

## Draft Data Validation Plan Review Form Tier II

This Plan Review Form is #		of		forms completed in the review of this closure plan.
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Facility Name		Validator/DO	
ID Number		Date of Report	
Date Review of Report is Completed		Report is: New, Amended, Revised	
Sample Report Title:			
Lab Name:	Media Type(s):	Analyses Requested:	Notes:
Tier I Report Attached?		Tier I Report Completed by:	
		Date:	

Data Qualifiers and their meanings used throughout the Tier II 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

Note: The criteria used in the Tier II Data Validation checklist are derived mostly 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 rules), ASTM method criteria are used.

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.

**Tier II**

**Section 1**

**Volatile Organic Data Review**

1.0 Volatile Organic Data Review - Initial Calibration																					
<b>Initial Calibration</b>																					
<p><b>Organic methods rely on an Initial Calibration in which 5-9 different concentrations of all target analytes are analyzed to establish a “working range” for sample analysis. The results of this initial calibration may be used for a period of weeks in which Initial Calibration Verification (ICV) Quality Control (QC) samples and Continuing Calibration Verification (CCV) QC samples will be analyzed to confirm the system is still operating as it was during the Initial Calibration. Problems with the Gas Chromatograph (GC) column (e.g., organic “junk” accumulating in the end) may require that the end of the column be clipped off. As the retention time data is modified from this action, the need for a new initial calibration will be triggered. Samples from this point on must all refer back to this new initial calibration date.</b></p>																					
<p>1.1 Is the GC/MS system hardware-tuned to meet the criteria for 5-50 ng injection or purging of 4-bromofluorobenzene (BFB) prior to sample analysis?</p> <p><b>Note: The ion abundance criteria used in this checklist is from SW-846, Method 8260B. The U.S. EPA’s National Functional Guidelines have different criteria.</b></p> <table border="0"> <thead> <tr> <th style="text-align: left;"><u>m/z</u></th> <th style="text-align: left;"><u>Ion Abundance Criteria</u></th> </tr> </thead> <tbody> <tr> <td>50</td> <td>15% to 45% of mass 95</td> </tr> <tr> <td>75</td> <td>30% to 60% of mass 95</td> </tr> <tr> <td>95</td> <td>Base peak, 100% relative abundance</td> </tr> <tr> <td>96</td> <td>5% to 9% of mass 95</td> </tr> <tr> <td>173</td> <td>0% to &lt;2% of mass 174</td> </tr> <tr> <td>174</td> <td>&gt;50% of mass 95</td> </tr> <tr> <td>175</td> <td>5% to 9% of mass 174</td> </tr> <tr> <td>176</td> <td>&gt;95% but &lt;101% of mass 174</td> </tr> <tr> <td>177</td> <td>5% to 9% of mass 176</td> </tr> </tbody> </table> <p><i>Action: If the initial tune was not performed correctly, all data generated using this initial calibration should be qualified “R.”</i></p>	<u>m/z</u>	<u>Ion Abundance Criteria</u>	50	15% to 45% of mass 95	75	30% to 60% of mass 95	95	Base peak, 100% relative abundance	96	5% to 9% of mass 95	173	0% to <2% of mass 174	174	>50% of mass 95	175	5% to 9% of mass 174	176	>95% but <101% of mass 174	177	5% to 9% of mass 176	<p>Provide details on which ion abundance is out of compliance. In addition, list all affected sample IDs:</p>
<u>m/z</u>	<u>Ion Abundance Criteria</u>																				
50	15% to 45% of mass 95																				
75	30% to 60% of mass 95																				
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176	>95% but <101% of mass 174																				
177	5% to 9% of mass 176																				
<p>1.2 Has the GC/MS system hardware been tuned to meet BFB tuning criteria within 12 hours prior to sample analysis?</p> <p><b>Note: Tuning is valid up to 12 hours after tuning analysis. Assess whether the laboratory tuned the instrument at the required time. If necessary, obtain a run log chart from the laboratory to confirm the time of tuning.</b></p> <p><i>Action: If the tune was not performed at the required time, the Data Validator may qualify all data as “R” or “J.”</i></p>																					
<p>1.3 Was the initial calibration performed with a minimum of five concentration levels for each target analyte?</p> <p><b>Note: An additional data source on calibration procedures is the laboratory’s QAP.</b></p> <p><i>Action: If the initial calibration did not include at least five different concentration levels, require that the laboratory provide an explanation. The Data Validator may, at his or her discretion, qualify the data as “J” or “R,” depending on the number and concentration range of the calibration standards. This information should be included in the bench sheets provided by the laboratory. If it is not, the laboratory should be notified and information submitted to the agency.</i></p>	<p><u>Calibration Date:</u></p> <p><u>Instrument ID:</u></p> <p><u>Sample ID:</u></p> <p><u>Calibration Concentrations:</u></p>																				

1.0 Volatile Organic Data Review - Initial Calibration							
<p>1.4 A. Is the percent Relative Standard Deviation (%RSD) for individual analytes in the initial calibration below 15% RSD? B. If not, are alternate calibration equations used for those analytes not meeting the 15% criteria? C. Is the <u>average</u> %RSD for all analytes below 15% RSD?</p> <p><b>Note: Method 8260B requires the RSD to be 15% (or lower) as evidence of sufficient linearity to employ an average relative response factor (RRF). A laboratory may choose other criteria. For example, the NFGs specify 30% maximum RSD. Method 8000 (Section 7.5.1.2) specifies a linearity criterion of 20% RSD. That criterion pertains to GC and High Performance Liquid Chromatography methods other than GC/MS.</b></p> <p>The reviewer should be aware that the following compounds may display poor response and linearity. The acceptance criteria for these compounds are the same as for other target analytes, but the Data Validator may use this knowledge in his or her data assessment.</p> <table><tr><td>Chloroform</td><td>1,1-Dichloroethene</td></tr><tr><td>Ethyl benzene</td><td>1,2-Dibromo-3-chloropropane</td></tr><tr><td>Toluene</td><td>Vinyl chloride</td></tr></table> <p><i>Action: If the %RSD for each analyte is below 15%, no qualification is necessary.</i></p> <p><i>If one or two analytes had a low level calibration point removed to better the %RSD calculation, and the detection limit was not raised, qualify all undetected data as estimated. Positive data should not be qualified. If high level calibration points were removed, "J" any data points that are above the remaining calibration standard concentration levels.</i></p> <p><i>If the average RSD for all calibration analytes is above 15% and average response factors are used, qualify all positive and undetected data for those individual analytes that exceeded the 15% RSD criteria requirement as estimated .</i></p> <p><i>Additional qualification, including rejection of data, may be warranted if other quality control criteria is not within requirements.</i></p>	Chloroform	1,1-Dichloroethene	Ethyl benzene	1,2-Dibromo-3-chloropropane	Toluene	Vinyl chloride	<p><b>Additional Information: The laboratory has the option of using linear regression or quadratic calibration equations for individual analytes that don't meet the 15% criteria. Information concerning the acceptability for these alternate calibration equations must be supplied by the laboratory.</b></p> <p>The laboratory also has the option to re-analyze a particular calibration standard if it is suspected that the standard has degraded. In addition, the lab may remove either the low level or high level calibration point from the %RSD analysis for <u>up to two</u> individual analytes to bring the %RSD within criteria.</p> <p>If certain individual analytes have %RSDs above the criteria, but the average of all the analytes is below 15% RSD, then the average RRF is acceptable for use.</p> <p>Record the analytes in the initial calibration that used an alternate calibration curve or had %RSD values greater than 15%.</p>
Chloroform	1,1-Dichloroethene						
Ethyl benzene	1,2-Dibromo-3-chloropropane						
Toluene	Vinyl chloride						

