Response to Public Comments regarding the drafts of the Updated Reasonable Confidence Protocols, posted April 2023

The Department of Energy and Environmental Protection (DEEP) would like to extend its appreciation to the parties who provided comments to the latest updates to the Reasonable Confidence Protocols. Written public comments received during the public comment period and the DEEP's responses are summarized below.

General Comments

Comment: Requesting confirmation that Estimated Values ("E") should no longer be reported. Report only the correct value (the E statement was removed from several RCPs). This is NOT on the 8270 list and should be added.

Response:
DEEP is confirming that estimated, "E", values will no longer be accepted. Refer to the updated Table 1A language: *Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported.* If there is not enough sample volume to run a dilution the data user should consider resampling for reanalysis to obtain an accurate concentration.

No revisions will be made in response to this comment.

RCP Language: *"Retain reported and unreported analytical data and information for a period of 10 years."*

Comment: 10 years is too long for a lab to be responsible for data. Please consider making the record retention period no more than 7 years.

Response:
DEEP acknowledges the resources in the form of physical and/or digital space may be a limited commodity to laboratories. DEEP's intent was to account for record retention time periods that vary based on accreditation policy. To allow for flexibility, the language regarding record retention will be adjusted, see below. This language will allow laboratories flexibility in record retention policies dependent upon the policy per accreditation. For example, National Environmental Laboratories Accreditation Conference (NELAC) accreditation requires a minimum of 5 years while American Industrial Hygiene Association (AIHA) accreditation requires a minimum of 10 years, record retention by the laboratory should be consistent with their accreditation requirements.

DEEP is making the following revision to all RCPs:

Retain reported and unreported analytical data and information for a minimum of 5 years or as required under applicable accreditation policies.

RCP Language: *"These data must meet or fall within the performance standards as presented in Section…and Table 1A of this RCP."*

Comment: Preference for the previous language that the data should be meet or exceed performance standards.

Response:
Using the language "meet or fall within" was intended to clarify that recoveries are only acceptable if they fall within the specific recovery ranges. Exceedances of defined acceptance criteria would result in the need for a narration in the lab report narrative. For example, if the acceptance range is defined as 70-130% recovery and a QC sample recovers at 140% which exceeds the range the criteria hasn't been met and must be narrated. Therefore, it is more accurate to state recovery should fall within the range.

No revisions will be made in response to this comment.

RCP Language: All

Comment: Clerical errors such as typographical errors, grammatical errors, and/or minor citation errors.

Response:
All clerical errors noted by submitted comments were addressed in all identified RCPs.

RCP Language: *Blank concentrations must be ≤ ½ Reporting Limit/Lower Limit of Quantitation (RL/LLOQ).*

Comment: Suggest using NELAC blank "pass/fail" criteria in place of ≤ ½ RL/LLOQ.

Response:
Language from Chapter One, page 17, and EPA Method 6020B Section 9.7.1 states "*Blanks are generally considered to be acceptable if target analyte concentrations are less than ½ the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are ≥10X the blank). Other criteria may be used depending on the needs of the project. For method specific details see methods 6010, 6020 for inorganics and Method 8000 for organics).*" It was DEEP's intentions to agree with EPA methodology, however, after further review and receiving additional feedback, DEEP acknowledges achieving ≤½ RL/LLOQ may present a challenge for a number of analytes. Therefore, DEEP will revise the RCPs to the original blank acceptance criteria of <RL/LLOQ.

DEEP is making the following revision to all RCPs:

Blank acceptance criteria <RL/LLOQ.

RCP Language: *Recommendation for use of Standard Reference Materials (SRM) as additional QC sample for solid matrices.*

Comment: Suggest removing recommendation for use of SRM.

Response:
Note the use of a Standard Reference Material (SRM) as presented in RCPs 6010, 6020, 7000, 7196, and 7470/7471, is a recommendation and not a requirement, therefore data users may choose to request a SRM or not. DEEP highly recommends the use of SRMs to demonstrate the accuracy and effective leaching of various metal elements from complex soil matrices during the sample preparation (i.e., digestion) process. This additional evidence of analytical accuracy is particularly useful when used for risk-based cleanup goals.

No revisions will be made in response to this comment.

RCP Language: *"It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet Remediation Standard Regulation ("RSR") criteria."*

Comment: The current language requires RLs/LLOQs to meet RSR criteria which may not be as conservative as what may be achievable by laboratories and may not be adequate enough to meet the cleanup objectives of the site (e.g., background). Suggest revising language to "…meet the project Data Quality Objectives ("DQOs")."

Response:
DEEP acknowledges the suggested language provides clearer expectations for RL/LLOQs for all cleanup activities and is more protective of human health and the environment.

DEEP is making the following revision to all RCPs:

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet project Data Quality Objectives ("DQOs").

RCP Language: *"Typical Reporting Limits/Lower Limits of Quantitation" Tables in all RCPs*

Comment: Do laboratories have to follow the RLs listed in these tables?

Response:
DEEP understands RL/LLOQs may vary between laboratories based on instrumentation and laboratory conditions. The RL/LLOQs provided in the "Typical Report Limits/Lower Limits of Quantitation" are meant to serve as guidance for data users to understand which analytical methods can, or can't, achieve the Data Quality Objectives (DQOs) necessary to meet their project-specific goals. These tables are not intended to be used to dictate what RLs/LLOQs laboratories must report.

DEEP is making the following revision to all RCPs:

Add footnote to “Typical Reporting Limits/Limits of Quantitation” Tables: [1]Note these values are intended to serve as guidance to EPs when planning analytical needs to achieve the data quality objectives to meet project-specific goals. These tables are not intended to dictate what RL/LLOQs laboratories must report.

Comment: The RCPs should specify the current promulgated method because labs must adjust their practices to meet the latest promulgated EPA methods. If the RCPs don’t specifically state it, labs may falsely believe that they can report data under any version of an EPA method.

Response:
Each of the RCPs include the following language to address reference to promulgated methods:

"All method references are to the latest promulgated version of the method found in *Test Methods for Evaluating Solid Waste, SW-846."*

The workgroup chose to include this language to allow flexibility for the RCPs to reference the latest EPA updates to all applicable Methods without the need for frequent revisions of the RCPs.

No revisions will be made in response to this comment.

Specific Comments

RCP 8081 - Pesticides

Section Reference: 1.2 Summary of SW-846 Method 8081

RCP Language: *"Preliminary identification of target analytes is accomplished by comparing the retention time of the chromatographic peaks of the sample to known pesticides analyzed under the exact same conditions. Confirmation is accomplished either by analysis of the same extract on a dissimilar column, again comparing the retention times of the chromatographic peaks of the sample to known pesticides analyzed under the exact same conditions, or by using at least one other independent qualitative technique such as GC/MS."*

Comment: The RCP mentions gas chromatography/mass spectrometry (GC/MS), but Method 8081 uses Electron Capture Detection (ECD) instead of MS. Why is GC/MS referenced in the RCP?

Response:
The use of GC/MS is allowed by the EPA Method 8081. EPA Method 8081, Section 1.6, states *"Compound identification based on single-column analysis should be confirmed on a second column or should be supported by at least one other qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm the measurements made with the primary column. GC/MS (e.g., Method 8270) is also recommended as a confirmation technique, if sensitivity permits (also see Sec. 11.7 of this method). GC/AED may also be used as a confirmation technique if sensitivity permits (see Method 8085)."*

The RCP language referenced above mentions the use of GC/MS as a possible means of a qualitative technique to confirm a detection as is mentioned in the above referenced section of EPA Method 8081.

No revisions will be made in response to this comment.

Section Reference: 1.3.2 Cross-Contamination/Carry Over

RCP Language: *"Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of pesticides or other compounds which cause a detector response, such as polychlorinated biphenyls ("PCBs"). Syringes on the autosampler may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of solvent blanks. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run (ghost peaks)."*

Comment: Typically, a solvent blank (SB) is not run every other sample etc. Per the method, it does not state that the sample is clean based on Solvent Blank. It should read that the instrument is free of contamination. This would be able to be seen based on a sample that has run with a "ND".

Response:
The language referenced by the comment mentions the use of solvent blanks to demonstrate the system to be clean. However, DEEP acknowledges the use of autosampler instrumentation has changed how laboratories demonstrate they have minimized carry-over contamination between high concentration and low concentration samples. Specifically, it is not often possible to anticipate which samples have high concentrations and which samples have low concentrations when arranging the samples on the autosampler, therefore it is not necessarily possible for the analyst to know when to program the instrument to analyze solvent blanks to demonstrate the instrument is clean. The referenced RCP language warrants clarification.

DEEP is making the following revision to RCP 8081:

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of chlorinated pesticides or other compounds which cause a detector response, such as polychlorinated biphenyls (“PCBs”). Concentrations of chlorinated pesticides which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. Low-level samples that immediately follow high-level samples need to be inspected for possible carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample analysis.

Section Reference: 1.4.1 Reporting Limits/Lower Limits of Quantitation for SW-846 Method 8081

RCP Language: *“To meet the detection limits, it may be necessary to modify the analytical method by using increased sample volume or mass. In such cases the modifications must be noted in the laboratory report narrative.”*

Comment: Disagree that this should be reported in the narrative. Modifications, such as sample volume/mass adjustments can be found through an audit of a bench sheet.

Response:
The purpose of narrating in the lab report is to document any, and all, modifications of the method that would affect the initial determination of the instrument RL/LLOQ which must be reported to DEEP per the RCPs. It is important that data users and regulators understand why the RL/LLOQ may have deviated from the project Data Quality Objectives and clean-up goals. Data users may need to justify why modification of the method was necessary so they can explain discrepancies with the regulations to regulators. Failure to provide such justification may result in rejection of the data.

No revisions will be made in response to this comment.

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: No specific reference made.

Comment: *Per Method 8000 (9.4.1); When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. In the case of very contaminated samples or when the lab does not receive enough samples to perform a single matrix spike, an LCS and LCS duplicate (LCSD) may be performed to document precision and bias. An LCS should be included with each preparation batch. The LCS is an aliquot of the same clean (control) matrix used for the method blank(s) and of a similar weight or volume as the method blank and field samples. The LCS is spiked with similar analytes at the same concentrations as in the matrix spike and is processed identically to the samples.* The laboratory should only be responsible for running an LCSD when this is the case. Not for every batch.

Response:
It has become common practice in most laboratories to include an LCS/LCSD in each batch. It is DEEP's intent to maintain consistency in data quality between all laboratories used for characterization, investigation, and remediation purposes. The use of an LCS/LCSD for the batch versus only using an MS/MSD provides all data users whose samples are submitted in the same batch with details on analytical precision. Whereas, if only MS/MSD is used to report the analytical precision, that data is only applicable to the data user whose sample was used for the MS/MSD. Therefore, it is the use of an LCS/LCSD will provide data users with more complete QC for the purposes of their data validation and intended use of the data.

No revisions will be made in response to this comment.

Section Reference: Table 1A - DDT deliverable.

RCP Language: *"Required Deliverable: Yes"*

Comment: Why is Breakdown a required deliverable? If you do not pass the Breakdown you cannot proceed with analysis. It would be similar to requiring an 8260 analysis to show a passing Tune as deliverable. This will confuse EP. They will want to know what the bias is based on the recovery. Please consider removing this as a required deliverable.

Response:
Endrin/DDT breakdown has been a required deliverable since the inception of the 2006 RCPs and will continue to be required.

No revisions will be made in response to this comment.

Section Reference: Table 1A – Identification and Qualification, Required Performance Standard, Item 3

RCP Language: *"(3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The lab must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on secondary column as well."*

Comment: Commentor expressed concern with the approach to report the higher of the two results. When response from both columns is appropriately calibrated and QC passes criteria,

any difference between the columns in a field sample result may be due to unknown compounds at the same retention time, whether or not chromatographic interference is visible on the chromatogram.

