "U".</i></p>	
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4.2 Metal Spike Recovery

Metal Spike Recovery

Spikes are elements of known composition that are added to blanks and to samples that measure accuracy and precision of the analyses. At least one spike (termed a matrix spike or prep spike) should be included for each batch of samples. Spike recovery criteria listed in this section are determined from U.S. EPA's National Functional Guidelines for Inorganic Data Review. The criteria applied by an individual laboratory may vary. The laboratory should be consulted and its QA/QC criteria supplied to the validator.

<p>4.2.1 Confirm that at least one pre-digestion spiked (matrix spike) sample was analyzed per batch, matrix type and concentration or sample delivery group?</p> <p><i>Action: If not present, contact the facility for re-submittal.</i></p>	
<p>4.2.2 Are all spike recoveries (except Hg and Ag) within control limits (e.g., 75% to 125%)?</p> <p>Note: When the spike sample result is less than the instrument detection limit, the percent recovery calculation should use a value of zero (not the detection limit) for the sample result.</p> <p><i>Action: Is the sample concentration ≥ 4 times the spiked concentration? If yes, disregard spike recoveries for analytes whose concentrations in samples are > 4 times the spike added. If no, circle those analytes whose concentration is < 4 times the spike added.</i></p>	<p>List those elements out of control:</p>

4.2 Metal Spike Recovery	
<p>4.2.3 Based on the results of 4.2.2, if the sample results were <4x the spike amount and spike recoveries were out of criteria, a post-digestion spike should be analyzed.</p> <p>Note: Post-digestion spikes are not required for Ag or Hg, however, one typically is run if the LCS was out of control. The post digestion spike confirms a matrix interference and should not be used for qualification</p> <p><i>Action: Contact the facility/laboratory for an explanation if a post-digestion spike was not analyzed. If a satisfactory explanation is not available, use professional judgment to qualify sample results.</i></p>	
<p>4.2.4 Are any aqueous spike recoveries (pre and post digestion):</p> <ol style="list-style-type: none"> 1. Less than 30%? 2. Between 30% and 74%? 3. Between 126% and 150%? 4. Greater than 150%? <p>Note: The TCLP extract should be handled as an aqueous sample.</p> <p><i>Action: If < 30%, and the sample results are below the detection limit, all data should be qualified as "R." Detected values may be qualified as "J-" or R depending on professional judgment and the project's DQOs.</i></p> <p><i>If between 30% and 74%, qualify all positive data as "J-" and non-detected data as "UJ."</i></p> <p><i>If between 126% and 150%, qualify positive as "J+." All undetected compounds are acceptable.</i></p> <p><i>If > 150% note for possible positive bias. Evaluator may qualify data "R" based on professional judgment and the eventual end use of the data.</i></p>	
<p>4.2.5 Are any soil/solid/waste spike recoveries (pre and post digestion):</p> <ol style="list-style-type: none"> 1. Less than 10%? 2. Between 10% and 74%? 3. Between 126% and 200%? 4. Greater than 200%? <p><i>Action: If < 10%, those elements out of control limits should be qualified as "R."</i></p> <p><i>If between 10% and 74%, qualify those detected elements in the samples out of control limits as "J-".</i></p> <p><i>If between 126% and 200%, qualify positive data, for those elements out of control limits, as "J+."</i></p> <p><i>If > 200%, qualify all positive data, for those elements out of control limits, as "R."</i></p>	

4.2 Metal Spike Recovery	
4.2.6	<p>If the pre-digestion spike was outside the QC limits for Atomic Adsorption furnace analysis (e.g., SW-846 methods in the 7000 series), was a post-digestion spike performed?</p> <p><i>Action: Samples should not be qualified based on post-digestion spike results. The results are used to confirm a matrix interference. If a post-digestion spike was not prepared, the data validator may reject the data.</i></p>
4.2.7	<p>Based on the results from 4.2.6, were the post-digestion spike recoveries within the quality control range (75% to 125%)?</p> <p><i>Action: If > 125%, qualify all positive data as "J+". If < 75%, qualify both positive and non-detect data as estimated and flag this data with either a "J-" or "UJ".</i></p>