1.0 Volatile Organic Data Review - Initial Calibration															
<p>1.5 <b>Optional Question:</b> Does the calibration curve used correspond to the appropriate sample introduction technique employed to generate the calibration curve?</p> <p>Method 5030, 5mL (water analysis) Method 5030, 25mL (low level water analysis) Method 5035, soils (excluding TCLP) Method 5035, wastes</p> <p><b>Note: Methods 5030 and 5035 are purge and trap techniques for water and soil, respectively. SW-846, Method 5030 usually requires a 5 mL volume. A 25 mL volume is usually required by drinking water methods (it results in a lower detection limit). Method 5035 requires 5 grams of soil or waste for extraction.</b></p> <p><i>Action: If sample volume is inconsistent with the method of calibration, all associated data should be qualified as "R."</i></p>															
<p>1.6. The Initial Calibration RRFs for the volatile target compounds and surrogates listed below must be greater than or equal to 0.01. The RRF for all other volatile target compounds and surrogates (<b>S</b>) must be greater than or equal to 0.05.</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%;"><b>Acetone</b></td> <td style="width: 50%;"><b>1,2-Dichloropropane</b></td> </tr> <tr> <td><b>2-Butanone</b></td> <td><b>1,2-Dibromo-3-chloropropane</b></td> </tr> <tr> <td><b>Carbon Disulfide</b></td> <td><b>4-Methyl-2-pentanone</b></td> </tr> <tr> <td><b>Chloroethane</b></td> <td><b>2-Hexanone</b></td> </tr> <tr> <td><b>Chloromethane</b></td> <td><b>1,2-Dichloropropane-d<sub>6</sub> (S)</b></td> </tr> <tr> <td><b>Cyclohexane</b></td> <td><b>2-Hexanone-d<sub>5</sub> (S)</b></td> </tr> <tr> <td><b>Chloroethane-d<sub>5</sub> (S)</b></td> <td><b>2-Butanone-d<sub>5</sub> (S)</b></td> </tr> </table> <p><i>Action: If any volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" listed above, and 0.05 for all other volatile compounds), use professional judgment for positive results to qualify the data as "J" or "R."</i></p> <p><i>If any volatile target compound in the initial calibration has an RRF value less than the minimum criterion (0.01 for the "poor performers" listed above, and 0.05 for all other volatile compounds), qualify non-detected compounds as "R."</i></p>	<b>Acetone</b>	<b>1,2-Dichloropropane</b>	<b>2-Butanone</b>	<b>1,2-Dibromo-3-chloropropane</b>	<b>Carbon Disulfide</b>	<b>4-Methyl-2-pentanone</b>	<b>Chloroethane</b>	<b>2-Hexanone</b>	<b>Chloromethane</b>	<b>1,2-Dichloropropane-d<sub>6</sub> (S)</b>	<b>Cyclohexane</b>	<b>2-Hexanone-d<sub>5</sub> (S)</b>	<b>Chloroethane-d<sub>5</sub> (S)</b>	<b>2-Butanone-d<sub>5</sub> (S)</b>	
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<p>1.7 Do the average RRFs for the System Performance Check Compounds (SPCCs) in the initial calibration standards meet the following acceptance criteria?</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 70%;"><b>Chloromethane</b></td> <td style="width: 30%;"><b>&gt; 0.10</b></td> </tr> <tr> <td><b>1,1-Dichloroethane</b></td> <td><b>&gt; 0.10</b></td> </tr> <tr> <td><b>Bromoform</b></td> <td><b>&gt; 0.10</b></td> </tr> <tr> <td><b>Chlorobenzene</b></td> <td><b>&gt; 0.30</b></td> </tr> <tr> <td><b>1,1,2,2-Tetrachloroethane</b></td> <td><b>&gt; 0.30</b></td> </tr> </table> <p><b>Note: SPCCs not within criteria indicate that the integrity of analytical system has been compromised. Corrective action should be performed by the laboratory before recalibration is attempted. If corrective action was not performed use the following action to qualify data.</b></p> <p><i>Action: For any target analyte with an RF &lt; 0.05, qualify associated non-detects as "R" and flag associated positive data as "J."</i></p>	<b>Chloromethane</b>	<b>&gt; 0.10</b>	<b>1,1-Dichloroethane</b>	<b>&gt; 0.10</b>	<b>Bromoform</b>	<b>&gt; 0.10</b>	<b>Chlorobenzene</b>	<b>&gt; 0.30</b>	<b>1,1,2,2-Tetrachloroethane</b>	<b>&gt; 0.30</b>	<p>List affected sample IDs and the rationale for qualification:</p>				
<b>Chloromethane</b>	<b>&gt; 0.10</b>														
<b>1,1-Dichloroethane</b>	<b>&gt; 0.10</b>														
<b>Bromoform</b>	<b>&gt; 0.10</b>														
<b>Chlorobenzene</b>	<b>&gt; 0.30</b>														
<b>1,1,2,2-Tetrachloroethane</b>	<b>&gt; 0.30</b>														

1.0 Volatile Organic Data Review - Initial Calibration	
<p>1.8 Are the relative response factors for the Calibration Check Compounds (CCCs) within acceptable limits of %RSD?</p> <p><b>Note: The relative standard deviation criteria for stability is: %RSD &lt; 15% for all analytes <u>except</u> for CCCs where the relative standard deviation criteria is: %RSD &lt; 30%. CCCs are considered to be “poor performers,” and are singled out for evaluation with the idea that if they are within acceptable criteria then other compounds are also likely acceptable. The CCCs are:</b></p> <p><b>1,1-Dichloroethene Chloroform Toluene 1,2-Dichloropropane Ethylbenzene Vinyl Chloride</b></p> <p><i>Action: If %RSD &gt; 30% for any CCC, then the lab should have taken action to eliminate system leaks and/or column reactive sites before attempting re-calibration.</i></p> <p><i>If the analysis was performed and the %RSD &lt; 15% in the initial calibration for compounds other than CCCs, qualify positive results for those analytes as “J” and un-detected analytes as “R.”</i></p> <p><i>When %RSD &gt; 90%, flag all non-detects for that analyte as “R.”</i></p>	<p>List the response factors that are out of compliance and the %RSD. List all affected sample IDs:</p>

**Table 1.0  
System Performance Check Compounds and Calibration Check Compounds  
For SW-846, Method 8260B**

SPCC	Minimum Average Response Factor	CCC	%RSD
Chloromethane	0.10	1,1-Dichloroethene	< 30%
1,1-Dichloroethane	0.10	Chloroform	< 30%
Bromofom	0.10	1,2-Dichloropropane	< 30%
Chlorobenzene	0.30	Toluene	< 30%
1,1,2,2-Tetrachloroethane	0.30	Ethylbenzene	< 30%
		Vinyl Chloride	< 30%

*Prior to analysis of samples, the tuning criteria of the instrument must be evaluated by injection of a 5ng to 50ng aliquot of 4-Bromofluorobenzene into the instrument. In addition, the initial calibration must be verified every twelve hours by analyzing a calibration standard (referred to as a 12-hour standard or sometimes an Initial Calibration Verification (ICV) or a Continuing Calibration Verification (CCV)) near the mid-point of the calibration curve. System Performance Check Compounds (SPCC) in this standard must be examined every twelve hours and meet the criteria listed in the above table. Calibration Check Compounds (CCC), also in the ICV and/or CCV, must be examined to determine the validity of the initial calibration. These compounds must meet the criteria listed in this table. If problems are encountered, a new 5-point calibration should be performed.*