Response:
After thorough discussion, the workgroup and regulators agreed it is more protective of human health and the environment to report the higher of the two values for environmental cleanup purposes.

No revisions will be made in response to this comment.

RCP 8082 - PCBs

Section Reference: Section 1.2.1 Sample Extraction and Cleanup

RCP Language: *"It is highly recommended that extracts for PCB analysis be routinely subjected to a sulfuric acid cleanup using SW-846 Method 3665. This cleanup technique will remove (i.e., destroy) most other organic compounds including many single component organochlorine or organophosphorus pesticides as well as phthalate compounds, which could potentially interfere with the quantitation of PCB Aroclors or congeners. Other optional extraction clean-up methods are included in Table 2.0."*

Comment: Recommend adding SW-846 Method 3665 to Table 2.0 along with a description of the method. It is mentioned in the previous paragraph but should be listed in the Table as an optional cleanup.

Response:
The RCP language states that method SW-846 Method 3665 is the "highly recommended" cleanup method, whereas the methods provided in Table 2.0 are "optional extraction cleanup methods". To provide more clarification, DEEP will revise the Table 2.0 title.

DEEP is making the following revision to RCP 8082:

Table 2.0: Optional Extraction Cleanup Methods

Section Reference: Section 1.3.2 Cross-contamination / Carryover

RCP Language: *"Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of PCBs or other compounds which cause a detector response, such as phthalates. Syringes on the autosampler may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of solvent blanks. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run (i.e., ghost peaks)."*

Comment: Typically, a solvent blank (SB) is not run every other sample etc. Per the method it does not state that the sample is clean based on Solvent Blank. It should read that the instrument is free of contamination. This would be able to be seen based on a sample that has run with a ""ND""."

Response:
The language referenced by the comment mentions the use of solvent blanks to demonstrate the system to be clean. However, DEEP acknowledges the use of autosampler instrumentation has changed how laboratories demonstrate they have minimized carry-over contamination between high concentration and low concentration samples. Specifically, it is not often possible to anticipate which samples have high concentrations and which samples have low concentrations when arranging the samples on the autosampler, therefore it is not necessarily possible for the analyst to know when to program the instrument to analyze solvent blanks to demonstrate the instrument is clean. The referenced RCP language warrants clarification.

DEEP is making the following revision to RCP 8082:

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of PCBs or other compounds which cause a detector response, such as phthalates. Syringes on the autosampler may also become contaminated in the same manner. Concentrations of chlorinated pesticides which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. Low-level samples that immediately follow high-level samples need to be inspected for possible carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample analysis.

Section Reference: Section 1.3.3 Sulfur Interferences

RCP Language: "*The presence of elemental sulfur (S) will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples and can be removed through the use of SW-846 Method 3660."*

Comment: This should read to reflect early eluting PCB aroclors. Still important to state that PCB will be affected though.

Response:
DEEP notes the comment is reference language that is original to the inception of the RCPs and was not updated in this update cycle. However, it is acknowledged that clarification is warranted.

DEEP is making the following revision to RCP 8082:

The presence of elemental sulfur (S) will result in broad peaks that interfere with the detection of early-eluting PCB Aroclors. This cleanup technique will remove (destroy) most other organic compounds including many single component organochlorine or organophosphorus pesticides as well as phthalate contaminants which could potentially interfere with the quantitation of PCB Aroclors or congeners.

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: No specific reference made.

Comment: *Per Method 8000 (9.4.1); When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. In the case of very contaminated samples or when the lab does not receive enough samples to perform a single matrix spike, an LCS and LCS duplicate (LCSD) may be performed to document precision and bias. An LCS should be included with each preparation batch. The LCS is an aliquot of the same clean (control) matrix used for the method blank(s) and of a similar weight or volume as the method blank and field samples. The LCS is spiked with similar analytes at the same concentrations as in the matrix spike and is processed identically to the samples.* The laboratory should only be responsible for running an LCSD when this is the case. Not for every batch.

Response:
It has become common practice in most laboratories to include an LCS/LCSD in each batch. It is DEEP's intent to maintain consistency in data quality between all laboratories used for characterization, investigation, and remediation purposes. The use of an LCS/LCSD for the batch versus only using an MS/MSD provides all data users whose samples are submitted in the same batch with details on analytical precision. Whereas, if only MS/MSD is used to report the analytical precision, that data is only applicable to the data user whose sample was used for the MS/MSD. Therefore, it is the use of an LCS/LCSD will provide data users with more complete QC for the purposes of their data validation and intended use of the data.

No revisions will be made in response to this comment.

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: *"Batch MS/MSD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged."*

Comment: I believe most laboratories do not include MS/MSD data for samples that are not the MS/MSD from the site.

Response:
This language is not intended to direct laboratories on how to choose MS/MSDs, but rather to emphasize to EP's that requesting "batch MS/MSD" results will not necessarily provide them appropriate information specific to the conditions of their site (i.e., sample heterogeneity, matrix interference, etc.). Therefore, EPs are generally discouraged from using that data as part of any data package presented to DEEP. Rather the language in this section was intended to provide EPs information on the benefits of collecting site-specific MS/MSD samples to better understand potential matrix interferences that may be present at their site and could impact how they characterize a release(s).

No revisions will be made in response to this comment.

Section Reference: Table 1A – Surrogates, Required Performance Standard, Item 1

RCP Language: *"(1) … Recommended surrogates: PCB Aroclor Analysis: TCMX and DCB.*

PCB Congener Analysis: TCMX or DBOFB and BZ198."

Comment: The request is to use DCB and TCMX as the surrogates, but also to have DCB as the Internal Standard. Cannot apply the ISTD/Surrogate as same. One is pre-extraction one is post extraction.

Response:
Use of internal standards is optional. Note in Table 1A and report deliverables "if employed"/"if used" are included in notes to indicate they are not required. Per method 8082, alternative compounds can be used for internal standards or surrogates as needed for analysis, DEEP would expect demonstration of QA/QC to support the use of those alternative compounds.

DEEP is making the following revision to RCP 8082:

For clarification, DEEP will add "if employed" to Table 4.0: IDOC requirements to maintain consistency with language in Table 1A.

Section Reference: Table 1A – Identification and Qualification, Required Performance Standard, Item 4

RCP Language: *"(4) Secondary column analysis: lab must utilize a second dissimilar column to confirm positive results above the RL/LLOQ. The lab must report the higher of the two results. All required QA/QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well."*

Comment: Commentor expressed concern with the approach to report the higher of the two results. When response from both columns is appropriately calibrated and QC passes criteria, any difference between the columns in a field sample result may be due to unknown compounds at the same retention time, whether or not chromatographic interference is visible on the chromatogram.

Response:
After thorough discussion, the workgroup and regulators agreed it is more protective to human health and then environment to report the higher of the two values for environmental cleanup purposes.

No revisions will be made in response to this comment.

RCP 8151 - Herbicides

Section Reference: Table 1A – Identification and Qualification, Required Performance Standard, Item 3

RCP Language: *"(3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The lab must report the higher of the two results unless obvious interference, in which case, report lower result. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well."*

Comment: Commentor expressed concern with the approach to report the higher of the two results. When response from both columns is appropriately calibrated and QC passes criteria, any difference between the columns in a field sample result may be due to unknown compounds at the same retention time, whether or not chromatographic interference is visible on the chromatogram.

Response:
After thorough discussion, the workgroup and regulators agreed it is more protective to human health and then environment to report the higher of the two values for environmental cleanup purposes.

No revisions will be made in response to this comment.

Section Reference: Table 1A – Continuing Calibration Verification, Column 3, Item 1

RCP Language: (1) Prior to samples, every 12 hours or every 10 samples, whichever is more frequent, and at the end of the analytical sequence.

Comment: Where is the 10-sample suggestion originally referenced? It is not included in Method 8151. Section 11.7.4 of 8000D says "Therefore, more frequent verification of calibration (i.e., after every 10 samples) may be necessary for some types of detectors (i.e., electron capture, electrochemical conductivity, photoionization, fluorescence detectors). This is ONLY a suggestion. Suggest referencing every 10-samples as a suggestion in the RCP.

Response:
The referenced language is original to the inception of the RCPs, is general practice in laboratories who have provided guidance to DEEP and ensures consistent evaluation of instrument performance throughout an analytical sequence. DEEP has the authority to maintain conservative approaches to ensure data quality objectives for intended uses of data that are tantamount to protecting human health and the environment.

No revisions will be made in response to this comment.

Section Reference: Table 1A – Continuing Calibration Verification, Column 3, Item 4

RCP Language: *“(4) %D must be ≤15% for each target analyte.”*

Comment: Where was the ≤15% drift (%D) requirement derived? Other methods reference %D of 20 percent. EPA 8000D 11.7 "If the % Difference (when using average RF calibration) or % Drift (for all other types of calibration) of an analyte is within ±20% of the expected concentration or amount based on the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the calibration curve to quantitate sample results. The ±20% criterion may be superseded in certain determinative methods."

Response:
The referenced RCP language is original to the inception of RCP 8151. DEEP has the authority to maintain conservative approaches to ensure data quality objectives for intended uses of data that are tantamount to protecting human health and the environment.

No revisions will be made in response to this comment.

Section Reference: 1.6 Analyte List for SW-846 Method 8151; Table 1B

RCP Language: *Dinoseb**

Comment: Can DEEP provide clarification as to the reason for the asterisk next to Dinoseb in Table 1B?

Response:
The asterisk in Table 1B was used to identify Dinoseb as an analyte that is only applicable to aqueous samples. DEEP will add a footnote to the table for clarification.

DEEP is making the following revision to 8151:

Dinoseb is only a target analyte for aqueous samples.

Section Reference: 1.8 Sample Containers, Preservation, and Holding Times; Table 5.0

RCP Language: *Holding time: 24 hours from esterification to analysis*

Comment: Request clarification on original of the “24 hours” timeframe and concerned about wording. There are times where a sample has to be re-analyzed outside of 24 hours from esterification. This wording would force the lab to re-extract the sample. For example, if there is a bad injection on a Friday and the same has to be reanalyzed after running over the weekend. Reanalyzing the LCS with the samples in question would show that nothing was lost after esterification process.

Response:
It is understood that the holding time of 24 hours for methylated extracts may result in the need for re-extraction, the concern of trans-esterification dictates the need for analyzing the methylated extracts as quickly as possible. The workgroup has decided to define that timeframe as 24 hours to ensure the extracts are analyzed as quickly as possible. It is at the discretion of the analyst and the data user if additional QC data can be used to demonstrate either the potential, or absence, of bias if the extract is analyzed past 24 hours since methylation. A data validator would need to review and narrate how the data may still be usable for the intended use, particularly if the intended use is to verify a site as meeting the Remediation Standard Regulations (RSRs).

DEEP will add language regarding considerations for esterification to Section 1.3.3 “Special Precautions” of the RCP for clarification.

DEEP is making the following revision to 8151:

The following language will be added to Section 1.3.3 "Special Precautions": Esterification duration is critical to the herbicide recoveries. Methylated extracts are subject to trans-esterification and other unwanted side reactions. Sample extracts must be analyzed immediately after the methylation procedure has been performed in order to minimize the trans-esterification

and other potential reactions that may occur. This is critical in the evaluation of whether the holding time criteria were achieved. The following language will be added as footnote #4 added to table 5.0: Re-esterification may be warranted if the analyst suspects potential influence on extracts. It is at the discretion of the analyst and data user if additional QC analysis may be used to demonstrate either the potential, or absence, of bias if the extra is analyzed past 24 hours past methylation.

RCP CT ETPH

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: No specific language referenced.

