PUBLIC COMMENT SUMMARIES AND RESPONSES
UPDATE VI TO SW-846
PHASE II - METHODS 8260D AND 8270E
FOR VOLATILE AND SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

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INTRODUCTION

On April 28, 2017, the U.S. EPA published a notice to announce the availability of, and to request comment on SW-846 Update VI, Phase II – Methods 8260D and 8270E for Volatile and Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry.

EPA welcomed the public to submit comments on two revised analytical methods (8260D version 04/21/17 and 8270E version 04/20/17). The comment period was open from April 28, 2017 to June 28, 2017. The Agency has received public comment on Update VI, Phase II, and after consideration, is placing these new and revised methods, guidance, and chapters in the SW-846 methods compendium. EPA is issuing this update as guidance.

Seven groups of comments were received, which comprise sixty-five individual comments. As these comments were received in groups, they are labeled as comment #1(a), #1(b), etc. for each commenter, whose identities are removed in this document. Only the comments that pertain to each method are listed in each Methods section. Of these, thirty-nine comments pertained to Method 8260D only, twenty-five pertained to 8270E only and one comment pertained to both methods. This document provides draft summaries and responses to the public comments submitted to date regarding Methods 8260D and 8270E. Complete copies of the comments can be found in docket EPA-HQ-OLEM-2017-0133 at regulations.gov.

METHOD 8260D

Comment #1(E):
Commenter #1 made a comment on Method 8260D regarding blank contamination. The note here on forcing the calibration model through the origin for analytes that consistently show up in blanks is helpful. Another commenter expressed concern, but I would guess the intention behind this is the very reasonable judgment that it's not environmentally friendly for everyone to have to replace their HVAC systems and remodel their laboratory because of a little acetone from the preparation laboratory down the hall lingering in the air. If every blank has hits for PCE (per- or tetrachloroethene) and BTEX (benzene, toluene, ethylbenzene and xylenes) compounds that's a different story. It may be helpful to include language that makes a distinction between small, unavoidable, and insignificant hits of VOCs from known sources and those situations where reoccurring hits in a blank require corrective action.

Response #1(E):
The Agency thanks the commenter for their input. The following text has been added to Method 8260D, Sec. 9.5.2: "The analyst (or laboratory) should document detected common laboratory contaminants and distinguish those from situations (e.g., carryover), where corrective action may be required."

Comment #1(F):
Commenter #1 made a comment on Method 8260D regarding tune check requirements in 8260D, Sec. 11.4. Daily tune checks are no longer required. This is reasonable, because if the mass spectrometer (MS) is not tuned well enough to generate quality data, the problem will be indicated in the continuing calibration verification (CCV).

Response #1(F):
The Agency thanks the commenter for their input and concurs with the comment.

Comment #1(G):
The Commenter #1 made a comment on 8260D, Sec. 11.4.3.2. This section discusses reporting non-detects in field samples when more than 20% of CCV compounds are out of range. If it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. A reference to the discussion of a "sensitivity verification standard" in Method 8000 Sec. 11.7.1 and the acceptance criteria for it would be useful. Does the "applicable quantitation limit" refer to the established lower limit of quantitation (LLOQ) (Sec. 9.9), or does it refer to the data needed by the project? For example, when performing analysis for the purpose of determining whether a sample meets LDRs, then the lowest limit that needs to be reported would be in the mg/kg range, which is likely much higher than the LLOQ. Must the sensitivity check be at the LLOQ if the analyst chooses to report a non-detect value at an elevated reporting limit?

For example: A laboratory must determine if a sample contains methylethylketone (MEK) at the lower data reporting limit (36 mg/kg). The laboratory has established their lowest calibration standard, 2.5 µg/L, as their LLOQ for MEK. Five grams (g) of solid sample is dispersed in methanol per Method 5035. An aliquot is added to deionized (DI) water (a 1/1000 dilution) and added to a volatile organic analyte (VOA) vial, which goes onto a purge & trap system. A 25 µg/L CCV does not meet the criteria described in this section. The sample appears non-detect for MEK. Must a sensitivity check be run at 2.5 µg/L? Since the action limit is (after dilution) 36 µg/L, could the CCV itself be used as a "sensitivity check"? I don't advocate a position on this, but I think this could use some clarification.