2.0 Volatile Organic Data Review - Continuing Calibration	
Initial Calibration Verification and Continuing Calibration	
<p><b>A continuing calibration standard for both target compounds and surrogates must be analyzed every 12 hours following analysis of the instrument performance check sample (tune) and before the analyses of method blanks and samples. The continuing calibration standards must all have Relative Response Factors (RRFs) greater than or equal to 0.05 except for SPCCs and CCCs, and the initial calibration RRF and the continuing calibration RRF should be within +/- 25% Difference (%D). The Tier II Data Validator must verify that the continuing calibration was analyzed at the proper frequency and in the proper sequence. In addition, at least one target compound's continuing calibration RRF should be recalculated and compared to the reported result.</b></p>	
<p>2.1 Has a continuing calibration verification (CCV) or 12-hour standard (a.k.a. midpoint calibration standard) been analyzed for every 12 hours of sample analysis?</p> <p><i>Action: List all sample analyses that were not within 12 hours of the previous continuing calibration analysis. Call laboratory for an explanation or re-submission of the data. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	
<p>2.2 Were samples analyzed within 12 hours of either the initial calibration or the 12-hour standard?</p> <p><i>Action: If samples were not analyzed within the 12-hour window, all positive results should be qualified as "J." If other criteria are also outside of requirements, the Data Validator may qualify data as "R."</i></p>	
<p>2.3 The continuing calibration RRFs in the 12-hour standard for the volatile target compounds and surrogates listed in question 1.6 must be greater than or equal to 0.01. The RRF for all other volatile target compounds and surrogates must be greater than or equal to 0.05.</p> <p><i>Action: If any volatile target compound has an RRF value less than the minimum criterion (0.01 for those compounds listed in question 1.6 and 0.05 for all other volatile compounds), use professional judgment for positive results to qualify the data as "J" or "R."</i></p> <p><i>If any volatile target compound has an RRF value less than the minimum criterion (0.01 for those compounds listed in question 1.6 and 0.05 for all other volatile compounds), qualify non-detected compounds as "R."</i></p>	
<p>2.4. The Percent Difference (%D) between the initial calibration average RRF and the 12-hour standard continuing calibration RRF must be within <math>\pm 50.0\%</math> for the volatile target compounds and surrogates listed in question 1.6. The %D for all other volatile target compounds and surrogates must be within <math>\pm 25.0\%</math>.</p> <p><i>Action: If %D value for any of the volatile target compounds listed in question 1.6 are outside the <math>\pm 50.0\%</math> criterion, qualify positive results as "J" and non-detected compounds as "UJ."</i></p> <p><i>If %D value for any other volatile target compound is outside the <math>\pm 25.0\%</math> criterion, qualify positive results as "J" and non-detected compounds as "UJ."</i></p>	

2.0 Volatile Organic Data Review - Continuing Calibration	
<p>2.5 Do the RRFs of the SPCCs (listed in Table 1.0) in the 12-hour standard meet the initial SPCC criteria (Table 1.0) for each 12-hour shift?</p> <p><b>Note: RRFs in the ICV or CCV should be compared to initial calibration RFs.</b></p> <p><i>Action: Corrective action should be taken to solve the source of the problem. If the source of the problem could not be determined, a new 5-point calibration should have been generated. List all sample analyses that were affected. Call the laboratory for explanation or re-submission. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	
<p>2.6 The system should be re-calibrated if any of the SPCCs fail to meet minimum RRF or maximum %D criteria.</p> <p><b>Note: For data review purposes, all compounds must be considered for qualification when the %D exceeds the 50% criterion.</b></p> <p><i>Action: Ask the laboratory for an explanation. If analyses were performed without corrective action, initially "R" all data. Upon explanation, the Tier II Data Validator may use best professional judgement to qualify positive sample results as "J-."</i></p>	
<p>2.7 Do the RRFs of the SPCCs (Table 1.0) in any batch specific CCV standard (or 12-hour standard) meet the initial SPCC criteria (Table 1.0)?</p> <p><b>Note: RRFs in the CCV are compared to initial 12-hour calibration RFs.</b></p> <p><i>Action: Corrective action should be taken to solve the source of the problem. If the source of the problem could not be determined, a new 5-point calibration should have been generated. List all sample analyses that were affected. Call the laboratory for explanation or re-submission. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	
<p>2.8 Do any Calibration Check Compounds (CCCs) in the CCV have a % Difference (%D) between the 12-hour standard and the initial calibration which exceeds the +/- 25 % criteria?</p> <p><b>Note: The lab may establish their own criteria (e.g., +/- 20%).</b></p> <p><i>Action: Corrective action should be taken to solve the source of the problem. If the source of the problem could not be determined, a new 5-point calibration should have been generated. List all sample analyses that were affected. Call the laboratory for explanation or re-submission. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	

2.0 Volatile Organic Data Review - Continuing Calibration	
<p>2.9 The Tier II Data Validator should evaluate relative response factors (RRFs) for the target compounds, surrogates, 12-hour standards, and any batch specific continuing calibration verification. The RRFs in the CCV should be greater than 0.05.</p> <p><b>Note: A 12-hour standard is needed once per day and a continuing calibration verification is needed for each batch or every 20 samples. The National Functional Guidelines (NFGs) allow that any 2 volatile target compounds may fail to meet minimum RRF or maximum %D criteria as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0 percent.</b></p> <p><i>Action: If the RRF is less than 0.05, qualify all positive results as "J" and all undetected compounds as "R."</i></p>	

**Table 2**  
**Internal Standards (IS) and Associated Target Compounds for SW-846, Method 8260B**

Bromochloromethane (IS)	1,4-Difluorobenzene (IS)	Chlorobenzene-d <sub>5</sub> (IS)
Chloromethane	1,1,1,-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	Bromoform	1,1,2,2,-Tetrachloroethane
Methylene Chloride	1,2-Dichloropropane	Toluene
Acetone	Trans-1,3-Dichloropropane	Chlorobenzene
Carbon Disulfide	Trichloroethene	Ethylbenzene
1,1-Dichloroethene	Dibromochloromethane	Styrene
1,1-Dichloroethane	1,1,2-Trichloroethane	Total Xylenes
1,2-Dichloroethene(total)	Benzene	Bromofluorobenzene (surr.)
Chloroform	cis-1,3-Dichloropropene	Toluene-d <sub>8</sub> (surr.)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d <sub>4</sub> (surr.)		

3.0 Volatile Organic Data Review - Internal Standards
<b>Internal Standards</b>
<p><b>Internal standards are the primary means of identifying and quantifying volatile organic compounds in samples. The evaluation of internal standard data is therefore critical to data validation. Internal standards are added to each sample and all QC samples and are evaluated in comparison to the initial calibration. Internal standard area counts must not vary by more than a factor of two (-50 to +/- 100%) from the associated 12-hour calibration verification standard. In addition, the retention time of internal standards must not vary by more than +/- 30 seconds from the associated 12-hour calibration verification standard. The internal standards and their associated target compounds are listed in Table 2.</b></p>

<b>3.0 Volatile Organic Data Review - Internal Standards</b>	
<p>3.1 Are area counts available to determine if the sample internal standards are within criteria as compared to the 12-hour standard?</p> <p><i>Action: If the data is not present, the Data Validator should request that the facility obtain the data from its laboratory. If the data is not available, the Tier II Data Validator may qualify data based upon professional judgement.</i></p>	<p>Record actions taken, such as when the facility was contacted, names of facility personnel and when the information was submitted:</p>
<p>3.2 Is the area counts for the internal standards in a sample outside of the <math>\pm 50\%</math> range of the area counts of the 12-hour standard?</p> <p><i>Action: All positive results associated with an internal standard outside of the criteria should be qualified as "J."</i></p> <p><i>Non-detected compounds quantified using an internal standard whose area count is greater than 100% should not be qualified.</i></p> <p><i>Non-detected compounds quantified with an internal standard whose area count is less than 50% of the associated calibration standard should be qualified as "UJ."</i></p> <p><i>If extremely low area counts are associated with the internal standards, qualify all associated target compounds as "R."</i></p>	
<p>3.3 Do any internal standards retention times vary by more than 30 seconds (0.5 minutes) as compared to the 12-hour standard?</p> <p><i>Action: The Tier II Data Validator should request raw data chromatograms and assess whether false positives or negatives exist for any target compounds associated with that internal standard. Based upon this evaluation, the data may either be qualified as "J" or "R" using professional judgement.</i></p>	

<b>4.0 Volatile Organic Data Review - Target Compound Identification</b>	
<b>Target Compound Identification</b>	
<p><b>One of the primary goals of sample analyses is to accurately identify the presence of target compounds. The procedure used by SW-846, Method 8260B to identify the presence of target compounds is to compare the retention time of unknown compounds in a sample to the retention time of known compounds in the calibration standard. The Relative Retention Times (RRT) of the compounds in the samples must be within <math>\pm 0.06</math> RRT units of the RRTs of the same compound in the initial calibration standards. In addition, the mass spectra of the standard must match within certain tolerances of the mass spectra of the calibration standard. For example, all ions in the standard mass spectra at a relative intensity of 10% must also be present in the sample spectrum. In addition, the relative intensities of the ions in the sample must agree within <math>\pm 20\%</math> between the sample and the standard spectra. Ions that are present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be considered and accounted for.</b></p>	
<p>4.1 Are the Relative Retention Times (RRTs) of reported compounds within <math>\pm 0.06</math> RRT units of the RRT for the same compounds in the initial calibration?</p> <p><i>Action: Use this information in conjunction with the mass spectral analysis information to qualify data. If a target compound identification is in error, qualify that compound as "R."</i></p>	