Comment: *Per Method 8000 (9.4.1); When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. In the case of very contaminated samples or when the lab does not receive enough samples to perform a single matrix spike, an LCS and LCS duplicate (LCSD) may be performed to document precision and bias. An LCS should be included with each preparation batch. The LCS is an aliquot of the same clean (control) matrix used for the method blank(s) and of a similar weight or volume as the method blank and field samples. The LCS is spiked with similar analytes at the same concentrations as in the matrix spike and is processed identically to the samples.* The laboratory should only be responsible for running an LCSD when this is the case. Not for every batch.

Response:
It has become common practice in most laboratories to include an LCS/LCSD in each batch. It is DEEP's intent to maintain consistency in data quality between all laboratories used for characterization, investigation, and remediation purposes. The use of an LCS/LCSD for the batch versus only using an MS/MSD provides all data users whose samples are submitted in the same batch with details on analytical precision. Whereas, if only MS/MSD is used to report the analytical precision, that data is only applicable to the data user whose sample was used for the MS/MSD. Therefore, it is the use of an LCS/LCSD that will provide data users with more complete QC for the purposes of their data validation and intended use of the data.

No revisions will be made in response to this comment.

Section Reference: 1.4.1 Reporting Limits/Lower Limits of Quantitation for the CT-ETPH Method; Table 2.0

RCP Language: *Table 2.0: Typical Reporting Limits. Water = 100 µg/L & Soil/Sediment 100 mg/kg*

Comment: The RL may be higher than needed. Typically, clients are looking for around 50mg/Kg and 100ug/L.

Response:
The comment references the reporting limits provided in Table 2.0 of the ETPH RCP. It should be noted the RLs/LLOQs listed in the Table 2.0 are "typical" meaning they are commonly achievable

by most, if not all, laboratories. It is acknowledged that laboratories may be able to achieve lower reporting limits/lower limits of quantitation and may use lower RL/LLOQs to meet project DQOs. Note, that it is acceptable for laboratories to report lower RL/LLOQs if they can be achieved for this method.

No revisions will be made in response to this comment.

Section Reference: Section 1.4.1 Reporting Limits/Lower Limits of Quantitation for the CT-ETPH Method; Table 2.0

RCP Language: *Table 2.0: Typical Reporting Limits. Water = 100 µg/L & Soil/Sediment 100 mg/kg*

Comment: The reporting limits for water defined in the RCP of 100 µg/L conflicts with the "Extractable Petroleum Hydrocarbon Fractions Using the ETPH Analytical Method and Criteria Development Technical Support Document" dated, July 2012 that states the RL/LLOQ for ETPH in aqueous samples should be 250 µg/L.

Response:
DEEP acknowledges the 2012 Technical Guidance Published by DEEP & the Connecticut Department of Public Health (DPH) denoted a need for the reporting limit to be raised to 250 µg/L due to instrument limitations to achieve an RL/LLOQ of 100 µg/L. However, since the 2012 Technical Document was published, technology has improved, and it is DEEP's understanding that achieving an RL/LLOQ <250 µg/L is regularly achievable while achieving an RL/LLOQ of 100 µg/L still presents a challenge for current instrumentation. As such, DEEP will adjust the RL/LLOQ for aqueous matrices to 150 µg/L. Note, that it is acceptable for laboratories to report lower RL/LLOQs if they can be achieved for this method.

DEEP is making the following revision to RCP CT-ETPH:

Table 2.0, Water Matrix, 150 µg/L

Section Reference: Table 1A - Discrimination Check, Required Deliverable & 1.6 Routine Reporting Deliverables for the CT-ETPH Method Table 4.0

RCP Language: *Required Deliverable: Yes*

Comment: Would prefer to make Discrimination check not required as part of the deliverable. Difficult to contain in QC report. Not a standard QC issue.

Response:
Discrimination checks have been a required deliverable since the inception of the 2006 ETPH RCP and no change was proposed in this revision process. This comment is outside of the scope of the revision process.

No revisions will be made in response to this comment.

Section Reference: Section 1.2.1 Sample Extraction and Cleanup; Table 1.0

RCP Language: *Table 1.0 Extraction Methods*

Comment:
Please add Solid Phase Extraction (SPE) (EPA Method 3535) and Waste Dilution (EPA Method 3580) to list of method extractions. These methods have been established by the analytical community. Method 3535 uses less solvent in the sample preparation which means less waste is produced by the laboratory community.

Response:
QA workgroup came to consensus that use of SPE and Waste Dilution as extraction methods for ETPH would be appropriate, as long as laboratories demonstrate Quality Assurance/Quality Control measures used to meet the Data Quality Objectives for the intended purpose of the data requested by the data user. The use of SPE is beneficial to decreasing volumes of solvent used in the laboratory. Method 3580 will allow consultants to analyze pure product if necessary to better compare to a known release.

DEEP is making the following revision to RCP CT-ETPH:

Add Method 3535, Matrix: Aqueous, Description: Solid-Phase Extraction ("SPE") to Table 1.0.

Add Method 3580, Matrix: NAPL, Description: Waste Dilution to Table 1.0.

Section Reference: Table 1A - Method Blank, Required Corrective Action, Item 1

RCP Language: *"(1) Locate source of contamination and correct problem. Reanalyze method blank. Re-extract samples if method blank contaminated."*

Comment:
Consider adding language found in the EPH RCP "(2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample."

Response:
DEEP acknowledges the benefit of adding similar language from EPH into the same corrective actions under ETPH. Table 1A will be adjusted to include the suggested language.

DEEP is making the following revision to RCP CT-ETPH:

Adding the following language to Table 1A - "(2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample."

Section Reference: Table 1A - Laboratory Control Sample Duplicate

RCP Language: No applicable language.

Comment: There is no mention of the use of a LCSD and/or if it is required. The use of a LCSD is typical laboratory practice to evaluating laboratory precision. If optional, or required, the use of an LCSD should be noted somewhere in the RCP or it should be included on this table.

Response:
Although not published in the original method, DEEP acknowledges the benefit of the evaluation of an LCSD for DQOs, therefore information for the LCSD will be added to Table 1A.

DEEP is making the following revision to RCP CT-ETPH:

Add LCSD row to Table 1A including acceptance criteria and corrective actions similar to the Massachusetts Department of Environmental Protection (MADEP) EPH Method.

RCP MA EPH

Section Reference: 1.1 Method Review

RCP Language: "*The Extractable Petroleum Hydrocarbons ("EPH") Method ("the EPH Method") is based on a solvent extraction, silica gel solid-phase extraction ("SPE")/fractionation process and gas chromatography ("GC") analysis using a flame ionization detector ("FID") to identify and quantify both Target Polynuclear Aromatic Hydrocarbons ("PAH") analytes and method-defined aliphatic and aromatic hydrocarbon fractional ranges in water, soils and sediments. Extractable aliphatic hydrocarbons are collectively quantified within two specific ranges: C9 through C18, and C19 through C36. Extractable aromatic hydrocarbons are collectively quantified within the C11 through C22 range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150°C and 265°C. This method may also be used to identify and quantify specific Target PAH Analytes, including Diesel PAH analytes."*

Comment: This should allow for the analysis by GC/MS as well as FID. It does so in METPH method.

Response:
The EPH method was established/validated using FID to analyze hydrocarbon ranges, the method was not established/validated using GC/MS to quantify the hydrocarbon ranges. Therefore, it is important to note the use of GC/MS should not be used to analyze hydrocarbon ranges. Tables 1A and 5.0, Section 1.5.1, and the new Appendices A-1 and A-2 acknowledge the use of GC/MS to quantify PAH analytes and target aliphatic/aromatic hydrocarbons, as such the RCP guidance document does suggest the use of GC/MS is allowed. It should be noted that using GC/MS for quantifying hydrocarbon ranges would be considered a modification of the method and requires notification on the RCP certificate checklist that a modification from the RCP was used.

No revisions will be made in response to this comment.

Section Reference: 1.2 Summary of the EPH Method

RCP Language: *"A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated in a Kuderna-Danish apparatus."*

Comment: Request to also include suitable alternatives to glassware utilized by laboratories as not all laboratories use Kuderna-Danish equipment.

Response:
DEEP acknowledges the importance of allowing flexibility with laboratory equipment and resources. It is understood the use of QC samples will demonstrate the efficacy of the alternative equipment when achieving DQOs.

DEEP is making the following revision to RCP EPH:

A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated in a Kuderna-Danish apparatus, or suitable alternative equipment that can achieve the necessary Data Quality Objectives.

Section Reference: 1.2. Summary of the EPH Method; Table 1.0

RCP Language: *Table 1.0 EPH Method Marker Compounds*

Comment: Suggest adding a footnote to the table that start and stop times for ranges should be based on the start or end of the peak elution and not the absolute peak retention time.

Response:
DEEP acknowledges that adding the footnote for clarification in Table 1.0 will allow variances in peak widths dependent on chromatographic conditions between laboratories.

DEEP is making the following revision to RCP EPH:

Add the following footnote to Table 1.0: Start and stop times for ranges should be based on the start or end of the peak elution and not the absolute peak retention time.

Change "Beginning Marker" and "Ending Marker" to "Start Time" and "Stop Time" for language consistency with the added footnote.

Section Reference: 1.2.1 Sample Analysis Procedure

RCP Language: *[1] "It should be noted that the recommended hexane elution volume (20 mL) is critical and may need to be adjusted for each lot of silica gel/cartridges to optimize sample extraction and fractionation efficiencies."*

[2] "Aliphatic and aromatic extracts are introduced into the gas chromatograph separately by directly injecting 1 to 4 µL of each extract using the solvent flush technique."

[3] "The sequence ends when the set of sample extracts has been injected or when qualitative and/or quantitative QC criteria are exceeded."

Comment: [1] Suggest language revision: "It should be noted that the hexane elution volume is critical and should be verified or adjusted for each lot of silica gel/cartridges to optimize sample extraction and fractionation efficiencies."

[2] Strike "using the solvent flush technique." Not all labs use this injection technique.

[3] The sequence ends with a successful closing Continuing Calibration Verification (CCV).

Response:
[1] DEEP acknowledges that the 20 mL specified in the language may not be applicable to various types of silica cartridges available for laboratory use. The specific volume will be struck from the RCP language.

[2] DEEP acknowledges that laboratories may use different injection techniques and/or equipment. The suggested revision will be made.

[3] DEEP acknowledges, the language in the RCP inaccurately reflects that an analytical sequence could end with the successful injection of sample extracts. It is necessary to accept the suggested language to clarify the necessary QC sample analysis to successfully end an analytical sequence.

DEEP is making the following revision to RCP EPH:

It should be noted that the hexane elution volume is critical and should be verified or adjusted for each lot of silica gel/cartridges to optimize sample extraction and fractionation efficiencies.

Aliphatic and aromatic extracts are introduced into the gas chromatograph separately by directly injecting 1 to 4 µL of each extract.

The sequence ends with the successful closing Continuing Calibration Verification (CCV) QC sample.

Section Reference: Table 1A - Continuing Calibration Verification, Required Performance Standard, Item 1.

RCP Language: "(*1) Prior to samples, every 24 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence."*

Comment: RCP method says 20 samples or 24 hours. CCV should be analyzed every 12 hrs. Page 48 Section 9.10.2.9 of MA EPH Method indicates these times are associated with the Tuning Requirements. For FID the requirement is to analyze the CCV every 24 hr.

Response:
The comment references the EPH Method to clarify the timing requirement for the Continuing Calibration Verification QC standard. The comment suggests the time used in Table 1A is incorrectly referencing the Tuning Requirements under Section 9.10.2.9. It should be noted Section 9.10.2.9 of the 2019 EPH Method states "*The analytical batch for EPH analyses may*

include the analysis of up to 20 samples completed within 12 hours of the batch's tune." Note, Section 9.7.3 (Continuing Calibration) of the EPH Method states "*A Continuing Calibration Standard must be analyzed daily prior to sample analysis, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence.*" The new proposed language in the RCP reflects the language of the promulgated EPH Method.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Continuing Calibration Verification, Required Performance Standard, Items 5 & 6.