Overall, the revisions seem to reflect a greater emphasis on establishing data quality objectives (DQOs) appropriate to particular projects. The more guidance on this, the better, as these methods are used in a wide variety of circumstances and for a wide variety of purposes. Though not a Method Defined Parameter (MDP), it's understandable that some in the environmental industry feel pressure to treat these methods as though they are, out of an abundance of caution toward whatever some regulatory body might someday decide about modifications they made in good faith. It becomes less about the scientifically defensible and more about not getting in trouble. So, to the degree (whatever that is) that the Agency wants to extend flexibility to analysts applying these methods to their own projects it pretty much has to be spelled out.

Response #1(G):
The Agency thanks the commenter for their input. The intent of the SW-846 is to be guidance for performance based environmental testing. The individual laboratory and data users must decide what LLOQ best meets their needs for use of the data. Some poorly performing compounds will have higher LLOQs (such as MEK) due to poor purge efficiency or overall lower stability or poor detector response. In these situations, the analyst must use professional judgment and set DQOs and LLOQs at reasonable levels for the project's needs. The project planning team and the laboratory should set the LLOQ at a reasonable level for the project's data use in the sampling analysis plan (SAP) and quality control (QC) documents. In the example provided, the CCV level could be used to confirm the non-detect at the reporting level.

Comment #2(A):
Commenter #2 commented on 8260D, Sec. 11.3.6. Sec. 11.3.6 should say something along the lines of "only after initial calibration (ICAL)." As it's currently worded it sounds like it is to be run prior to every 12-hour shift. A valid batch definition would be a great addition to this method. Specifically clarifying the time limits, as that's the most common disagreement in the field. The LLOQ section needs to be clarified especially since most laboratories will not have sufficient historical data for some time. Currently it states that the laboratory control standard (LCS) limits can be used plus or minus 20%. This is confusing because the LCS is not usually and probably should not be at the same concentration as the LLOQ.

Response #2(A):
The Agency thanks the commenter for their input. Sec. 11.3.6 actually discusses the initial calibration verification (ICV) standard, which is only required after the ICAL and from a separate source from the calibration standards. The LCS and LLOQ are not made using the same standard. The LLOQ is a low-level standard run at or near the limit of quantitation. The term LCS (or CCV for many volatile analyses) is usually used to refer to a mid-point standard of the same source as the ICAL. The ICV should be run after an ICAL and before any samples are analyzed.

Comment #2(B):
Commenter #2 commented on Method 8260D, Sec. 9.5.4. The commenter feels that this section could use some clarification. Analysis of method blanks (MB) should be sufficient as long as the source shows no prior problems. Following this logic, if a different lot was used mid-batch separate blanks would not need to be prepared.

Response #2(B):
The Agency thanks the commenter for their input. MBs are intended to be representative of the DI water and any reagents used in preparation of the samples and QC in a given batch. If the laboratory changes reagents mid-batch, an extra MB would be appropriate, but not

expressly required by the Method. The laboratories are always welcome to employ more stringent QC criteria than the method requires. The final sentence in Sec. 9.5.4 has been changed to the following: “However, if reagents are changed during a preparation batch, separate blanks should be prepared for each set of reagents.”

Comment #2(C):
Commenter #2 commented that in Method 8260D, Sec. 11.3.5.4, that the EPA's intention for having all calibration points reprocessed as opposed to some of them, would be helpful.

Response #2(C):
The Agency thanks the commenter for their input. Sec. 11.3.5.4 is referencing the refitting or % error calculation listed in Sec. 9 of Method 8000D as a check of the quantitative accuracy of the calibration curve. The refit check is recommended, not required as the Method uses the word “should” rather than “must”. The intention is to give the analyst an additional measure of quantitative accuracy for the data. Calculating the Relative Standard Error (RSE) in Sec. 9 of Method 8000D for all the calibration points gives the analyst the information for the entire calibration range.