<b>4.0 Volatile Organic Data Review - Target Compound Identification</b>	
<p>4.2 Check the sample compound spectra against spectral data of the standard. All ions in the standard mass spectra at a relative intensity of 10% must also be in the identified sample compound.</p> <p><i>Action: Use this information in conjunction with the mass spectral analysis information to qualify data. If target compound identification is in error, qualify that compound as "R."</i></p>	
<p>4.3 Check the sample compound spectra against spectral data of the standard. The relative intensity of the ions in the sample must agree within +/- 20% of the value obtained from the standard spectrum.</p> <p><i>Action: Use this information in conjunction with the mass spectral analysis information to qualify data. If target compound identification is in error, qualify that compound as "R."</i></p>	

**Tier II**  
**Section 2**  
**Semi-Volatile Data Review**

1.0 Semi-Volatile Data Review - Initial Calibration																											
<b>Initial Calibration</b>																											
<p>Semi-volatile organic methods rely on an Initial Calibration in which 5-9 different concentrations of all target analytes are analyzed to establish a “working range” for sample analysis. The results of this initial calibration may be used for a period of weeks in which the initial calibration will be confirmed by Continuing Calibration Verification (CCV) Quality Control (QC) samples. Problems with the Gas Chromatograph (GC) column (e.g., organic “junk” accumulating in the end) may require that the end of the column be clipped off. As the retention time data is modified from this action, the need for a new Initial Calibration must be triggered. Samples from this point on may all refer back to this new initial calibration date.</p>																											
<p>1.1 Is the Gas Chromatograph/Mass Spectrometer (GC/MS) system tuned to meet the criteria for 50 ng injection of Decafluorotriphenylphosphine (DFTPP) prior to sample analysis?</p> <p><b>Note: The ion abundance criteria used in this checklist is from SW-846, Method 8270C. U.S. EPA’s National Functional Guidelines have slightly different criteria.</b></p> <table border="0"> <thead> <tr> <th style="text-align: left;"><u>Mass</u></th> <th style="text-align: left;"><u>Ion Abundance Criteria</u></th> </tr> </thead> <tbody> <tr> <td>51</td> <td>30% to 80% of mass 198</td> </tr> <tr> <td>68</td> <td>&lt;2% of mass 69</td> </tr> <tr> <td>70</td> <td>&lt;2% of mass 69</td> </tr> <tr> <td>127</td> <td>25% to 75% of mass 198</td> </tr> <tr> <td>197</td> <td>&lt;1% of mass 198</td> </tr> <tr> <td>198</td> <td>Base peak, 100% relative abundance</td> </tr> <tr> <td>199</td> <td>5% to 9% of mass 198</td> </tr> <tr> <td>275</td> <td>10% to 30% of mass 198</td> </tr> <tr> <td>365</td> <td>&gt;0.75% of mass 198</td> </tr> <tr> <td>441</td> <td>Present but less than mass 443</td> </tr> <tr> <td>442</td> <td>&gt;40-110% of mass 198</td> </tr> <tr> <td>443</td> <td>15% to 24% of mass 442</td> </tr> </tbody> </table> <p><b>Note: All ion abundances must be normalized to m/z 198 (except the ones that are normalized to m/z 69, as noted above).</b></p> <p><i>Action: If a base peak, other than m/z 198 is used for normalization, then qualify all associated data as “R.”</i></p> <p><i>If the ion abundance criteria is not met then judgement should be used to qualify associated data as “J” or “R.”</i></p>	<u>Mass</u>	<u>Ion Abundance Criteria</u>	51	30% to 80% of mass 198	68	<2% of mass 69	70	<2% of mass 69	127	25% to 75% of mass 198	197	<1% of mass 198	198	Base peak, 100% relative abundance	199	5% to 9% of mass 198	275	10% to 30% of mass 198	365	>0.75% of mass 198	441	Present but less than mass 443	442	>40-110% of mass 198	443	15% to 24% of mass 442	<p>Indicate samples affected and qualification of sample results:</p>
<u>Mass</u>	<u>Ion Abundance Criteria</u>																										
51	30% to 80% of mass 198																										
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<p>1.2 Has the GC/MS system hardware been tuned to meet DFTPP tuning criteria within 12 hours prior to sample analysis?</p> <p><b>Note: Tuning is valid up to 12 hours after tuning analysis. Assess whether the laboratory tuned the instrument at the required time(s). If necessary, obtain a run log chart from the laboratory to confirm the time of tuning.</b></p> <p><i>Action: If the tune was not performed at the required time, the Data Validator may qualify all data as “R” or “J.”</i></p>																											

1.0 Semi-Volatile Data Review - Initial Calibration									
<p>1.3 Is the initial calibration performed with a minimum of five concentration levels for each target analyte?</p> <p><b>Note: Typical calibration levels are between 5 and 160 µg/L. The initial calibration verification is analyzed every twelve hours or when the CCV indicates a need. The Data Validator should record the date of the initial calibration, the instrument ID, the sample ID associated with each instrument, and the calibration concentrations used.</b></p> <p><i>Action: No qualification should be taken on the number of calibration standards alone, but any discrepancies should be noted and used in conjunction with other information to qualify data.</i></p>	<p><u>Calibration Date:</u></p> <p><u>Instrument ID:</u></p> <p><u>Sample ID:</u></p> <p><u>Calibration Concentrations Used:</u></p>								
<p>1.4 Initial calibration standard Relative Response Factors (RRFs) for the semi-volatile target compounds and surrogates listed below must be greater than or equal to 0.010. The RRF for all other semi-volatile target compounds and surrogates (<b>S</b>) must be greater than or equal to 0.05.</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%; vertical-align: top;"> <p><b>2,2'-Oxybis(1-Chloropropane)</b></p> <p><b>4-Chloroaniline</b></p> <p><b>Hexachlorobutadiene</b></p> <p><b>Hexachlorocyclopentadiene</b></p> <p><b>2-Nitroaniline</b></p> <p><b>3-Nitroaniline</b></p> <p><b>2,4-Dinitrophenol</b></p> <p><b>4-Nitrophenol</b></p> <p><b>Acetophenone</b></p> <p><b>4,6-Dinitro-2-methylphenol-d<sub>2</sub> (S)</b></p> </td> <td style="width: 50%; vertical-align: top;"> <p><b>Benzaldehyde</b></p> <p><b>Pentachlorophenol</b></p> <p><b>4-Nitroaniline</b></p> <p><b>4,6-Dinitro-2-methylphenol</b></p> <p><b>N-Nitrosodiphenylamine</b></p> <p><b>3-3'-Dichlorobenzidine</b></p> <p><b>4-Chloroaniline-d<sub>4</sub> (S)</b></p> <p><b>4-Nitrophenol-d<sub>4</sub> (S)</b></p> <p><b>Caprolactam</b></p> </td> </tr> </table> <p><i>Action: If any semi-volatile target compound has an RRF value less than the minimum criterion (0.01 for the compounds listed above, and 0.05 for all other semi-volatile compounds), use professional judgment for positive results to qualify the data as "J" or "R."</i></p> <p><i>If any semi-volatile target compound has an RRF value less than the minimum criterion (0.01 for the compounds listed above, and 0.05 for all other semi-volatile compounds), qualify non-detected compounds as "R."</i></p>	<p><b>2,2'-Oxybis(1-Chloropropane)</b></p> <p><b>4-Chloroaniline</b></p> <p><b>Hexachlorobutadiene</b></p> <p><b>Hexachlorocyclopentadiene</b></p> <p><b>2-Nitroaniline</b></p> <p><b>3-Nitroaniline</b></p> <p><b>2,4-Dinitrophenol</b></p> <p><b>4-Nitrophenol</b></p> <p><b>Acetophenone</b></p> <p><b>4,6-Dinitro-2-methylphenol-d<sub>2</sub> (S)</b></p>	<p><b>Benzaldehyde</b></p> <p><b>Pentachlorophenol</b></p> <p><b>4-Nitroaniline</b></p> <p><b>4,6-Dinitro-2-methylphenol</b></p> <p><b>N-Nitrosodiphenylamine</b></p> <p><b>3-3'-Dichlorobenzidine</b></p> <p><b>4-Chloroaniline-d<sub>4</sub> (S)</b></p> <p><b>4-Nitrophenol-d<sub>4</sub> (S)</b></p> <p><b>Caprolactam</b></p>							
<p><b>2,2'-Oxybis(1-Chloropropane)</b></p> <p><b>4-Chloroaniline</b></p> <p><b>Hexachlorobutadiene</b></p> <p><b>Hexachlorocyclopentadiene</b></p> <p><b>2-Nitroaniline</b></p> <p><b>3-Nitroaniline</b></p> <p><b>2,4-Dinitrophenol</b></p> <p><b>4-Nitrophenol</b></p> <p><b>Acetophenone</b></p> <p><b>4,6-Dinitro-2-methylphenol-d<sub>2</sub> (S)</b></p>	<p><b>Benzaldehyde</b></p> <p><b>Pentachlorophenol</b></p> <p><b>4-Nitroaniline</b></p> <p><b>4,6-Dinitro-2-methylphenol</b></p> <p><b>N-Nitrosodiphenylamine</b></p> <p><b>3-3'-Dichlorobenzidine</b></p> <p><b>4-Chloroaniline-d<sub>4</sub> (S)</b></p> <p><b>4-Nitrophenol-d<sub>4</sub> (S)</b></p> <p><b>Caprolactam</b></p>								
<p>1.5 Do the average response factors (RFs) generated from the initial calibration curve for the System Performance Check Compounds (SPCCs) meet the following acceptance criteria:</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 70%;"><b>N-Nitroso-di-n-propylamine</b></td> <td style="text-align: right;"><b>&gt; 0.05</b></td> </tr> <tr> <td><b>Hexachlorocyclopentadiene</b></td> <td style="text-align: right;"><b>&gt; 0.05</b></td> </tr> <tr> <td><b>2,4-Dinitrophenol</b></td> <td style="text-align: right;"><b>&gt; 0.05</b></td> </tr> <tr> <td><b>4-Nitrophenol</b></td> <td style="text-align: right;"><b>&gt; 0.05</b></td> </tr> </table> <p><i>Action: If the SPCCs are out of criteria, the laboratory should take corrective action and a new 5 point calibration should be generated.</i></p> <p><i>If sample analyses were performed, for any target analyte with an RF &lt; 0.05, qualify associated non-detects as "R" and flag associated positive data as "J."</i></p>	<b>N-Nitroso-di-n-propylamine</b>	<b>&gt; 0.05</b>	<b>Hexachlorocyclopentadiene</b>	<b>&gt; 0.05</b>	<b>2,4-Dinitrophenol</b>	<b>&gt; 0.05</b>	<b>4-Nitrophenol</b>	<b>&gt; 0.05</b>	
<b>N-Nitroso-di-n-propylamine</b>	<b>&gt; 0.05</b>								
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<b>4-Nitrophenol</b>	<b>&gt; 0.05</b>								