RCP Language: "*(5) Opening CCV: %D must be ≤25 for all target PAH analytes and hydrocarbon ranges. (6) Closing CCV: up to four compounds may exhibit %D or % drift >25 but <40.*

Comment: RCP Method says that Percent Drift is less than 25%. MA EPH Method (Page 48) states "PAH less than 20%, EPH Data less than 25%)

Response:
The comment references page 48 of the MA EPH method regarding percent drift. It should be noted page 48 of the MA EPH method contains Table 7 pertaining to using "Modified SW-846 Method 8270E" in substitution for the primary EPH analysis; this is also narrated in Section 9.10.2 of the EPH method. Therefore that reference would not be in agreement with the primary procedure for using the EPH Method.

Note an earlier section in the EPH Method, Section 9.7.3.5, states "*The %D or Percent Drift for each Target PAH Analyte, surrogate, and hydrocarbon range must be ≤25.",* while for the closing CCV it states *"…four compounds may exhibit %Ds or Percent Drifts greater than 25% but less than 40%*." The language used in the RCP reflects the language of the promulgated EPH Method.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Fractionation Check Standard, Required Deliverable & Table 5.0 Report Deliverables

RCP Language: *Fractionation Check is a required deliverable.*

Comment: This seems excessive, LCS/LCSD recoveries as well as breakthrough monitoring/reporting should be sufficient. All fractionation lot checks will be kept on file at the laboratories and lot checks can be performed if requested.

Response:
The QA Workgroup discussed and agreed that the Fractionation Check is not a necessary Report Deliverable for the Data User. Table 1A and Table 5.0 will be revised to reflect that the Fractionation Check is not a Required Deliverable.

DEEP is making the following revision to RCP EPH:

Section Reference: Table 1A - General Report Issues, Required Performance Standard, Item 2

RCP Language: *"(2) Dilutions- if diluted and undiluted analyses are performed, the lab should report results for the lowest dilution within the valid calibration range for each analyte…"*

Comment: Statement 2 seems to be incomplete as it does not clarify between target analytes and hydrocarbon ranges.

Response:
Language will be added specifying target PAH analytes and hydrocarbon ranges for clarification.

DEEP is making the following revision to RCP EPH:

Table 1, General Reporting Issues, Item 2: Dilutions- if diluted and undiluted analyses are performed, the lab should report results for the lowest dilution within the valid calibration range for each target PAH analyte and hydrocarbon range.

Section Reference: Table 1A - General Report Issues, Required Performance Standard, Item 5

RCP Language: *"(5) Concentrations below the reporting limit should be report as "ND" with the sample specific RL/LLOQ also reported."*

Comment: "Sample specific" should read "analyte specific".

Response:
DEEP acknowledges that the RL/LLOQ is analyte specific rather than sample specific. This language will be revised.

DEEP is making the following revision to RCP EPH:

Table 1A, General Reporting Issues, Item 5: Concentrations below the RL/LLOQ should be report as "ND" with the analyte specific RL/LLOQ also reported.

Section Reference: 1.5 Analyte List for the EPH Method

RCP Language: *Table 1B Analyte List*

Comment: There is no mention of adjusted C11-C22 aromatics vs unadjusted C11-C22 aromatics. Requesting clarification if there is a need to define one versus the other, or report both.

Response:
To provide clarification to this question, DEEP will add footnotes from the EPH method Appendix 3 to Table 1B including: "(1) Hydrocarbon Range data exclude area counts of any surrogate(s) and/or internal standards eluting in that range (2) C11-C22 Aromatic Hydrocarbons exclude the

concentrations of Target PAH Analytes. (3) C11-22 Unadjusted Aromatic Hydrocarbons include the concentration of Target PAH Analytes."

DEEP is making the following revision to RCP EPH:

Add the following footnotes to Table 1B: (1) Hydrocarbon Range data exclude area counts of any surrogate(s) and/or internal standards eluting in that range. (2) C11-C22 Adjusted Aromatic Hydrocarbons exclude the concentrations of Target PAH Analytes. (3) C11-22 Unadjusted Aromatic Hydrocarbons include the concentration of Target PAH Analytes.

Section Reference: 1.6 Routine Reporting Deliverables for the EPH Method; Table 5.0

RCP Language: *Table 5.0 Report Deliverables; Initial Calibration Verification Required Deliverable = Yes*

Comment: The ICV is not typically reported in lab report packages, except for when full data packages are requested. Suggestion is to make this a non-deliverable.

Response:
DEEP agrees this was likely a clerical error. As such, the ICV will not be required as a Report Deliverable which would also be consistent with Table 1A in this RCP.

DEEP is making the following revision to RCP EPH:

ICV Report Deliverable = No

RCP MA VPH

Section Reference: 1.1 Method Review

RCP Language: *"The Volatile Petroleum Hydrocarbons Method (the "VPH Method") uses purge-and-trap sample concentration, gas chromatographic ("GC") separation using photoionization and flame ionization detectors ("PID/FID") in-series. This method is designed to identify and quantify both target analytes and method-defined aliphatic and aromatic hydrocarbon fractional ranges in water, soils and sediments. Volatile aliphatic hydrocarbons are collectively quantified within two specific ranges: C5 through C8, and C9 through C12. Volatile aromatic hydrocarbons are collectively quantified within the C9 to C10 range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 36°C and 220°C. This method may also be used to identify and quantify benzene, toluene, ethylbenzene, xylenes ("BTEX"), naphthalene, and methyl tertiary butyl ether ("MTBE") as Target Analytes."*

Comment: This should reference that you are permitted to use GC/MS.

Response:
The use of GC/MS is discussed in the new Appendix 1 of the RCP, Section A-1.2, and does suggest the use of GC/MS is allowed. The RCP guidance document references the latest promulgated version of the MA VPH method (Section 1.1) which would also reflect the use of GC/MS under certain conditions. There is a separate VPH method using GC/MS, but there is no

promulgated RCP for that method. To utilize the alternative method from the RCP, it must be noted on the RCP certificate form and the data user will need to submit a demonstration of equivalency when using the data acquired using a method without a promulgated RCP.

No revisions will be made in response to this comment.

Section Reference: 1.2.2 Analysis of Water Samples

RCP Language: "*Water samples may be analyzed directly without sample preparation. The analysis of water samples is described in detail in the VPH Method. In general, a sample aliquot is introduced to the purge chamber using a 5 mL gas-tight syringe. If necessary, samples may be diluted prior to injection into the purge chamber. In such cases, sample dilutions must be performed as expeditiously as possible, and the diluted sample should be transferred to a gas-tight syringe without delay."*

Comment: In almost all cases it is an automated system and this is a bit misleading.

Response:
This language is intended to provide a general background of how the method works for the data user. It is not intended to direct laboratories on how to conduct an analysis. DEEP expects laboratories are following approved Standard Operating Procedures required by applicable accreditation programs.

No revisions will be made in response to this comment.

RCP MA APH

Section Reference: Table 1A – Initial Calibration Verification & Laboratory Control Sample, Required Corrective Action & Required Analytical Response Action (inclusive)

RCP Language: *All language in both Required Corrective Action & Required Analytical Response Action columns*

Comment: There is an apparent conflict between the "Required Corrective Action" and "Required Analytical Response" between the ICV and LCS. The Method states the LCS can be analyzed in lieu of analyzing an ICV, therefore the Corrective Actions & Analytical Responses for both QC samples should be the same. Can DEEP clarify why they are not the same?

Response:
DEEP acknowledges the discrepancy noted in the comment. The corrective actions and required analytical responses should be the same for both QC samples as noted by the Commenter. The original language in the ICV will be updated to match the LCS.

DEEP is making the following revision to RCP APH:

Revised the language in both referenced columns in the ICV row to match the language in the LCS columns.

Section Reference: Table 1A - General Reporting Issues, Required Analytical Response Action, Item 4

RCP Language: *"(4) If canister vacuum on receipt is >15 in. Hg or if the laboratory receipt canister vacuum differs from final field vacuum by more than ±5 in Hg, the data user should be contacted before analysis can proceed; the canister pressure anomalies must be explained in the laboratory narrative."*

Comment: Commentor noted, at their laboratory, they notify clients of the ± 5 in. Hg. However, if <15 in. Hg, they don't pressurize the sample to avoid further dilution of sample.

Response:
Details related to pressurization of the canisters can be found in Section 1.5 Special Analytical Considerations for the APH Method in the RCP which includes more details of what actions laboratories may take if cannister pressure deviates from the required pressure defined in the APH Method.

No revisions will be made in response to this comment.

RCP 8260 – Volatiles by GC/MS

Section Reference: Section 1.3.2 Cross-contamination/Carryover

RCP Language: *"Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of VOCs. Autosampler positions on the purge and trap unit may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of method blanks at the same autosampler position as the high sample. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run."*

Comment: Why do method blanks have to be run at the position of high level sample? The position does not affect carryover as it has already been introduced to the system.

Response:
DEEP acknowledges the language used in this section may refer to older methodologies that utilized different autosampler technology than is available today. The language will be revised to remove reference to autosampler positions.

DEEP is making the following revision to RCP 8260:

Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of VOCs. Autosampler positions on the purge and trap unit may also become contaminated in the same manner. If a high sample is inadvertently analyzed, the system must be demonstrated to be clean by analysis of method. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may

also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later run.

Section Reference: Table 1A – Initial Calibration, Required Performance Standard, Item 1

RCP Language: *"(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed."*

Comment: There appears to be a conflict in the tuning language in Table 1A of the 8260 RCP with the language in EPA Method 8260D. Method 8260 requires a new calibration when tune adjustments are made. However, the RCP Table 1A appears to only require a tuning every 12 hours.

Response:
The 12 hour tune referenced in Table 1A "GC/MS Tunes with BFB" row is in reference to running a tune verification check against the calibration curve as is required by Method 8260D every 12 hours. It is important to note that this should not be conflated with analyzing a new calibration curve. As noted by the Commenter, Method 8260D requires a new calibration when the instrument has been re-tuned, i.e., a tune check was conducted and failed to meet criteria, resulting in instrument maintenance/re-tune. Item 1 under Initial Calibration references that a new calibration must be conducted when "instrument maintenance is performed." To provide clarification, a note "(e.g., when re-tuned)" will be added to Item 1 of the Required Performance Standard under the Initial Calibration.

DEEP is making the following revision to RCP 8260:

Table 1A, Initial Calibration, Required Performance Standard, Item 1: Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed (e.g., if the instrument is retuned).

Section Reference: Table 1A - Initial Calibration, Required Performance Standard, Item 10

RCP Language: *"(10) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%."*

Comment: Method 8260D Section 11.3.5.4 states "*The recalculated concentration of the calibration standard corresponding to the LLOQ, especially where linear regression fits are used, should be within ±50% of the standard's true concentration if it is the lowest point recovery is +/- 50%.*"

Response:
DEEP acknowledges the recovery range indicated in Table 1A does not agree with EPA Method 8260D. The recovery range will be revised to 50-150% per EPA Method 8270D.

DEEP is making the following revision to RCP 8260:

Table 1A, Initial Calibration, Required Standard Performance, Item 10: If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 50-150%.

Section Reference: Table 1A - Initial Calibration, Required Performance Standard, Item 6 **and** Continuing Calibration Verification, Required Performance Standard, Item 5

RCP Language: "*(6) Minimum RFs for each compound as per SW-846 8260 for lowest concentration standard and for average RF."* ***And*** *"(5) Minimum RFs as per SW-846 8260."*

Comment: Section 11.3.4.2 in Method 8260D: Table 4 contains minimum RFs that may be used as guidance in determining if the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 4 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information is provided as guidance only. Therefore, this requirement should be removed from the RCP table.