Comment #2(D):
Commenter #2 commented that in Method 8260D, Sec. 11.4.2 should specify that a MB would be acceptable. It looks like that was the intention but it is somewhat unclear since there are many different kinds of blanks.

Response #2(D):
The Agency thanks the commenter for their input. No changes were made to this section. Sec. 11.4.2 does discuss the requirement to run a blank with criteria for acceptance referenced in Sec. 9.5. Alternate types of blanks besides the MB may also be used to demonstrate acceptability.

Comment #2(E):
Commenter #2 commented on Method 8260D, Sec. 9.6.2. The LCS section of the QC summary chart needs a few fixes. Ideally, instead of referring to Method 8000 and since this method supersedes it, it should state that the LCS acceptance criteria is based on in-house developed control charts or historical data. It's much more likely that an LCS is not similar to a CCV and even more so not identical, both phrases appear in those two sections. Having those options is acceptable but the words similar and identical should be taken out. Also, the LCS is prepared using sand and organic-free reagent water, however the CCV does not contain sand regardless of whether solid or liquid samples are being analyzed. If the EPA disagrees or has other intentions it should be clarified.

Response #2(E):
Method 8000D is the base method for chromatography in SW-846. Method 8260D may have more stringent criteria for some QC requirements, in which case the specific method criteria does supersede the base method. Method 8000D is still an excellent resource for basic QC practices for chromatography. Many laboratories will use the same run for the CCV and LCS as volatiles analysis using purge and trap does not involve additional preparatory steps. The ICV is used as the second source verification immediately following the calibration and before the analysis of samples. The laboratory may elect to run additional LCS standards from a second source as a part of batch QC, but this is not required. The text in Sec. 9.6.2 has been

updated to state, “The LCS for solid matrices may be prepared in clean sand or organic-free reagent water. However, the use of sand is not required.”

Comment 2(F):
Commenter #2 commented on Method 8260D that Appendix A should be fixed on Item 19 to "were updated to be 508 compliant."

Response 2(F):
The Agency agrees with the commenter. The wording was updated in Appendix A, #19 to state, “A table of contents was added and all graphics and tables in this method were updated to be 508 compliant.”

Comment #3:
Commenter #3 commented on Methods 8260D and 8270E, saying they appreciated the opportunity to provide comments on the proposed methods. Commenter #3 agrees with all proposed changes to these methods and commends EPA for making the following improvements:

- Allowing the use of more modern technology which has become available since the last publication of these methods to facilitate more flexibility and sensitivity.
- Revising tune verification procedures to reduce frequency for increased analytical efficiency.
- Adding language to make minimum Response Factors (RFs) in Table 4 (Guidance Response Factor Criteria for Initial Calibration) guidance only rather than requirements to more accurately represent the intent of this table.

Response #3:
The Agency thanks the commenter for their input.

Comment #4(A):
Commenter #4 commented on Method 8260D, Sec. 9.9.1.2, which discusses LLOQ requirements. Commenter #4 states, in our opinion, the allowance to verify the LLOQ on a particular instrument only once every three years is a bit minimal. Suggest that the LLOQ should be verified at least once per year on each instrument.

Response #4(A):
The Agency thanks the commenter for their input. The method requirements are a minimum level of acceptable QC. Individual laboratories and data users are welcome to use more stringent criteria where needed.

Comment #4(B):
Commenter #4 commented on Method 8260D, regarding the note in Sec. 11.3.12. Commenter #4 states, in our opinion, bromofluorobenzene (BFB) analysis is not useful for selected ion monitoring (SIM) analysis (since it is not performed in SIM mode). There should be a requirement for demonstrating mass accuracy and resolution with polyfluorotetrabutylalcohol (PFTBA), similar to the requirements for tandem MS and chemical impact (CI). We suggest merging the SIM requirement with the second note.

Suggested text: BFB tune checks are not appropriate for SIM analysis, CI analysis or tandem MS analysis using selected reaction monitoring (SRM). However, the laboratory must demonstrate, prior to the ICAL, that the MS system achieves mass accuracy and mass resolution criteria specified by the instrument manufacturer for the PFTBA internal calibrant or another appropriate chemical.