1.0 Semi-Volatile Data Review - Initial Calibration	
<p>1.6 A. Is the %RSD for individual analytes (except CCCs, see Table 3 and question 1.7 ) in the initial calibration below %15? B. If not, are alternate calibration equations used for those analytes not meeting the 15% criteria? C. Is the <u>average</u> %RSD for all analytes below 15%?</p> <p><b>Note: Reference Method 8000, Section 7.5.1.2. Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8270C requires 15% RSD as evidence of sufficient linearity to employ an average relative response factor (Avg. RRF).</b></p> <p><i>Action: If the %RSD for each analyte is below 15% RSD, no qualification is necessary.</i></p> <p><i>If one or two analytes had a low level calibration point removed to better the %RSD calculation, and the detection limit was not raised, qualify all undetected data as estimated. Positive data should not be qualified.</i></p> <p><i>If the average RSD for all calibration analytes is above 15% and average response factors are used, qualify all positive and undetected data for those individual analytes that exceeded the 15% RSD criteria requirement as estimated .</i></p> <p><i>Additional qualification, including rejection of data, may be warranted if other quality control criteria is not within requirements.</i></p>	<p>Additional Information: The laboratory has the option of using linear regression or quadratic calibration equations for individual analytes that don't meet the 15% criteria. Information concerning the acceptability for these alternate calibration equations must be supplied by the laboratory.</p> <p>The laboratory also has the option to re-analyze a particular calibration standard if it is suspected that the standard has degraded. In addition, the lab may remove either the low level or high level calibration point from the %RSD analysis for <u>up to two</u> individual analytes to bring the %RSD within criteria. If a low level standard is removed to improve the %RSD, the detection limit should be raised accordingly. If the high level standard is removed, the linear range is shortened for that analyte.</p> <p>If certain individual analytes have %RSDs above the criteria, but the average of all the analytes is below 15% RSD, then the average RRF is acceptable for use.</p> <p>Record the analytes in the initial calibration that used an alternate calibration curve or had %RSD values greater than 15%.</p>
<p>1.7 Do the Calibration Check Compounds (CCC) listed in Table 3 in the initial calibration standards meet a percent Relative Standard Deviation (%RSD) of 30% or less?</p> <p><i>Action: If any of the CCCs have a %RSD of greater than 30%, the system is considered too unstable for analysis. The system should undergo corrective action. If the analysis was not terminated, qualify all non-detected compounds as "R" and any associated positive results based upon the best professional judgement of the validator.</i></p>	

**Table 3  
Calibration Check Compounds (CCC) for SW-846, Method 8270C**

Base/Neutral Fraction Compounds	Acid Fraction Compound
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

<b>2.0 Semi-Volatile Data Review - Continuing Calibration Verification</b>	
<b>Continuing Calibration Verification</b>	
<p><b>A mid-range continuing calibration standard containing all calibration compounds and surrogates must be analyzed every 12 hours following analysis of the instrument performance check sample and before the analyses of method blanks and samples. This is termed the 12-hour standard. In addition, a continuing calibration verification (CCV) must be analyzed once per batch or every 20 samples, which ever is more frequent (some labs just refer to their 12-hour standard as a CCV). The continuing calibration standards must all have Relative Response Factors (RRFs) greater than or equal to 0.05 and %D between the initial calibration average RRF and the continuing calibration verification (CCV) RRF must be within +/- 25%. The Tier II Data Validator must verify that the continuing calibration was analyzed at the proper frequency and in the proper sequence. In addition, at least one target compound's continuing calibration RRF should be recalculated and compared to the reported result.</b></p>	
<p>2.1 Has a mid-point calibration standard (a.k.a. 12-hour standard or CCV) been analyzed for every 12 hours of sample analysis?</p> <p><i>Action: List all samples analyses that were not within 12 hours of the previous continuing calibration analysis. Call the laboratory for an explanation or for re-submission of the data. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	
<p>2.2 Continuing calibration RRFs for the semi-volatile target compounds in the 12-hour standard listed in question 1.4 must be greater than or equal to 0.01. The RRF for all other semi-volatile target compounds and surrogates must be greater than or equal to 0.05.</p> <p><i>Action: If any semi-volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" and 0.05 for all other semi-volatile compounds), use professional judgment for positive results, based on mass spectral identification, to qualify the data as "J" or "R."</i></p> <p><i>If any semi-volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" and 0.05 for all other volatile compounds), qualify non-detected compounds as "R."</i></p>	
<p>2.3 The Percent Difference (%D) between the average initial calibration RRFs and the 12-hour standard RRF must be within <math>\pm 50.0\%</math> for the semi-volatile target compounds and surrogates listed in question 1.4. The %D for all other semi-volatile target compounds and surrogates must be within <math>\pm 25.0\%</math>, except for 2,4-Dinitrotoluene, 2-Nitrophenol, and 2,4-Dimethylphenol, for which the %D must be within <math>\pm 30.0\%</math>.</p> <p><i>Action: If %D value for any of the semi-volatile target "poor performers" is outside the <math>\pm 50.0\%</math> criterion, qualify positive results with "J" and non-detected compounds "UJ."</i></p> <p><i>If %D value for 2,4-Dinitrotoluene, 2-Nitrophenol, and 2,4-Dimethylphenol is outside the <math>\pm 30.0\%</math> criterion, qualify positive results with "J" and non-detected compounds "UJ."</i></p> <p><i>If %D value for any other semi-volatile target compound is outside the <math>\pm 25.0\%</math> criterion, qualify positive results with "J," and non-detected compounds as "UJ."</i></p>	