Response:
DEEP acknowledges the comment and the language change in 8260D that adjusted the RF requirements to guidance. However, DEEP has the authority to maintain conservative approaches to ensure data quality objectives for intended uses of data that are tantamount to protecting human health and the environment.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Continuing Calibration Verification, Required Corrective Action, Item 3

RCP Language: *"(3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D <40."*

Comment: Method 8260D doesn't specify a minimum recovery value for CCV.

Response:
The language used in Table 1A refers to the language used in Section 11.4.3.1 of Method 8260D: *"The calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20% of the expected value. NOTE: For the RF calibration model, % difference (%D) between the calculated RF of an analyte in the calibration verification standard and the RFavg of that analyte from the ICAL is the same value as % drift for calculated vs. expected concentration."* Further information is available in Section 11.4.3.2 of 8260D. The language used in the RCP reflects the language of the latest version of Method 8260.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Laboratory Control Sample Duplicate

RCP Language: *LCS Duplicate Row*

Comment: Should only have to run LCSD when an MS/MSD cannot be analyzed. Please consider removing the LCSD as a required QC sample.

Response:
It has become common practice in most laboratories to include an LCS/LCSD in each batch. It is DEEP's intent to maintain consistency in data quality between all laboratories used for characterization, investigation, and remediation purposes. The use of an LCS/LCSD for the batch versus only using an MS/MSD provides all data users whose samples are submitted in the same batch with details on analytical precision. Whereas, if only MS/MSD is used to report the analytical precision, that data is only applicable to the data user whose sample was used for the MS/MSD. Therefore, it is the use of an LCS/LCSD that will provide data users with more complete QC for the purposes of their data validation and intended use of the data.

No revisions will be made in response to this comment.

RCP 8270 – Semi-Volatiles by GC/MS

Section Reference: Section 1.2.2 GC/MS Analysis in Full Scan Mode

RCP Language: *"Quantitation is accomplished by using the response of a major (quantitation) ion relative to an internal standard and a response factor generated from a five-point curve."*

Comment: Suggest revising language to read "…a calibration curve consisting of a minimum of five points, or six if non-linear regression is used." This would bring the language into agreement with EPA Method 8270 and language in RCP Table 1A.

Response:
DEEP acknowledges the RCP language will benefit from reflecting the language in EPA Method 8270 and also that laboratories should have flexibility to include more points in the calibration curve to improve correlation coefficients and detection accuracy.

DEEP is making the following revision to RCP 8270:

"Quantitation is accomplished by using the response of a major (quantitation) ion relative to an internal standard and a response factor generated from a calibration curve consisting of a minimum of five points, or six if non-linear regression is used."

Section Reference: Section 1.2.3 GC/MS System Operating in the Selective Ion Monitoring ("SIM") Mode

RCP Language: *"Sample preparation, chromatographic conditions, analyte identification, and analyte quantification are the same whether the GC/MS system is operated in the full scan or SIM mode.""*

Comment: Suggest revising language for clarification of statements. As currently written, the language is slightly inaccurate. Suggested language: "*GC/MS SIM is an invaluable tool for*

improving detection limits. Coupled with full scan data, positive identification of analytes of concern is preserved. For some analytes, sensitivity may be increased by a factor of ten (10), as compared with a GC/MS system operated in the full scan mode."

Suggest revising the language of the first sentence of the subsequent paragraph: "*Sample preparation, chromatographic conditions, and analyte quantification are the same whether the GC/MS system is operated in the full scan or SIM mode."*

Response:
DEEP acknowledges the suggested language changes clarify that the functionality of GC/MS SIM is limited to quantitation purposes, rather than identification purposes, and therefore must be coupled with scan data for positive identification purposes. The existing language conflates the two detection modes, although they each serve different purposes.

DEEP is making the following revision to RCP 8270:

GC/MS SIM is an invaluable tool for improving detection limits. Couple with full scan data, positive identification of analytes of concern is preserved. For some analytes, sensitivity may be increased by a factor of ten (10), as compared with a GC/MS system operated in the full scan mode.

Sample preparation, chromatographic conditions, and analyte quantification are the same whether the GC/MS system is operated in the full scan or SIM mode.

Section Reference: Table 1A – Initial Calibration, Required Performance Standard, Item 1

RCP Language: *"(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed."*

Comment: There appears to be a conflict in the tuning language in Table 1A of the 8270 RCP with the language in EPA Method 8270E. Method 8270 requires a new calibration when tune adjustments are made. However, the RCP Table 1A appears to only require a tuning every 12 hours.

Response:
The 12-hour tune referenced in Table 1A "GC/MS Tunes with DFTPP" row is in reference to running a tune verification check against the calibration curve as previously required by Method 8270. It is important to note that this should not be conflated with analyzing a new calibration curve. However, DEEP acknowledges the latest updates to Method 8270 (i.e., 8270E) removed the requirement for the tune verification check every 12 hour, rather the tune verification check is now required to be conducted before every initial calibration.

Details for recalibration can be found in Method 8000D, Section 9.2.5. This referenced section requires recalibration of the instrument when the acceptance criteria for the Initial Calibration Verification and/or Continuing Calibration Verification cannot be achieved. Additionally, recalibration should occur when major instrument maintenance is performed, see Item 1 under Initial Calibration in Table 1A of the 8270 RCP. To provide clarification, a note "(see Method 8000 for guidance)" will be added to Item 1 of the Required Performance Standard under the Initial Calibration.

DEEP is making the following revision to RCP 8270:

Table 1A, Initial Calibration, Required Performance Standard, Item 1: Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed (see Method 8000 for guidance).

Table 1A, GC/MS Tunes with DFTPP, Required Performance Standard, Item 2: Prior to initial calibration.

Section Reference: Table 1A - Initial Calibration, Required Performance Standard, Items 6 **and** Continuing Calibration Verification, Required Performance Standard, Item 5

RCP Language: "*(6) Minimum RFs for each compound as per SW-846 8270 for lowest concentration standard and for average RF."* ***And*** *"(5) Minimum RFs as per SW-846 8270."*

Comment: Section 11.3.4.2 in Method 8270E: Table 4 contains minimum RFs that may be used as guidance in determining if the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 4 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information is provided as guidance only. Therefore, this requirement should be removed from the RCP table.

Response:
DEEP acknowledges the comment and the language change in 8270E that adjusted the RF requirements to guidance. However, DEEP has the authority to maintain conservative approaches to ensure data quality objectives for intended uses of data that are tantamount to protecting human health and the environment.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Initial Calibration, Required Corrective Action, Items 1 & 2

RCP Language: *"(1) Recalibrate if >10% of target analytes exceed %RSD, "r" or "r2" criteria. (2) If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98 or r2 >0.98."*

Comment: SW-846 method 8000 and 8270E states that if >10% of analytes exceed the 20% Relative Standard Deviation (RSD) or the r value >0.99, the instrument must be recalibrated. The RCP states that as long as the RSD <40% and r value >0.98 it passes. There seems to be a conflict between the EPA Method and the RCP language.

Response:
The referenced language is presenting for scenarios in which <10% of analytes exceed the 20% RSD requirement (per column 3), as long as they don't exceed 40%RSD and r value is >0.98 the analysis can continue, but narration is required for any analytes outside of this performance standard. The language in 8270E is presenting for the scenario in which >10% of analytes exceed

the 20% RSD and the r value is <0.99 which would require recalibration. The scenarios are the same.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Initial Calibration, Required Performance Standard, Item 9

RCP Language: *"(9) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve, recoveries must be 70-130%."*

Comment: Section 11.3.6 in 8270E states that when recalculating the lowest calibration point against the curve it must pass within +/- 50% of the true value not 70-130%.

Response:
DEEP acknowledges the recovery range indicated in Table 1A does not agree with EPA Method 8270E. The recovery range will be revised to 50-150% per EPA Method 8270E.

DEEP is making the following revision to RCP 8270:

(9) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve, recoveries must be 50-150%.

Section Reference: Table 1A - Tunes and DFTPP, Required Performance Standard; Note

RCP Language: *"Note: Pentachlorophenol tailing must be evaluated when analyzing for acid SVOCs and benzidine tailing must be evaluated when analyzing for base-neutral SVOCs."*

Comment: Both compounds give insight to the performance of the instrument. Suggest losing the wording of splitting them to assess samples that only contain a specific fraction. Method 8270E stipulates calculating tailing for both compounds before analysis of samples can begin.

Response:
The current language provides the flexibility for laboratories to assess specific classes of SVOCs (i.e., acids or base-neutrals) separately when only reporting either one or the other.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Continuing Calibration Verification, Required Corrective Action, Items 1 & 3

RCP Language: "*(1) Recalibrate if >20% all target analytes or >15% of analytes from a particular class (base-neutral or acid) exceed %D criteria. (3) If ≤20% of compounds exceed criteria, recalibration is not required as long as %D <40."*

Comment: Disagree that 'class' criteria should be a part of the evaluation.

Response:
Use of the term "class" in the RCP language is in reference to the chemical classification of a group of compounds. In the context of this RCP, the "class" refers to the groups of either base-neutral or acid compounds. The RCP current language in Table 1A allows for the evaluation of select classes (i.e., base-neutral or acid) if data user specifically requests a class of compounds versus select target analytes.

No revisions will be made in response to this comment.

Section Reference: Section 1.6 Analyte List for SW-846 Method 8270, Table 1B

RCP Language: *Table 1B: Acetophenone CAS #98862*

Comment: Add Acetophenone as an analyte.

Response:
Acetophenone was added in the updated version of the 8270 RCP. Please refer to the DEEP QA webpage where both the red-lined and final draft versions were made available.

No revisions will be made in response to this comment.

RCP 6010 – Metals ICP-OES

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: "*The use of site-specific MS/MSDs samples are required for solids samples (soil/sediment)."*

Comment: Clients do not routinely request solid sample MS/MSD and would be too much QC per batch. Change requirement to recommended or to make effort to MS/MSD sites in testing history.

Response:
The purpose of requiring MS/MSD for site evaluation is for data users to demonstrate the precision of analyte measurements in complex environmental matrices. This data will provide more substantial evidence that the data user has either demonstrated compliance or thoroughly evaluated the site conditions for their Conceptual Site Model. The data will also provide more insights to DEEP when the site attempts to verify they have demonstrated compliance with the RSRs.

No revisions will be made in response to this comment.

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: *"The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing*

performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD."

Comment: It appears the RCP is only requiring MD/MS pairs as opposed to MS/MSD pairs. Typically, certification programs require MS/MSD pairings. Is DEEP stating that MSD samples will no longer be an option?

Response:
Language in Section 1.4.2 of the RCP will be revised for clarification that MS/MSDs are required for soils, whereas MS/MSD *or* MS/MD pairings are strongly recommended for site-specific information. Information for the MSD sample will be added to Table 1A for clarification and consistency.

DEEP is making the following revision to RCP 6010:

The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MSD or MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MSD or MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MSD/MDs. A laboratory may substitute a MS/MSD in lieu of the MS/MD.

Table 1A – Matrix Spike Duplicate ("MSD") (site-specific) May elect to use in lieu of "MD".

Section Reference: Table 1A - Low-Level Calibration Verification, Required Performance Standard, Item 2

RCP Language: *"(2) Prepared using same source as initial calibration standards."*

Comment: Calibration standards tend to be multicomponent. Suggest removing requirement to use the same source standard for the calibration and for the low-level calibration verification QC sample.