Response #4(B):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and the following language has been added to the notes in Sec. 11.3.1.2: "NOTE: All subsequent standards, field samples, and QC samples associated with this analysis must use identical MS instrument conditions with the exception of SIM analysis. BFB may be analyzed in full scan mode while standards, samples, and QC are analyzed in SIM. As an alternative to BFB for SIM analysis, the laboratory may also use an alternative detector verification, such as PFTBA, or the manufacturer's recommended detector check.

NOTE: BFB tune checks are not appropriate for CI or tandem MS analysis using SRM. However, the laboratory must demonstrate, prior to the ICAL, that the MS system achieves mass accuracy and mass resolution criteria specified by the instrument manufacturer for the PFTBA internal calibrant or another appropriate chemical."

Comment #4(C):
Commenter #4 commented on Method 8260D, Sec. 11.4.3.2 (second part of the section): "In these cases, the affected target analytes may still be reported as non-detects in field samples if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit." Commenter #4 states that this is a valuable and sensible allowance, but it would be useful to include some direction on how adequate sensitivity is to be demonstrated.

Suggested text:
In these cases, the affected target analytes may still be reported as non-detects in field samples if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. Adequate sensitivity may be demonstrated by including analysis of a standard spiked at or below the LLOQ in the analytical batch. Sufficient sensitivity is demonstrated for analytes that meet all applicable qualitative identification criteria.

Response #4(C):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and the following language now appears in Sec. 11.3.5.2: "If more than 10% of the compounds included with the ICAL (or more than 10% of those that will be reported) exceed the 20% RSD limit and do not meet the minimum correlation criteria ($r^2 \geq 0.99$ or relative standard error (RSE) ≤20%) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Correct the source of the problem; then repeat the calibration procedure beginning with Sec. 11.3. If compounds fail to meet these criteria, the associated concentrations may still be determined but they must be reported as estimated. In order to report non-detects, it must be demonstrated that there is sufficient accuracy to detect the failed compounds at the applicable LLOQ (see Secs. 11.3.5.4 for refitting standards and 11.4.3.2 for CCV). Refer to Method 8000 for further discussion of RSE. Example RSE calculations can be found in Reference 16."

Comment #4(D):
Commenter #4 commented on Method 8260D, Sec. 11.5.3 stating that several references are made to using "the same clean control material used for calibration standards" with a reference to Ottawa sand. Use of Ottawa sand as an artificial matrix for "soil" calibration standards, QC samples, and blanks is unnecessary and potentially counterproductive as Ottawa sand would not be added to samples during the course of analysis and any background associated with sand would be irrelevant with respect to sample detections.

Suggested Text:
Replace references to “clean control material” with “aliquots of VOA free water”.

Responses #4(D):
The Agency thanks the commenter for their input. Sec. 11.5.3 has updated as follows: “See Secs. 7.12, 9.62 and Method 8000 for more guidance on the selection and preparation of the matrix spike and the LCS. The LCS for solid matrices may be prepared in clean sand or organic-free reagent water. However, the use of sand is not required.”

Comment #4(E):
Commenter #4 commented on Method 8260D, Sec. 11.6.1.2. We have not observed this to be a problem and would consider a retention time (RT) shift of >10 seconds relative to the CCV to be an indication of a malfunctioning instrument.

Suggested text: Remove the note.

Response #4(E):
The Agency thanks the commenter for their input. The note in Sec. 11.6.1.2 is not a requirement, merely an acknowledgment that some analyte’s RT may be affected by matrix interferences, which could shift RT forward. If a laboratory noticed significant RT shifts, they should investigate the cause and take appropriate corrective action.

Comment #4(F):
Commenter #4 commented on Method 8260D, Sec. 11.6.1.2, that ±10 seconds is a really wide window.

Suggested text:
The RT should be within ±2 seconds of the RT for this analyte in the CCV run at the beginning of the 12-hour period (delta RT 0.034 minute) or within ±2 seconds relative to the shift of the associated internal standard (IS) (delta RT of the IS ± 10 seconds).