<b>2.0 Semi-Volatile Data Review - Continuing Calibration Verification</b>	
<p>2.4 Do the RRFs of the SPCCs in the 12-hour standard (CCV) meet the initial SPCC criteria (0.05) for each 12-hour shift?</p> <p><b>Note: The 12-hour standard (CCV) RRFs are compared to the initial calibration.</b></p> <p><i>Action: If the RRFs of the SPCCs do not meet the criteria, corrective action should be taken to solve the source of the problem. If the source of the problem could not be determined, a new 5-point calibration should have been generated. List all sample analyses that were affected. Call the laboratory for explanation or re-submission. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	
<p>2.5 Do any CCCs (Table 3) have a percent Difference (%D) between the initial calibration RRFs and continuing calibration (12-hour standard) RRF that exceeds the +/- 30% criteria?</p> <p><i>Action: Corrective action should be taken to solve the source of the problem. If the source of the problem could not be determined, a new 5-point calibration should have been generated. List all sample analyses that were affected. Call the laboratory for an explanation or a re-submission. If continuing calibration data are not available, flag all associated sample data as "R."</i></p>	
<p>2.6 Were samples analyzed within 12 hours of either the Continuing Calibration Verification (12-hour standard) or the initial calibration?</p> <p><i>Action: If samples were not analyzed within the 12-hour window, all positive results should be regarded as "J." If other criteria is also outside of requirements, the Data Validator may qualify data as "R."</i></p>	
<p>2.7 The Tier II Data Validator should evaluate relative response factors (RRFs) for all the target compounds and surrogates in any batch specific continuing calibration verification. The RRFs in the CCV should be greater than 0.05 and the %D between the batch CCV, and the 12-hour standard should be less than 30%.</p> <p><b>Note: A 12-hour standard is needed once per day, and a continuing calibration verification is needed for each batch or every 20 samples. The NFGs allow four compounds to have less than 40% RRF while still considering the data acceptable.</b></p> <p><i>Action: If the %D is greater than or equal to 30% and the RRF is greater than 0.05, all positive results for these compounds should be qualified as "J" and all non-detected compounds qualified using the Data Validator's professional judgement.</i></p> <p><i>If the RRF is less than 0.05, qualify all positive results as "J" and all undetected compounds as "R."</i></p>	

**Table 4**  
**Internal Standards (IS) and Associated Target Compounds for SW-846, Method 8270C**

1,4-Dichlorobenzene-d <sub>4</sub> (IS)	Napthalene-d <sub>8</sub> (IS)	Acenaphthene-d <sub>10</sub> (IS)
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	2-Nitroaniline
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethyl Phthalate
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline
4-Methylphenol	4-Chloroaniline	Acenaphthene
N-Nitroso-Di-n-Propylamine	Hexachlorobutadiene	2,4-Dinitrophenol
1,2-Dichlorobenzene-d <sub>4</sub> (surr.)	4-Chloro-3-methylphenol	4-Nitrophenol
2-Fluorophenol (surr.)	2-Methylnaphthalene	Dibenzofuran
Phenol-d <sub>5</sub> (surr.)	Nitrobenzene-d <sub>5</sub>	2,4-Dinitrotoluene
2-Chlorobenzene-d <sub>4</sub> (surr.)		2,6-Dinitrotoluene
Hexachloroethane		Diethyl Phthalate
		4-Chlorophenyl-phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr.)
		2,4,6-Tribromophenol (surr.)
Phenanthrene-d <sub>10</sub> (IS)	Chrysene-d <sub>12</sub> (IS)	Perylene-d <sub>12</sub> (IS)
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl Phthalate
N-Nitrosodiphenylamine	Butylbenzyl Phthalate	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	bis(2-Ethylhexyl)phthalate	Benzo(k)fluoranthrene
Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
Pentachlorophenol	3,3'-Dichlorobenzidine	Ideno(1,2,3-cd)pyrene
Phenanthrene	Chrysene	Dibenz(a,h)anthracene
Carbazole	Terphenyl-d <sub>14</sub> (surr.)	Benzo(g,h,i)perylene
Anthracene		
Di-n-butyl Phthalate		
Fluoranthene		

<b>3.0 Semi-Volatile Data Review - Internal Standards</b>	
<b>Internal Standards</b>	
<p><b>Internal standards are the primary means of identifying and quantifying volatile organic compounds in samples. The evaluation of internal standard data is therefore critical to data validation. Internal standards are added to each sample and all QC samples. Internal standards are added to samples and blanks, and are evaluated in comparison to the initial calibration. Internal standard area counts must not vary by more than a factor of two (-50 to +/- 100%) from the associated calibration standard. In addition, the retention time of internal standards must not vary by more than +/- 30 seconds from the associated calibration standard. The internal standards and their associated target compounds are listed in Table 4.</b></p>	
<p>3.1 Is raw data available to determine if the internal standards are within criteria?</p> <p><i>Action: No action is necessary if the data is present. If it is not, the facility should provide the data from its laboratory. If the data is not available, the Tier II Data Validator may qualify data based upon professional judgement.</i></p>	<p>Note actions taken, such as when the facility was contacted, facility personnel and when the information was submitted:</p>
<p>3.2 Are the area counts for internal standards for the sample or blank outside of +50% of the area for the associated 12-hour standard (CCV)?</p> <p><i>Action: All positive results associated with an internal standard outside of the criteria should be qualified as "J."</i></p> <p><i>Non-detected compounds quantified using an internal standard whose area count is greater than 100% should not be qualified.</i></p> <p><i>Non-detected compounds quantified with an internal standard whose area count is less than 50% of the associated calibration standard should be qualified as, "UJ."</i></p> <p><i>If extremely low area counts are associated with the internal standards, qualify all associated target compounds as "R."</i></p>	
<p>3.3 Do any internal standards retention times vary by more than 30 seconds compared to the 12-hour calibration standard?</p> <p><i>Action: The Tier II Data Validator should request raw data chromatograms and assess whether false positives or negatives exist for any target compounds associated with that internal standard. Based upon this evaluation, the data may either be qualified as "J" or "R" using professional judgement.</i></p>	

<b>4.0 Semi-Volatile Data Review - Target Compound Identification</b>	
<b>Target Compound Identification</b>	
<p>One of the primary goals of sample analyses is to accurately identify the presence of target compounds. The procedure used by SW-846, Method 8270C to identify the presence of target compounds is to compare the retention time of unknown compounds in a sample to the retention time of known compounds in the calibration standard. The Relative Retention Time (RRT) of the compound in the sample must be within +/- 0.06 units of the RRT of the initial calibration standards. In addition, the mass spectra of the standard must match the mass spectra of the calibration standard within certain tolerances. For example, all ions in the standard mass spectra at a relative intensity of 10% must also be present in the sample spectrum. In addition, the relative intensities of the ions in the sample must agree within +/- 20% between the sample and the standard spectra. Ions that are present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum must be considered and accounted for. Results for unknown compounds in samples are compared to an electronic "library" of compounds to determine if all results (retention time and mass spectra) match within specified tolerances to a library compound (or at least to a certain type of compound of specific size).</p>	
<p>4.1 Are the RRTs of reported compounds in the ICV within +/- 0.06 RRT units (minutes) of the RRT for the initial calibration?</p> <p><i>Action: Use this information in conjunction with the mass spectral analysis information to qualify data. If target compound identification is in error, qualify that compound as "R."</i></p>	
<p>4.2 Check the sample compound spectra against spectral data of the standard. All ions in the standard mass spectra at a relative intensity of 10% must also be in the identified sample compound.</p> <p><i>Action: Use this information in conjunction with the mass spectral analysis information to qualify data. If target compound identification is in error, qualify that compound as "R."</i></p>	
<p>4.3 Check the sample compound spectra against spectral data of the standard. The relative intensity of the ions in the sample must agree within +/- 20% of the value obtained from the standard spectrum.</p> <p><i>Action: Use this information in conjunction with the mass spectral analysis information to qualify data. If target compound identification is in error, qualify that compound as "R."</i></p>	