4.3 Quality Assurance Data Review - Inorganic Analysis - AA Analysis	
<p>Graphite Furnace Atomic Adsorption QC</p> <p>Atomic Adsorption analyses (SW-846 7000 series methods) require specialized QA/QC procedures that may be different than Inductively Coupled Plasma (ICP) Emission Analysis. Commonly, AA analysis is performed for mercury and selenium. Mercury analysis data validation is specifically detailed in the Inorganics Section of the Tier II Checklist. The Tier I Data Validator is directed to the Agency's Data Validation Review Manual and to specific methods detailed in SW-846. In general, external calibration procedures are commonly required by the method. In addition, duplicate injections and multiple concentration post-digestion spikes are required to establish precision and accuracy data.</p>	
4.3.1	<p>Were duplicate injection of samples performed and if so, were the duplicates within $\pm 20\%$ RPD for samples with concentrations above the detection limit?</p> <p>Note: Results are reported based upon the average of duplicate injections. If the acceptance criteria is not met, the sample should have been re-analyzed (i.e., with at least two additional injections).</p> <p><i>Action: If RSD criteria is not met or the sample was not rerun, qualify all positive data as "J."</i></p>
4.3.2	<p>If the samples were re-analyzed (i.e., 2 more injections), do the duplicate injections agree within 20% RSD?</p> <p><i>Action: If the RSD criteria is not met, qualify all positive results as "J."</i></p>
4.3.3	<p>Were Matrix Spike/Matrix Spike Duplicates analyzed at a rate of 1 in 20 or per batch?</p> <p><i>Action: If no MS/MSD were analyzed, qualify all positive results as "J" and all undetected results as "UJ."</i></p>

4.4 Spikes - Mercury Analysis	
<p>4.4.1 Was a matrix spike analyzed at required frequency (one pre-digestion for each group of samples with a similar matrix type and concentration or sample delivery group) and within limits?</p> <p>Note: Post-digestion spikes are not required for Mercury. However one typically is run if the LCS was out of control in order to show matrix interference.</p> <p><i>Action: If the spike recovery is greater than 125 % and the sample results are below the detection limit, the data is acceptable.</i></p> <p><i>If the spike recovery is greater than 125% or less than 75%, and the sample results are greater than the detection limit, then the positive data should be qualified as estimated ("J+" or "J-").</i></p> <p><i>If the spike recovery falls within the range of 30 to 74%, all non-detected data should be qualified as "UJ." All positive data should be qualified as estimated and flagged as "J-".</i></p> <p><i>If the spike recovery is less than 30% and the sample results are below the detection limit, qualify these results as rejected and flagged this data as "R."</i></p>	
<p>4.4.2 If the analyte concentration in the original sample is a factor of 50 above the IDL, was a serial dilution analysis performed and did it agree within a 10% difference of the original determination after correction for dilution?</p>	
<p>4.4.3 Was an LCS analyzed per batch and within QC limits (80 to 120%)? (An LCS is not required for aqueous samples of Mercury.)</p> <p>Note: The results for solid LCS should always be within the control limits. The laboratory should terminate the analysis, correct the problem, and the samples should be re-digested and re-analyzed for mercury.</p> <p><i>Action: If the LCS is outside of the control limit, qualify all positive results as estimated ("J+" or "J-").</i></p> <p><i>If the LCS results are higher than control limits and the sample results are below the detection limit, the results are acceptable.</i></p> <p><i>If the LCS result is below the lower control limit, initially qualify all results below the detection limit as "UJ.". Non-detected compounds may be qualified as rejected "R" based upon professional judgment and the project's DQOs.</i></p>	

Section 5.0 Data Validation Summary

5.0 Data Validation Summary	
<p>Data Validation Summary</p> <p>The results of the data validation must be summarized to be useful in making decisions concerning the use of the analytical data. The final decision on whether the data is usable for its intended purpose must be made in conjunction with the project management team and with the stated data quality objects for the project. The following items can be used as a general guideline on preparing a data validation summary. More information can be found in Chapter 14 of the Data Validation Manual.</p>	
5.1	State the regulatory requirement that prompted the samples to be taken.
5.2	List the Data Quality Objects for the sampling
5.3	Summarize the findings of each major category of quality assurance data (e.g. blanks, surrogates, spikes, etc.)
5.4	<p>Assess whether bias is present.</p> <p>Note: This can be accomplished qualitatively by reviewing the qualified QA/QC data. If the majority of the QA/QC data are flagged with a J-, then there may be a negative bias present. If the majority of the QA/QC data is flagged with a J+, then there may a positive bias. Additional information on the assessment of bias can be found in U.S. EPA's Guidance for <i>Data Quality Assessment: Practical Methods for Data Analysis (QA/G-9) EPA/600/R-96/084, July, 2000.</i></p>
5.5	Is the quality of the data sufficient to meet the data quality objectives of the project?