Responses #4(F):
The Agency thanks the commenter for their input. The method requirements are a minimum level of acceptable QC. Individual laboratories and data users are welcome to use more stringent criteria where needed.

Comment #4(G):
Commenter #4 commented on Method 8260D, Sec. 11.6.1.3. There are two stages to identification of a target analyte. The first is a determination of whether three (typically) characteristic ions are present and their responses maximize at the same time. The second is comparison with a reference spectrum which contains the full spectrum of the compound, not just the few ions more than 30% of the base peak. These two stages are confused in the existing text. This has unfortunate consequences, including assessors demanding that the second stage be conducted with spectra generated on the instrument, rather than from a National Institute of Standards and Technology (NIST) library. There are several problems with using instrument generated spectra for the second part, including (i) if an identification mistake is made, then the reference spectra is updated to reflect the mistake, making problems difficult to identify (ii) if there are any coelutions in the calibration standards, then the reference spectra generated from these standards may look very different from the spectrum obtained in a field

sample. This is especially an issue for the volatile oxygenates included in 8260 that have much lower RFs than some other analytes.

Suggested text:
Remove the requirement for a library spectrum generated from a standard (rather than NIST). Note that this change was made in 8270E but not in this method, 8260D, and that Appendix A implies that the change was intended to be made for this method.
Suggested revised 11.6.1.3: The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%. Use professional judgment in interpretation where interferences are observed. Qualitative identification of sample mass spectra not acquired in limited ion acquisition modes (i.e., SIM or selected reaction monitoring (SRM)) may also be supported by comparison to a reference library as described in Sec. 11.6.2.

Response #4(G):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and the language was revised to clarify that the ±30% criteria applies to qualifier ions and to indicate a preference for reference spectra used for expected qualifier ion ratios to be generated under precisely the same set of operating conditions used for analysis of unknowns. The Agency acknowledges that mass spectrometer settings can be standardized in order to make ion ratios comparable across instruments (including those found in a commercial library spectra), but EPA did not build appropriate controls into these methods to do so. Different mass spectrometer designs may have different limitations with regard to how adjustable the abundances of lower mass ions are relative to higher mass ions. The edits to the qualitative identification section were intended to make clear that reference spectra were not required to be generated on the same system, but it was the Work Group's consensus opinion that this is the most universal approach.

Comment #4(H):
Commenter #4 commented on Method 8260D, Sec. 11.6.1.4. The CCV level is probably the same or at least close to the mid-level, so we are not sure what is gained by this text.

Suggested text:
Unresolved structural isomers with similar mass spectra are identified as isomeric pairs. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is ≤ 50% of the average of the two peak heights, or ([valley height]/ [average peak height] is ≥50%). At a minimum, the resolution should be verified on the mid-point concentration of the ICAL.

Response #4(H):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and the following language was added to Sec. 11.6.1.4: "It is important to check the separation of structural isomers in the ICV and the daily CCV check standards to verify if the instrument performance is adequate regarding separation of compounds of interest which are structural isomers."

Comment #4(I):
Commenter #4 commented on Method 8260D, Reference 16. This document (R. Burrows, Basic RSE calculator v2 and instructions, December 2016) can be obtained from The

National Environmental Laboratory Accreditation Conference (NELAC) Institute (TNI) web site.

Suggested text:
Reference 16, R. Burrows, Basic RSE calculator v2 and instructions, December 2016
Available at: http://nelac-institute.org/docs/comm/emmec/Calculating%20RSE.pdf

Response #4(I):
The Agency agrees with the commenter and has made the change in the final version. The referenced spreadsheet can currently be found in the docket on regulations.gov.

Comment #5(B):
Commenter #5 commented on Method 8260D, Sec. 7.10. BFB tune verification standard is missing guidance on the quantity of BFB to use.