**Tier II**

**Section 3**

**Tentatively Identified Compounds**

<b>1.0 Tentatively Identified Compounds</b>	
<p><b>When a library search is completed in which a unknown mass spectrum of a compound is compared to the mass spectra of compounds contained in a computer library in an effort to identify the compound, the compounds identified in this manner are referred to as tentatively identified compounds (TICs). Evaluation</b></p> <p><b>TICs are compounds detected in samples that are not target compounds, internal standards, or surrogate standards. These compounds may be of interest due to potential toxicity, or otherwise indicate a potential constituent of concern, or because they may act as interferants in the analysis. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification. Evaluations of TICs are not commonly included as part of a data submittal. An evaluation of TICs may arise when a ground water sample is analyzed for the "Appendix IX" list.</b></p>	
<p>1.1 Were chromatograms provided and reviewed?</p> <p><i>Action: If chromatograms were not provided, request data from the facility.</i></p>	
<p>1.2 Were the TICs qualitatively identified via an electronic library search? Which search engine was used: NIST/EPA/NIH/ Wiley Mass Spectral Library?</p> <p><i>Action: Identify whether an electronic library search was employed and what spectral library was searched.</i></p>	
<p>1.3 Did the laboratory report the TICs as target compounds by any other analytical method (e.g., with semi-volatile compound analysis) ?</p> <p><i>Action: If so, do not report the compound as a TIC.</i></p>	
<p>1.4 May any of the TICs be eliminated due to being a target analyte, a system monitoring compound, or an internal standard?</p> <p><i>Action: If yes, remove the compound as a TIC and report it as a target compound.</i></p>	
<p>1.5 If target compounds have been identified, did the review verify that quantitation had been made?</p>	
<b>2.0 TICs in Blanks</b>	
<p>2.1 Were blank chromatograms examined to verify that TIC peaks present in samples were not found in the blanks?</p> <p><i>Action: If TICs were found in the blank, evaluate whether blank contamination exists.</i></p>	
<p>2.2 Were all reported TICs sufficiently below 10x the level in the blank?</p> <p><i>Action: If not, report the compound as blank contamination and qualify the TIC as undetected.</i></p>	

<b>1.0 Tentatively Identified Compounds</b>	
<p>2.3 When a low-level, non-target compound that is a common artifact or laboratory contaminant is detected in a sample, was a thorough check of blank chromatograms completed looking for peaks which are less than 10% of the internal standard height, but present in the blank chromatogram at similar relative retention times?</p> <p><b>Note: The 10X rule applies to the following organic compounds: Methylene chloride, Acetone, Toluene, 2-Butanone, Cyclohexane, Phthalate Esters.</b></p> <p><i>Action: If a compound was not found in any blanks, but is a suspected artifact or a common laboratory contaminant, were the results qualified as "UJ?" If a TIC was found in a blank and also in a sample, use professional judgement to determine whether the TIC is due to laboratory contamination.</i></p>	
<p>2.4 Were the blanks checked for:</p> <p>1) Common lab contaminants including: CO<sub>2</sub>, Siloxanes, Diethyl ether, Hexane, certain Freons, Phthalates at levels less than 100 ug/L or 4000 ug/kg?</p> <p>2) Solvent preservatives such as Cyclohexene (which is a Methylene chloride preservative) and its related byproducts, including: Cyclohexanone, Cyclohexenone, Cyclohexanol, Cyclohexenol, Chlorocyclohexene, and Chlorocyclohexanol?</p> <p>3) Aldol condensation reaction products of acetone including: 4-Hydroxy-4-methyl-2-pentanone, 4-Methyl-2-penten-2-one, and 5,5-Dimethyl-2(5H) furanone?</p> <p><i>Action: if these compounds were detected in the blank, and the sample, qualify those TICs as undetected according to the 10X rule.</i></p>	
<b>Actions/Reporting</b>	
<p>3.1 If there is less than 100% confirmation of positive results, did the reports indicate which samples have been confirmed, and the percentage of positives confirmed?</p>	
<p>3.2 Were all TICs qualified as "NJ" with approximated concentrations?</p>	
<p>3.3 If it was determined that a tentative identification of a non-target compound is not acceptable, was the tentative identification changed to "unknown" or an appropriate identification?</p>	

**Tier II**  
**Section 4**  
**Inorganic Data Review**

<b>1.0 ICP Metals Data Review - Initial Calibration</b>	
<b>Initial Calibration</b>	
<p>The review of initial calibration data for inorganic elements is complicated because of the lack of a prescribed procedure. SW-846 6010B requires that the instrument be calibrated according to the instrument manufacturer's recommendations. Several manufacturers only require that a blank and one calibration standard be used for calibration, whereas others require up to five standards be used to establish the calibration. Whatever method is used, the laboratory should be able to demonstrate adequate instrument response and linear range. The laboratory should be consulted as to standard operating procedures are required to calibrate the instrument.</p>	
<p>1.1 Was a minimum of one standard and a blank used for the initial calibration of the ICP instrument?</p> <p><b>Note: SW-846, Method 6010B does not describe a sole procedure for instrument calibration. The calibration procedure will vary by the manufacturer's recommended instrument calibration procedure. It may be necessary to consult the laboratory's QAP or contact the laboratory for the exact calibration procedure that is used.</b></p> <p><i>Action: If the minimum number of standards were not used, qualify all results as "R."</i></p>	
<p>1.2 Was an Initial Calibration Verification (ICV), a Calibration Blank, and a Continuing Calibration Verification (CCV) analyzed immediately after the initial or daily calibration? A CCV must also be analyzed after every tenth sample or every two hours, whichever is more frequent.</p> <p><b>Note: This sequence must be followed and is found in Section 7.4 of SW-846, Method 6010B. The ICV and CCV must be from a certified standard other than that used for the initial calibration, and must be within the calibrated range.</b></p> <p><i>Action: The ICV and CCV must be within +/- 10% percent (%R = found/true X100) of the expected value or the analysis should have been halted and the instrument re-calibrated. If the analysis was not halted, qualify all results as "R."</i></p>	
<b>2.0 ICP Metals Data Review - Interference Check Samples (ICS)</b>	
<b>Interference Check Samples (ICS)</b>	
<p>The ICS consists of two standard solutions, an A or AB solution (solution A consists of the interferents, and solution AB consists of the analytes mixed with the interferents). The ICS solutions are used to determine if non-target analytes (e.g., interfering elements) are elevating or depressing results for target metals. The ICS results can be used to demonstrate that correction for interfering elements is not necessary at the concentrations tested or to develop Inter-element Correction Factors (IECFs), which can be applied to sample results to account for the presence of these interfering metals.</p>	
<p>2.1 Was an Interference Check Sample (ICS) analyzed at the beginning and end of each analytical run or at a minimum of two every eight hours?</p> <p><b>Note: The ICS is NOT to be analyzed before the initial calibration verification standard.</b></p> <p><i>Action: Check the run log to verify that the ICS was run in the proper order and frequency. If discrepancies exist, consult the lab for clarification and re-submission of the data pack. If no new information is forthcoming, qualify all positive results using best professional judgement.</i></p>	

<b>2.0 ICP Metals Data Review - Interference Check Samples (ICS)</b>	
<p>2.2 Are the results of the AB standard within control limits of 80% - 120%?</p> <p><b>Note: The Tier II Data Validator should verify one of the percent recovery (%R) calculations (<math>\%R = \text{found/true} \times 100</math>).</b></p> <p><i>Action: If the results fall outside of the acceptance criteria, the Tier II Data Validator should use professional judgement to qualify positive or non-detected data as either estimated or rejected. The number of analytes outside of the criteria, the magnitude of the deviations and other relevant information should be used for this judgement.</i></p>	
<p>2.3 Are concentrations of interfering metals (e.g., Al, Ca, Fe, and Mg) in samples comparable or greater than concentrations in the ICS?</p> <p><b>Note: The concentration of these elements may not be reported. If not, additional information may be requested from the laboratory.</b></p> <p><i>Action: If the ICS recovery for an element is greater than 120%, and the sample results are less than the detection limit, the data is acceptable and shouldn't be qualified.</i></p> <p><i>If the ICS recovery for an element is greater than 120%, and the results are greater than the detection limit, qualify all the affected data as "J."</i></p> <p><i>If the ICS recovery for an element falls between 50% and 79% and the sample results are greater than the detection limit, qualify the affected data as "J." If the results are less than the detection limit, qualify these results as "UJ."</i></p> <p><i>If the ICS recovery for an element is less than 50%, qualify the affected data as "R."</i></p>	
<p>2.4 Are results greater than the detection limit for elements which aren't in the ICS solution? If yes, the possibility of a false positive exists.</p> <p><i>Action: For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results greater than the detection limit as "J."</i></p>	
<p>2.5 Are negative results observed for elements that are not present in the ICS solution, and is their absolute value greater than the detection limit? If so, the possibility of false negatives in the samples may exist.</p> <p><i>Action: For samples with comparable or higher levels of interferents, qualify results for the affected analytes less than the detection limit as "UJ."</i></p>	