Response: The low-level calibration verification (LLCV) QC sample is used to evaluate the low-end of the calibration curve for accuracy. Therefore, to properly evaluate recovery of all of the calibrated elements at the low-end of the curve, where there tends to be more instrument noise, it is important that the LLCV contain the same elements as the calibration curve. If the LLCV does not contain the same elements as the calibration standards, it may not be possible to evaluate if elements of interest are recovering well at the low end of the calibration curve.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Low-Level Calibration Verification, Required Performance Standard, Item 3

RCP Language: *"(3) Percent recoveries must be 70-130% for all target analytes."*

Comment: LLCV recovery at 80-120% too tight of a window for low level, specifically problem elements of arsenic, antimony and thallium. recommend keeping 50% for these elements and 30% for others.

Response:
EPA Method 6010C defined the LLCV recovery range as 65-135%, however the updated Method 6010D revised the recovery range to 80-120%. The laboratory community has provided feedback that the recovery range of 80-120% is difficult to achieve at the low end of the calibration curve. Therefore, DEEP will revise the 6010 RCP to reflect a recovery range of 70-130% for the LLCV which is regularly achievable by laboratories.

DEEP is making the following revision to RCP 6010:

Table 1A, Low-Level Calibration Verification, Required Performance Standard, Item 3: Percent recoveries must be 70-130% for all target analytes.

Section Reference: Table 1A - Continuing Calibration Verification; Item 2

RCP Language: *"(2) Prepared using same source as initial calibration standard."*

Comment: Calibration standards tend to be multicomponent. Suggest removing requirement to use the same source standard for the calibration and for the Continuing Calibration Verification QC sample.

Response: The CCV QC sample is used to evaluate the stability of the calibration of all analytes throughout the analytical sequence. Therefore, to properly evaluate recovery of all the calibrated analytes in the calibration curve, it is important that the CCV contain the same elements as the calibration curve. If the CCV does not contain the same elements as the calibration standards, it may not be possible to evaluate if elements of interest are recovering well throughout the analytical sequence.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Linear Range Check, Required Performance Standard, Items 1-4

RCP Language: "*(1) Check linear range annually (SW-846 6010). (2) Determine the upper limit of the linear dynamic range for each wavelength by determining the signal responses from a minimum of different concentration standards across the range. See SW-846 Method 6010 for details. (3) At a minimum the LDR should be checked every year. A minimum of 3 different concentration standards across the ICP range one should be near the upper limit of the range. (4) Should concentrations be reported above the curve, a daily LDR verification standard must be analyzed. Percent recoveries must be within 90 – 110 % for each target analyte."*

Comment: What is the purpose of including the Linear Range Check (formerly Linear Dynamic Range) row if it is no used under the latest EPA Method? If calibration curve goes to 10ppm and LDR is verified to 100ppm yearly (which meets requirement 3), then do not need daily LDR to go above 10ppm. This would happen every day. At this point you would be calibrating to 100ppm, which is not the point of LDR.

Response:
DEEP acknowledges clarification is needed in Table 1A to agree with the latest updates included in EPA method 6010D. The requirement for the Linear Dynamic Range study was removed when EPA method 6010D was published. EPA Method 6010D states "*The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover within 10% of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.*" The language in Table 1A will be adjusted to reflect this EPA Method language.

DEEP is making the following revision to RCP 6010:

Table 1, Linear Range Check, Performance Standard, Items 1-4:
(1) Should concentrations be reported above the curve, a daily LDR verification standard must be analyzed.
(2) Percent recoveries must be within 90 – 110 % for each target analyte.
(3) The linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run.

Section Reference: Table 1A - Laboratory Control Sample Duplicate, Required Deliverable

RCP Language: *"Yes (ONLY if no MD)"*

Comment: The language in the Required Deliverable column implies that the use of a LCSD is allowed ONLY if there is no MD in the analytical batch. Can laboratories still proceed with using LCS/LCSD pairs?

Response:
The language used in Table 1A is specific to the data user, however, the language as written may present confusion for laboratory professionals. It is understood that the use of LCS/LCSD pairs is

regularly included in batch analyses and benefits all data users whose samples are analyzed in the same analytical batch. Therefore, reference to "ONLY if no MD", will be replaced with "if analyzed". This language will allow laboratories to choose to employ either a matrix duplicate (MD) or an LCSD to demonstrate precision within an analytical batch.

DEEP is making the following revision to RCP 6010:

Table 1A, LCSD, Required Deliverable, Yes (if analyzed)

Section Reference: Table 1A – Method Blank, Required Corrective Action, Item 2

RCP Language: *"(2) If reanalysis is still outside of criteria, recalibrated and reanalyze all associated samples since last compliant MB-unless 3 applies."*

Comment: Believe the term "recalibrated" in item 2 should read "re-digested" which would be the first corrective step taken prior to recalibration.

Response:
This clerical error will be corrected.

DEEP is making the following revision to RCP 6010:

Table 1A, Method Blank, Item 2: If reanalysis is still outside of criteria, re-digest and reanalyze all associated samples since last compliant MB-unless 3 applies.

RCP 6020 – Metals ICP-MS

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: *"The use of site-specific MS/MSDs samples are required for solids samples (soil/sediment)."*

Comment: Clients do not routinely request solid sample MS/MSD and would be too much QC per batch. Change requirement to recommended or to make effort to MS/MSD sites in testing history.

Response:
The purpose of requiring MS/MSD for site evaluation is for data users to demonstrate the precision of analyte measurements in complex environmental matrices. This data will provide more substantial evidence that the data user has either demonstrated compliance or thoroughly evaluated the site conditions for their Conceptual Site Model. The data will also provide more insights to DEEP when the site attempts to verify they have demonstrated compliance with the RSRs.

No revisions will be made in response to this comment.

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: *"The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD."*

Comment: It appears the RCP is only requiring MD/MS pairs as opposed to MS/MSD pairs. Typically, certification programs require MS/MSD pairings. Is DEEP stating that MSD samples will no longer be an option?

Response:
Language in Section 1.4.2 of the RCP will be revised for clarification that MS/MSDs are required for soils, whereas MS/MSD *or* MS/MD pairings are strongly recommended for site-specific information. Information for the MSD sample will be added to Table 1A for clarification and consistency.

DEEP is making the following revision to RCP 6020:

The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MSD or MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MSD/MDs. A laboratory may substitute a MS/MSDin lieu of the MS/MD.

Table 1A – Matrix Spike Duplicate ("MSD") (site-specific) May elect to use in lieu of "MD"

Section Reference: Table 1A - Daily Performance Standard, Required Performance Standard, Item 2

RCP Language: *"(2) Daily performance standard should be a 10µg/L standard of 3 or more elements representative of the analytical mass range. Analyze five replicates or five integrations."*

Comment: Concentration of performance standard will be manufacturer specific and should not be specified.

Response:
DEEP understands that laboratories purchase daily performance standards from instrument manufacturers that may vary between instrument makes and models. Given this variability, DEEP will revise Table 1A to refer to manufacturer specifications.

DEEP is making the following revision to RCP 6020:

Table 1A, Daily Performance Standard, Required Performance Standard, Item 2: Daily performance standard (provided by the instrument manufacturer) should composed of 3 or more elements representative of the analytical mass range. Analyze five replicates or five integrations.

Section Reference: Table 1A - Interference Check Standards (ICSA/AB), Required Corrective Action

RCP Language: *"This is a method requirement of SW-846 6020. No corrective action required because instrument corrections are based on natural isotope abundances that cannot be changed."*

Comment: The natural isotopes abundances cannot be changed, however, the factors would still be calculated, based closely on the natural abundances.

Response:
To provide clarification on the Required Corrective Action for the Interference Check Standards (ICSA/AB) row in Table 1A will be revised to reflect the updates to Method 6020 such that the ICSA/AB will now be referred to as "Spectral Interference Checks (SICs)" and will clarify the corrective actions that need to be taken should the SICs fail to meet criteria.

DEEP is making the following revision to RCP 6020:

The Interference Check Standards (ICSA/AB) row in Table 1A will be replaced with a "Spectral Interference Checks" row including the Required Performance Standards, Required Corrective Actions, and Required Analytical Response Actions, per the latest EPA Method 6020.

Section Reference: Table 1A - Continuing Calibration Verification; Item 2

RCP Language: *"(2) Prepared using same source as initial calibration standard."*

Comment: Calibration standards tend to be multicomponent. Suggest removing requirement to use the same source standard for the calibration and for the Continuing Calibration Verification QC sample.

Response:
The CCV QC sample is used to evaluate the stability of the calibration of all analytes throughout the analytical sequence. Therefore, to properly evaluate recovery of all the calibrated analytes in

the calibration curve, it is important that the CCV contain the same elements as the calibration curve. If the CCV does not contain the same elements as the calibration standards, it may not be possible to evaluate if elements of interest are recovering well throughout the analytical sequence.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Linear Range Check, Required Performance Standard, Items 1-4

RCP Language: "*(1) Check linear range annually (SW-846 6010). (2) Determine the upper limit of the linear dynamic range for each wavelength by determining the signal responses from a minimum of different concentration standards across the range. See SW-846 Method 6010 for details. (3) At a minimum the LDR should be checked every year. A minimum of 3 different concentration standards across the ICP range one should be near the upper limit of the range. (4) Should concentrations be reported above the curve, a daily LDR verification standard must be analyzed. Percent recoveries must be within 90 – 110 % for each target analyte."*

Comment: What is the purpose of including the Linear Range Check (formerly Linear Dynamic Range) row if it is no used under the latest EPA Method? If calibration curve goes to 10ppm and LDR is verified to 100ppm yearly (which meets requirement 3), then do not need daily LDR to go above 10ppm. This would happen every day. At this point you would be calibrating to 100ppm, which is not the point of LDR.

Response:
DEEP acknowledges clarification is needed in Table 1A to agree with the latest promulgated EPA method 6020B states. The requirement for the Linear Dynamic Range study was removed from Method 6020 in 2014, see Section 9.6 of the method. EPA method 6020B states *"The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover within 10% of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range."* The language in Table 1A will be adjusted to reflect the latest EPA language.

DEEP is making the following revision to RCP 6020 Table 1A:

(1) Should concentrations be reported above the curve, a daily LDR verification standard must be analyzed.
(2) Percent recoveries must be within 90 – 110 % for each target analyte.
(3) The linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run.

Section Reference: Table 1A - Internal Standards, Required Performance Standard, Item 2

RCP Language: *"(2) Relative Intensity (RI) of IS must be 70-130% of IS in midpoint standard of the initial calibration curve."*

Comment: Software does not designate Relative Intensity (RI) based on one point, it is spread throughout the blank point and curve.

Response:
DEEP acknowledges the RCP language used, i.e., 70-130%, is not in agreement with EPA Method 6020B, but rather should read ≥30% of the internal standard (IS). The language used in Table 1A referencing the midpoint initial calibration (ICAL) standard is meant to provide a point of reference for evaluating the relative intensity of the internal standard. The midpoint ICAL standard is the most statistically supported standard on the calibration curve. Therefore, using this midpoint ICAL standard should provide the most accurate relative intensity of the internal standard.

DEEP is making the following revision to RCP 6020:

(2) Relative Intensity (RI) of IS must be ≥30%of IS in midpoint standard of the initial calibration curve.

Section Reference: Table 1A – Dilutions, Required Performance, Item 2

RCP Language: *"(2) Perform 1:5 dilution on same sample used for MS/MD."*

Comment: Serial dilutions are a requirement under Method 6020, however it is not listed as a required deliverable under the RCP as written.

Response:
DEEP acknowledges the RCP, as written, conflicts with Method 6020. Table 1A will be revised to agree with Method 6020 and to reflect similar language in RCP 6010 for consistency purposes. The Report Deliverable column will also be updated to agree with Method 6020.

DEEP is making the following revision to RCP 6020:

Dilutions, Required Performance Standard, Item 2: Perform 5x serial dilution on same sample used for MS/MD.