Response 5(B):
The recommended introduction amount is 50 nanograms (ng). Sec. 11.3.1.1 of Method 8260D states, "In the absence of other recommendations on how to acquire the mass spectrum of BFB, the following approach may be used: Introduce BFB with the same technique to be used for analysis of calibration standards and samples. Scale the mass of BFB introduced to prevent high abundance masses from saturating the detector (e.g., ≤50 ng)."

Comment #5(C):
Commenter #5 commented on Method 8260D, Section 9.4. Initial demonstration of proficiency (IDP) refers to Section 9.3 of Method 8000. Method 8000 says to compare mean and standard deviation (SD) to the single laboratory limits at the end of the determinative method. The performance data have now been separated from the method and archived separately. What is EPA's intention for these method performance criteria tables? Will they continue to be separate from the body of the method? Will they be dropped from the new method versions? If they are dropped, what criteria should be used to evaluate laboratory method performance?

Response #5(C):
The Agency strongly suggests that the laboratories develop their own statistical limits for acceptable recovery. The historical performance data and tables previously published in earlier methods have been removed and are available at: http://www.epa.gov/hw-sw846/validated-test-method-8260d-volatile-organic-compounds-gas-chromatographymass-spectrometry (see Sec. 13 of Method 8260D). The user can find additional information in Chapter One of SW-846 and in Method 8000D. As a general rule, the acceptance limits are set at ±3 SD of the mean recovery for that analyte by the laboratory's standard operating procedure (SOP).

Comment #5 (D):
Commenter #5 commented on Method 8260D, Sec. 11.3.1.2. This section says, "Compare BFB mass intensities to the criteria in Table 3. Alternatively, other documented ion ratio criteria may be used provided that method performance is not adversely affected…use other documented criteria provided they are used consistently throughout the ICAL, calibration verification, and sample analyses." These statements should be modified to say "the current approved version of documented criteria". This language needs to be clarified based on what has been observed in laboratory audits. Some laboratories are searching the Internet to come up with the widest possible BFB ranges and are using draft or out of date sources. While it

may be fine to allow some flexibility in BFB acceptance criteria, the source for the acceptance ranges should be currently approved by EPA.

Response #5(D):
The Agency thanks the commenter for their input. The Agency always encourages users to use the most up to date version of any method or guidance as that will contain the best information on current best practices.

Comment #5(E):
Commenter #5 commented on Method 8260D, Sec. 11.3.5.1. The section states, "Alternatively, the affected target analytes may be reported with an appropriate data qualifier". The initial calibration relative standard deviation (RSD) has already been widened from <15% in 8260B to <20% for using an average RF. If laboratories are allowed to report data based on calibration curves that fail the average RF criteria, as well as, the other types of calibration fits described in Method 8000 simply by using a qualifier, there is no incentive for them to ever stop and recalibrate or do needed instrument maintenance. While subsequent paragraphs do offer the guidance that less than 10% of analytes should be allowed to fail and they should not be critical to the project, that places a lot of discretion on the analyst. Many environmental laboratories spend very little time and effort on training analysts and focus on production only. With the widening of the %RSD limit and all of the curve choices available, all compounds must be able to meet some sort of standard or the data will be useless for decision making.

Response #5(E):
The Agency thanks the commenter for their input. The SW-846 Work Group felt that the listed acceptance criteria represented reasonable and realistically achievable requirements by laboratories. The allowance of reporting flagged data for compounds that do not pass QC criteria is not mandatory. If the laboratory's or project's DQOs require that data be within more stringent criteria, they may do so and perform whatever corrective action is appropriate when compounds are outside acceptance. The allowance of 10% of compounds to be outside criteria is in recognition of the large available list of target analytes that may be analyzed (some of which are very poor responders) by this method and the statistical unlikeliness of having 100% of a large list of compounds within criteria all of the time.

Comment #5(F):
Commenter #5 commented on Method 8260D, Sec. 11.4. This section states, "Tune checks (Sec. 11.3.1) are only required prior to ICAL." Tune checks provide important information about the continued system suitability. It provides resolution and abundance information in a way the CCV does not. Since BFB can either be introduced by a quick direct injection or as part of the CCV, there may be little, if any run time added for this QC check. Please do not drop the 12-hour tune check requirement.