2.0 ICP Metals Data Review - Interference Check Samples (ICS)	
2.6	<p>Are concentrations of Al, Ca, Fe and Mg in the samples greater than in the ICS, or are other elements present in the sample at greater than 10 ppm?</p> <p><i>Action: Estimate the concentration produced by an interfering element. If the estimate is greater than 2X the element's detection limit and greater than 10% of the reported concentration of the affected element, qualify the affected results as "J."</i></p>

3.0 Cold Vapor AA Method for Mercury Data Review	
<p><b>Calibration and Instrument Runs</b></p> <p><b>SW-846, Method 7470 is used for liquid samples including the TCLP extract. Method 7471A is used for solid samples and for sludges. The methods are subject to significant interferences from chlorides, and a pre-treatment step using potassium permanganate is required to remove these interferences.</b></p>	
3.1	<p>Was potassium permanganate solution added to eliminate interferences (typical interferences are from sulfide, copper, chlorides, and certain VOCs that absorb the 253.7 nm wave length absorbed by mercury vapor)?</p> <p><b>Note: Method 7471A states that up to 25mL of 5% permanganate solution can be used. The amount of added permanganate should be recorded. The same amount of permanganate solution should be added to blanks and calibration standards.</b></p> <p><i>Action: If permanganate solution was not added, qualify all positive results as "J" and all non-detected results as "UJ."</i></p>
3.2	<p>Was a working standard (Continuing Calibration Standard) of 0.1 µg/mL concentration prepared each day?</p> <p><i>Action: If a working standard was not prepared daily, qualify all positive results as "J" and all undetected results as "UJ."</i></p>
3.3	<p>Was the instrument calibrated daily (or each time the instrument was set up) per manufacturer's instructions, and at SOP specified levels?</p> <p><b>Note: Daily calibration with standards of varying concentration defines the reliable "working range" of the analysis.</b></p> <p><b>Note: The Contract Lab Program requires from five to eight calibration standards and a blank for calibration (typically, 0.0002 ppm, 0.0005 ppm, 0.001 ppm, 0.005 ppm and 0.010 ppm). SW-846, Method 7471A requires three calibration standards and a blank be used. The correlation coefficient for calibration must be <math>\geq 0.995</math> to demonstrate linearity.</b></p> <p><i>Action: If the specified number of calibration standards were not used or if the instrument was not calibrated daily, qualify all data as "R."</i></p>
3.4	<p>Was at least 1 blank analyzed before the calibration standards used to establish the curve?</p> <p><i>Action: If a blank was not analyzed qualify, all associated data as "J."</i></p>

<b>3.0 Cold Vapor AA Method for Mercury Data Review</b>	
<p>3.5 Did the calibration curve for mercury possess a correlation coefficient of greater than or equal to 0.995?</p> <p><i>Action: If the correlation coefficient is less than 0.995, qualify all positive data as "J," and all non-detected compounds as "UJ." If the deviation is significant, data may be qualified as "R" based upon professional judgement.</i></p>	
<p>3.6 Were the Initial Calibration Verification (ICV) and the Continuing Calibration Verification (CCV) analyzed with the appropriate frequency?</p> <p><b>Note: An Initial Calibration Verification (ICV) standard and a blank must be analyzed after the initial calibration. This ICV concentration should be near the mid-point of the working range. The CCV standard is analyzed at a minimum frequency of every 10 samples (10%) or every two hours. The CCV must be analyzed at the beginning and end of an analytical.</b></p>	
<p>3.7 Were the ICV and CCV within control limits (R) of 80% to 120%?</p> <p><b>Note: Typically, ICV = 0.0025 ppm and CCV= 0.005 ppm.</b></p> <p><i>Action: If the ICV or CCV %R is 65-79% or 121-135%, qualify all positive results greater than the detection limit as "J."</i></p> <p><i>If the ICV or CCV %R are between 121-135%, results below the detection limit are acceptable.</i></p> <p><i>If the ICV or CCV %R is 65-79%, results below the detection limit should be qualified as "UJ."</i></p> <p><i>If the ICV or CCV %R is less than 65%, qualify all positive results as "R."</i></p> <p><i>If the ICV or CCV %R is greater than 135%, qualify all the results greater than the detection limit as "R," and results less than the detection limit as acceptable.</i></p>	
<p>3.8 Was the CCV standard analyzed at the beginning of the run and also after the last analytical sample? A CCV must also be analyzed after every tenth sample or every two hours, whichever is more frequent.</p> <p><i>Action: Note any discrepancies, and use professional judgement along with other QA/QC information to qualify data.</i></p>	
<p>3.9 Was the same CCV standard solution used throughout the analytical runs for a sample delivery group of samples received?</p> <p><i>Action: If not, note any discrepancies, and use professional judgement along with other QA/QC information to qualify data.</i></p>	
<p>3.10 Was a blank analyzed after the ICV (also called the initial calibration blank ICB), and the CCV (also called the continuing calibration blank, CCB)?</p> <p><b>Note: This blank confirms that carry over is not biasing the next sample.</b></p> <p><i>Action: If no blank was analyzed, qualify all positive data in the next sample as "J." All non-detected data is acceptable.</i></p>	

<b>3.0 Cold Vapor AA Method for Mercury Data Review</b>	
<p>3.11 Was a CCB analyzed immediately after every initial and continuing calibration verification? Was the CCB analyzed at 10% or every two hours, whichever is more frequent during the run?</p> <p><i>Action: If a CCB was not analyzed with the correct frequency, note any discrepancies. If no blank was analyzed, qualify all positive data in the next sample batch as "J." All non-detected data is acceptable. If results of the blank were above the detection limit, use the Tier I Data Validation Manual for qualification.</i></p>	
<p>3.12 Was the CCB analyzed after the last CCV and after the last analytical sample of the run?</p> <p><b>Note: Results for the CCB must not exceed the Reporting Limit for mercury.</b></p> <p><i>Action: If high blank results are reported, note any discrepancies. If no blank was analyzed, qualify all positive data in the next sample batch as "J." All non-detected data is acceptable. If results of the blank were above the detection limit, use the Tier I Data Validation Manual for qualification.</i></p>	
<p>3.13 <u>Optional question for Contract Lab Program (CLP) data</u></p> <p>Was a low-level solution (CRA) prepared at the Reporting Detection Limit (RDL) or at the Instrument Detection Limit (IDL), whichever is greater, and was the CRA analyzed at the beginning of each sample analysis run, but not before the Initial Calibration Verification (CLP only)?</p> <p><i>Action: Note any discrepancies, and use professional judgement along with other QA/QC information to qualify data.</i></p>	
<p>3.14 <u>Optional question for CLP data</u></p> <p>Analysis of the CRA standard for Mercury is required for both the manual and cold vapor methods. Were the results and percent recovered reported?</p> <p><b>Note: There are no specific acceptance criteria established by EPA for Mercury at this time.</b></p> <p><i>Action: Note any discrepancies, and use professional judgement along with other QA/QC information to qualify data.</i></p>	
<p>3.15 If the lab failed to supply adequate calibration information, was the lab contacted and the necessary information requested?</p>	
<p>3.16 If the lab was contacted and the information was not available, did the Tier II Data Validator use professional judgement to assess the data?</p>	
<p>3.17 Were samples with concentrations greater than the highest calibration standard diluted and re-analyzed?</p> <p><i>Action: Qualify all positive results as "J." All non-detected data are acceptable.</i></p>	
<p>3.18 Does the lab data include the dilution factor used?</p> <p><i>Action: If not present, request the information from the facility.</i></p>	