Dilutions, Required Deliverable: Yes - ONLY if project specific MS requested by data user and analyte >25x RL/LLOQ

Section Reference: Table 1A - Laboratory Control Sample Duplicate, Required Deliverable

RCP Language: *"Yes (ONLY if no MD)"*

Comment: The language in the Required Deliverable column implies that the use of a LCSD is allowed ONLY if there is no MD in the analytical batch. Can laboratories still proceed with using LCS/LCSD pairs?

Response:

DEEP acknowledges the language used in Table 1A is specific to the data user, however, the language as written may present confusion for laboratory professionals. It is understood the use of LCS/LCSD pairs is regularly included in batch analyses and benefits all data users whose samples are analyzed in the same analytical batch. Therefore, reference to "ONLY if no MD", will be replaced with "if analyzed". This language will allow laboratories to choose to employ either a matrix duplicate (MD) or an LCSD to demonstrate precision within an analytical batch.

DEEP is making the following revision to RCP 6020:

Table 1A, LCSD, Required Deliverable, Yes (if analyzed)

Section Reference: 1.5 Analyte List for SW-846 Method 6020, Table 1B

RCP Language: *Table 1B: Analyte List, Mercury*

Comment: Mercury was removed as analyte without reasoning. It is part of method 6020 and should be included as acceptable for analysis.

Response:
DEEP offers the following justification for the exclusion of mercury from the RCP analyte list: Mercury was not included on the standard analyte list for this RCP protocol because of the special requirements for sample digestion and processing necessary to produce valid data that are not part of the standard operating procedure for method 6020. DEEP will add mercury back to the analyte list with the additional footnote in Table 1B: Mercury data collected based on RCP 6020 may be used for screening purposes, however, only if the sample digestion and processing precautions described in Section 11.1 of SW-846 Method 6020B are satisfied, and the overall QC and performance standards for this RCP protocol are met. Although mercury is not required to be reported to obtain "Reasonable Confidence" status for data using this RCP protocol, it must be given consideration as a contaminant of concern when sites with unknown, uncertain or complex history are assessed for potential contamination associated with "total metals" pursuant to 310 CMR 40.0191. Unless the special precautions can be met for mercury using Method 6020 as described above, the preferred analytical method for mercury is the RCP 7470/7471, based on SW-846 Methods 7470 and 7471 (cold vapor atomic absorption)

DEEP is making the following revision to RCP 6020:

Mercury will be added back to the analyte list including these footnotes: (1) Mercury data collected based on RCP 6020 may be used for screening purposes, however, only if the sample digestion and processing precautions described in Section 11.1 of SW-846 Method 6020B are satisfied, and the overall QC and performance standards for this RCP protocol are met. Although mercury is not required to be reported to obtain "Reasonable Confidence" status for data using this RCP protocol, it must be given consideration as a contaminant of concern when sites with unknown, uncertain, or complex history are assessed for potential contamination associated with "total metals" pursuant to 310 CMR 40.0191. Unless the special precautions can be met for mercury using Method 6020 as described above, the preferred analytical method for mercury is the RCP 7470/7471, based on SW-846 Methods 7470 and 7471 (cold vapor atomic absorption).
(2) Laboratory must be certified by CT DPH to analyze mercury using method 6020.

Section Reference: 1.4.2 General Quality Control Requirements

RCP Language: *"The minimum requirements for the QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and analysis of LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSDs sample is required for solids samples (soil/sediment). However, site-specific MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MD."*

Comment: It appears the RCP is only requiring MD/MS pairs as opposed to MS/MSD pairs. Typically, certification programs require MS/MSD pairings. Is DEEP stating that MSD samples will no longer be an option?

Response:
Language in Section 1.4.2 of the RCP will be revised for clarification that MS/MSDs are required for soils, whereas MS/MSD *or* MS/MD pairings are strongly recommended for site-specific information. Information for the MSD sample will be added to Table 1A for clarification and consistency.

DEEP is making the following revision to RCP 7470/7471:

The minimum requirements for the QA program include Initial Demonstration of Laboratory Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/or matrix spikes ("MS") to assess accuracy and LCS duplicates ("LCSD") or matrix duplicates ("MD") to assess precision. The use of site-specific MS/MSD sample are required for solids samples (soil/sediment). However, site-specific MS/MSD or MS/MD samples are strongly recommended from each site and for each matrix type sampled. Evaluation of sample matrix effects on element recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices and are in general discouraged. Field, rinsate, or other blanks should not be used for MS/MD's. A laboratory may substitute a matrix spike/matrix spike duplicate in lieu of the MS/MSD/MD. A laboratory may substitute a MS/MSD duplicate in lieu of the MS/MD.

Table 1A – Matrix Spike Duplicate ("MSD") (site-specific) May elect to use in lieu of "MD".

Section Reference: 1.2 Summary of SW-846 Methods 7470/7471

RCP Language: *"While SW-846 Method 7471 requires analysis of soil/solid samples in triplicate, the Department of Energy and Environmental Protection ("DEEP") does not require this for routine analysis of soil/sediment samples. In certain instances, however, triplicate analyses may be warranted where site homogeneity is in question."*

Comment: Reference to "triplicate" analysis is derived from archived Method 7471A. Suggest removing reference to this method to accurately reflect the version of Method 7471.

Response:
Reference to triplicate analysis will be removed to agree with the latest EPA Method 7471B.

DEEP is making the following revision to RCP 7470/7471:

Remove referenced paragraph from RCP.

Section Reference: Table 1A - Continuing Calibration Verification, Required Performance Standard Item 2 **and** Laboratory Control Sample, Required Performance Standard, Item 5.

RCP Language: "*(2) Prepared using same source as initial calibration standards."* **and** *"(5) Standard source same as initial calibration source."*

Comment: Request justification for why both CCV and LCS are specifically required to be from same the same standard source as the calibration curve.

Response:
DEEP understands LCS QC samples are typically prepared with a "second" source, that is a standard source that is different than the original source used to prepare the calibration standards. Using a "second" source for the LCS allows the analyst to evaluate the accuracy of the calibration curve in relation to a sample that contains analytes from a different source. DEEP acknowledges the language in the RCP conflicts with this general practice. The language in Table 1A of 7470/7471 will be adjusted to allow second source standard to be used for the LCS.

The CCV QC sample is used to evaluate the stability of the calibration of all analytes throughout the analytical sequence. Therefore, to properly evaluate recovery of all the calibrated analytes in the calibration curve, it is important that the CCV contain the same elements as the calibration curve. If the CCV does not contain the same elements as the calibration standards, it may not be possible to evaluate if elements of interest are recovering well throughout the analytical sequence. Therefore, DEEP will maintain the language that the CCV must be prepared with the same source standard as the calibration.

DEEP is making the following revision to RCP 7470/7471:

LCS, Required Performance Standard, Item 5: Prepared using second source standard.

Section Reference: Table 1A - Laboratory Control Sample Duplicate (inclusively)

RCP Language: *No applicable language.*

Comment: The RCP required that a LCSD must be analyzed if a site-specific MS/MSD is not performed. Clients rarely ask for a site-specific MS/MSD. Suggest an allowance for the lab to choose a sample for MS/MSD in place of LCSD.

Response:
It has become common practice in most laboratories to include an LCS/LCSD in each batch. It is DEEP's intent to maintain consistency in data quality between all laboratories used for characterization, investigation, and remediation purposes. The use of an LCS/LCSD for the batch versus only using an MS/MSD provides all data users whose samples are submitted in the same batch with details on analytical precision. Whereas, if only MS/MSD is used to report the analytical precision, that data is only applicable to the data user whose sample was used for the MS/MSD. Therefore, it is the use of an LCS/LCSD that will provide data users with more complete QC for the purposes of their data validation and intended use of the data.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Laboratory Control Sample Duplicate, Required Deliverable

RCP Language: *"Yes (ONLY if no MD)"*

Comment: The language in the Required Deliverable column implies that the use of a LCSD is allowed ONLY if there is no MD in the analytical batch. Can laboratories still proceed with using LCS/LCSD pairs?

Response:
DEEP acknowledges the language used in Table 1A is specific to the data user, however, the language as written may present confusion for laboratory professionals. It is understood the use of LCS/LCSD pairs is regularly included in batch analyses and benefits all data users whose samples are analyzed in the same analytical batch. Therefore, reference to "ONLY if no MD", will be replaced with "if analyzed". This language will allow laboratories to choose to employ either a matrix duplicate (MD) or an LCSD to demonstrate precision within an analytical batch.

DEEP is making the following revision to RCP 7470/7471:

Table 1A, LCSD, Required Deliverable, Yes (if analyzed)

Section Reference: Table 1A – Matrix Spike, Required Performance Standard, Item 3 **and** Matrix Spike Duplicate

RCP Language: *"(3) Percent recovery for mercury must be 75-125%."* ***and*** *No Matrix Spike Duplicate Row*

Comment: The MS/MSD acceptance criteria should be included for both aqueous and solid matrices as the method requirements differ between 7470 and 7471. The percent recovery ranges for solid samples (Method 7471B) is 80-120% while the range for aqueous samples (Method 7470A) is 75-125%. Table 1A as written only reflects the recovery range for aqueous samples.

Response:

DEEP acknowledges recovery range included in Table 1A does not accurately reflect the recovery ranges required for the specific matrices. The recovery ranges will be clarified and a new row for the Matrix Spike Duplicate will be included into Table 1A. Performance criteria, such as the RPD, will be included based on details from the Methods.

DEEP is making the following revision to RCP 7470/7471:

Table 1A, Matrix Spike, Item 3: Percent recoveries for Hg in solid samples must be 80-120% and aqueous samples must be 75-125%.

Table 1A, Matrix Spike Duplicate row.

RCP 7196 – Hexavalent Chromium

Section Reference: Table 1A - Initial Calibration, Required Performance Standard, Item 1

RCP Language: "*(1) Frequency: Daily prior to sample analysis."*

Comment: Request justification for daily calibration requirement. [Commenter's] laboratory data has demonstrated a stable calibration curve for as many as six months.

Response:
The requirement of a daily calibration is original to the inception of the RCPs in 2006. It is understood the color reagent used in the method, diphenylcarbazide, is relatively unstable and will degrade within a week even when properly stored in an amber container in a dark, temperature-controlled environment. Since this method is colorimetric it is important that the color reagent is not adding any background color that can affect the accuracy of measurements obtained by the spectrophotometer. It is also general analytical practice that at any time a new reagent is prepared, all standards should be prepared to ensure adequate matrix matching prior to recalibration. To ensure high quality data, DEEP maintains daily calibration is the preferred practice. DEEP has the authority to maintain conservative approaches to ensure data quality objectives for intended uses of data that are tantamount to protecting human health and the environment.

No revisions will be made in response to this comment.

Section Reference: Table 1A - Initial Calibration Verification and Continuing Calibration Verification, Required Performance Standard, Item 4

RCP Language: "*(4) Percent recovery must be 85-115%."*

Comment: Request justification for percent recovery range change from ±20% to ±15%. Suggested maintaining the ±20% recovery range.

Response:
The recovery criteria range was updated to meet the standards of EPA Method 7196A which defines the acceptance criteria range as 85-115%.

No revisions will be made in response to this comment.

Section Reference: Section 1.2.1 Sample Digestion/Preparation Methods

RCP Language: "*Aqueous samples for analysis by SW846 Method 7196 do not require preparation/digestion prior to analysis, whereas solid samples (soil, sediment, sludge, waste) must be prepared/digested using SW-846 Method 3060, an alkaline digestion procedure for extracting Cr(VI) from solid samples. Alkaline digestion is the required preparative step for the analysis of soils, sediments, and sludges and similar waste material. The pH of the digestate must be carefully adjusted and monitored during the alkaline digestion to maintain the native chromium species in the environmental sample (i.e., prevent transformation of one species of chromium to another species). During sample digestion using SW-846 Method 3060, the pH must be maintained at pH 7.5 ± 0.5 to obtain a valid extract for analysis by SW-846 Method 7196."*

Comment: In the SW846 method 3060A that is referenced for solid sample digestion, the sample pH is required to be 11.5 or greater. The pH requirement of 7.5 should be removed and changed to the appropriate requirement in the 3060A method.

Response:
The pH of 7.5 referenced in the RCP language is derived from Section 7.7 of Method 3060A that states *"…Adjust the pH of the solution to 7.5 ± 0.5 if the sample is to be analyzed using Method 7196 (adjust the pH accordingly if an alternate analytical method is to be used; i.e., 9.0 ± 0.5 if Method 7199 is to be used) and monitor the pH with a pH meter. If the pH of the digest should deviate from the desired range, discard the solution and redigest."*

The commenters reference to pH of 11 is assumed to derive from Section 5.7 of Method 3060A which states *"…The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater, if not, discard."* This section is related to the digestion reagent solution itself that must be added to the sample.

The RCP language as written reflects the language in EPA Method 7196.

No revisions will be made in response to this comment.

RCP TO-13 – PAHs in Air

Section Reference: 1.2 Summary of Method TO-13

RCP Language: *"The extract is concentrated by Kuderna-Danish (K-D) evaporator, followed by silica gel cleanup using column chromatography to remove potential interferences prior to analysis by GC/MS."*

Comment: Add allowances for equivalent concentration hardware; some labs do not use Kuderna-Danish (K-D) evaporators for concentration.

Response:

DEEP acknowledges the importance of allowing flexibility with laboratory equipment and resources. It is understood the use of QC samples will demonstrate the efficacy of the alternative equipment when achieving DQOs.

DEEP is making the following revisions to RCP TO-13:

The extract is concentrated by Kuderna-Danish (K-D) evaporator, or equivalent concentration hardware, followed by silica gel cleanup using column chromatography to remove potential interferences prior to analysis by GC/MS.

Section Reference: 1.2 Summary of Method TO-13

RCP Language: *"The analytical system is verified to be operating properly and calibrated using a five -point calibration."*

Comment: Suggest adding "…at a minimum a five-point…" language.

Response:
DEEP acknowledges the RCP language will benefit from permitting laboratories the flexibility to include more points in the calibration curve to improve correlation coefficients and detection accuracy.

DEEP is making the following revisions to RCP TO-13:

The analytical system is verified to be operating properly and calibrated using, at a minimum, a five -point calibration.

Section Reference: 1.2 Summary of Method TO-13

RCP Language: *"A preliminary analysis of the sample extract is performed to check the system performance and to ensure that the samples are within the calibration range of the instrument. If the preliminary analysis indicates nonperformance, then recalibrate the instrument, adjust the amount of the sample injected, adjust the calibration solution concentration, and adjust the data processing system to reflect observed retention times, etc."*

Comment: This language could use clarification. It is unclear if the language is referring to a CCV, dilution, or calibration non-conformances.

Response:
DEEP acknowledges the RCP language will benefit from clarification. The language was original to Method TO-13 which has not been updated since 1998. Given the age of the origin of the language, it is assumed the "sample extract" in this language is in reference to the current day CCV.

DEEP is making the following revisions to RCP TO-13:

A preliminary analysis of the continuing calibration verification (“CCV”) is performed to check the system performance. If the preliminary CCV analysis fails to meet acceptance criteria, then take corrective measures and recalibrate the instrument.

Section Reference: 1.2 Summary of Method TO-13

RCP Language: *“The samples and the blanks are analyzed and used (along with the amount of air sampled) to calculate the concentration of PAHs in the air sample.”*

Comment: Suggest adjusting “The samples and blanks…” language to “The extracts…” to ensure analysts are treating all calibration standards, QC samples, blanks, and field samples the same.

Response:
DEEP acknowledges the suggested language revision will provide clarity that each extract prepared by the laboratory analyst must follow the same preparation and analytical procedures.

DEEP is making the following revisions to RCP TO-13:

The extracts are analyzed and used (along with the amount of air sampled) to calculate the concentration of PAHs in the air sample.

Section Reference: 1.5 Sampling and Analysis

RCP Language: *“A "chain-of-custody" should be completed by the environmental professional (“EP”) when submitting samples to the laboratory.”*

Comment: Suggest making the “chain-of-custody” a requirement; therefore, change “should” to “must”.

Response:
DEEP agrees that chain-of-custody forms are imperative to ensuring adequate documentation of sample collection and delivery to the laboratory which is necessary for laboratory analysts.

DEEP is making the following revisions to RCP TO-13:

A "chain-of-custody" must be completed by the environmental professional (“EP”) when submitting samples to the laboratory.

Section Reference: 1.5 Sampling and Analysis

RCP Language: *"The use of the silica gel column cleanup is optional. However, if interferences are present, the extract must be put through the silica gel cleanup as described in the Sample Extraction, Concentration, and Cleanup Section 12.3 of EPA Method TO-13."*

Comment: The method does not dictate that cleanup must be used if interferences are present because it is not possible to know if interferences are present prior to analysis. Therefore, suggest revision to the language that clarifies that the use of silica gel cleanup is optional. However, if silica gel cleanup is utilized, the analyst must process the extracts using the procedure described in the Sample Extraction, Concentration, and Cleanup Section of EPA Method TO-13.

Response:
DEEP acknowledges the RCP language as written conflicts with the language in Method TO-13. The language will be revised to agree with Method TO-13.

DEEP is making the following revisions to RCP TO-13:

The use of the silica gel column cleanup is optional. However, if silica gel cleanup is used, the extract must be put through the silica gel cleanup as described in the Sample Extraction, Concentration, and Cleanup Section 12.3 of EPA Method TO-13.

Section Reference: 1.6 Quality Control Requirements for Method TO-13

RCP Language: *"Sample dilution or lower sample volume will also cause the RLs/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet Remediation Standard Regulation ("RSR") criteria. To meet the reporting limits, it may be necessary to modify the analytical method such as the use of SIM, an ion trap mass spectrometer, or other instrumentation of improved design to improve sensitivity. In such cases, the modifications must be described in the laboratory report narrative."*

Comment: The RCP language infers the employment of SIM analysis as a method modification. However, SIM may be a standard analysis for most laboratories. In other methods, SIM analysis is not referred to as a modification. Any method modification employed must be noted on the RCP certification form. Suggest removing this language because it does not accurately reflect the use of SIM analysis and may introduce confusion.

Response:
DEEP acknowledges this language was not original to the promulgation of this RCP and is assumed to be a clerical error.

DEEP is making the following revisions to RCP TO-13:

Sample dilution or lower sample volume will also cause the RLs/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet Remediation Standard Regulation ("RSR") criteria.

Section Reference: Table 1A, Laboratory Control Sample, Required Performance Standard, Item 2

RCP Language: *"(2) Standard source should be the same as initial calibration source."*

Comment: Typically, LCS standard sources are different than the standard source used for the calibration curve.

Response:
DEEP understands LCS QC samples are typically prepared with a "second" source, that is a standard source that is different than the original source used to prepare the calibration standards. Using a "second" source for the LCS allows the analyst to evaluate the accuracy of the calibration curve in relation to a sample that contains analytes from a different source. DEEP acknowledges the language in the RCP conflicts with this general practice. The language in Table 1A of 7470/7471 will be adjusted to allow second source standard to be used for the LCS.

DEEP is making the following revisions to RCP TO-13:

LCS, Required Performance Standard, Item 2: Standard source should be different from the initial calibration source

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Section Reference: Table 1A, Laboratory Control Sample Duplicate

RCP Language: *No applicable language.*

Comment: There is no mention of the use of a LCSD and/or if it is required. The use of a LCSD is typical laboratory practice to evaluating laboratory precision. If optional, or required, the use of an LCSD should be noted somewhere in the RCP or it should be included on this table.

Response:
Although not published in the original method, DEEP acknowledges the benefit of the evaluation of an LCSD for DQOs, therefore information for the LCSD will be added to Table 1A.

DEEP is making the following revisions to RCP TO-13:

Add LCSD row to Table 1A.

Section Reference: Table 1A, Internal Standard, Required Performance Standard, Item 3

RCP Language: *"(3) Recovery must be 50 – 100% compared to recent continuing calibration analysis."*

Comment: The recovery should be written as 50 – 200% rather than 50 – 100%.

Response:
DEEP acknowledges the recovery range in the RCP conflicts with the requirements under Method TO-13. The recovery range will be corrected to the suggested language.

DEEP is making the following revisions to RCP TO-13:

Internal Standard, Required Performance Standard, Item 3: "(3) Recovery must be 50 – 200% compared to recent continuing calibration analysis.

Section Reference: Table 1A, General Reporting Issues, Required Performance Standard

RCP Language: *All of Required Performance Standard*

Comment: There is not mention of reporting "J" flag results (i.e., concentrations detected <RL/LLOQ). Should there be allowance for reporting "J" flags.

Response:
DEEP remediation does not regulate target indoor air concentrations based on "J" flags, therefore only detections >RL/LLOQ should be reported, otherwise detections <RL /LLOQ should be reported as "ND".

No revisions will be made in response to this comment.

RCP TO-17 – VOCs in Air

Section Reference: Table 1A – Method Blank, Required Performance Standards, Item 1

RCP Language: *"(1) Analyze with every batch or ≤20 field samples, whichever is more frequent."*

Comment: Method blanks should also be analyzed before the samples and analyzed at the end of the analytical sequence to "close out" the sequence.

Response:
DEEP acknowledges the frequency defined in Table 1A does not agree with common laboratory practices which include running a method blank before and after analysis to indicate a clean analytical sequence.

DEEP is making the following revisions to RCP TO-17:

Table 1A, Method Blank, Required Performance Standard, Item 1: Analyze with every batch, or ≤20 field samples, whichever is more frequent, prior to sample analysis and at the end of the analytical sequence."

Section Reference: Table 1A – Laboratory Control Sample Duplicate

RCP Language: *No applicable language*

Comment: Some laboratories choose to run LCS/LCSD pairs as, often, most clients do not collect enough air volume to analyze a matrix duplicate. Please include an LCSD row in Table 1A to permit this substitute QC sample.

Response:
DEEP acknowledges that laboratories may choose to utilize LCS/LCSD QC samples to demonstrate analytical precision in lieu of a sample duplicate due to limited sample volume.

DEEP is making the following revisions to RCP TO-17:

Add LCSD row to Table 1A including acceptance criteria and corrective actions from Method TO-17.

Section Reference: Table 1A – Laboratory Control Sample, Required Performance Standard, Item 5

RCP Language: *"(5) Recovery must fall within 50-150%."*

Comment: Commenter noted their laboratory uses control chart limits rather than 50-150%.

Response:
DEEP prefers to maintain specified recovery criteria as a means of providing guidance to data users rather than utilizing laboratory defined limits to maintain conservative QC practices. However, if a laboratory can achieve tighter recovery criteria, that is acceptable.

No revisions will be made in response to this comment.

Section Reference: Section 1.9 Sample Collection, Storage and Holding Times, Table 4.0

RCP Language: Holding Time - *"30 days to analysis"*

Comment: While not necessarily standard, some laboratory may desorb samples into cans within 30 days of receipt and then analyze desorbed samples within 30 days.

Response:
DEEP acknowledges this desorption process may allow laboratories more flexibility with storing and analyzing air samples. The QA workgroup came to consensus to allow this additional 30 days of holding time as long as laboratories demonstrate Quality Control measures used to meet the Data Quality Objectives for the intended purpose of the data requested by the data user.

DEEP is making the following revisions to RCP TO-17:

Table 4.0, Holding Time: 30 days to desorb into can; Desorb to analysis 30 days

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