Response #5(F):
The Agency thanks the commenter for their input. The MS tune check defines the MS as working properly prior to the ICAL. If the CCVs are still within criteria, that sufficiently demonstrates that the detector is still operating within control as determined by this method and the laboratory's SOP. This is also consistent with other EPA test methods. Laboratories or data users that wish to use more stringent criteria, such as requiring tune check each 12-hour clock cycle, are welcome to do so.

Comment #5(G):
Commenter #5 commented on Method 8260D, Sec. 11.4.3. This section allows CCV standard criteria of up to 20% of the target compounds failing without the analyst taking any action. While there may be some failures in CCV criteria when there are a large number of compounds, 20% is too high. There is no distinction made between analytes that fail high vs

fail low. There is no mention of analytes critical to a project. It just says to qualify the results when a positive hit is detected in field samples. What about when the analytes are low in the CCV? This may have implications for an associated non-detected analyte. The same analytes should also not continue to fail on subsequent CCVs. Many laboratories will continue to ignore the same compound failing repeatedly which is an indication that something is wrong.

Response #5(G):
The Agency thanks the commenter for their input. The language in 11.4.3.2 has been changed to state, "If the %D or percent drift for a compound is ≤20%, then the ICAL for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. The analyst should strive to place more emphasis on meeting the CCV criteria for those compounds that are critical to the project. If the criterion is not met (i.e., greater than ±20%D or drift) for more than 20% of the compounds included in the ICAL (or more than 20% of those that will be reported), then corrective action must be taken prior to the analysis of samples. Target analytes that do not meet the CCV criteria and are reported in the associated samples must be qualified to indicate the reported concentrations are potentially estimated or biased values. In cases where compounds fail low, they may be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the LLOQ or project specific level of interest (e.g., by calibrating below the established LLOQ to confirm the non-detect, or by analyzing a standard near that level to confirm the analyte could be qualitatively identified if it were present [See Sec. 11.7 of Method 8000]). Alternatively, the non-detect could be qualified or the LLOQ raised to a higher level. In cases where compounds fail high in the CCV and are not found in the associated field samples, they may be reported without qualification."

Comment #5(H):
Commenter #5 commented on Method 8260D, Sec. 11.4.4. The commenter commented that thirty seconds seems like a lot since everyone is using capillary columns. 8260C says 10 seconds. That is more realistic since volatiles analysts seldom clip columns.

Response #5(H):
The Agency thanks the commenter for their input. The stated window is an acknowledgment that some sample's RT may be affected by matrix interferences which could shift RT forward. If a laboratory noticed significant RT shifts, they should investigate the cause and take appropriate corrective action. Retention time changes will depend on the type of chromatographic system used.

Comment #5(I):
Commenter #5 commented on Method 8260D, Sec. 11.4.5. Sec. 11.4.5 defines a factor of 2 as 50-200% and Sec. 11.5.6 defines a factor of two as -50% to +100%. This is not consistent.

Response #5(I):
The Agency thanks the commenter for their input. While the two terms are considered equivalent, the language has been changed throughout Method 8260D to say 50 - 200% for IS recovery to avoid confusion.

Comment #5 (J):
Commenter #5 commented on Method 8260D, Table 3. The relative abundance criteria for BFB ions 50 and 75 must be added back into the table. They provide important tuning information concerning the instrument's ability to see ions with m/z of less than 95 which comprise 73 of the 114 compounds in Table 1, or 64%.

Response #5(J):
The Agency thanks the commenter for their input. The current BFB requirements are updated using accepted method criteria from the SW-846 Work Group and are consistent with other EPA test methods.

Comment #5(K):
Commenter #5 commented on Method 8260D, Table 7. Table 7 states that "a duplicate and matrix spike, or matrix spike/matrix spike duplicate per preparation of 20 or fewer samples (not required per batch)." Since a batch is typically defined as 20 samples or less prepared and analyzed on the same day, please clarify the statement in parentheses.

Response #5(K):
The Agency thanks the commenter for their input. The intention of Table 7 is as written, that a set of sample duplicates and/or matrix spike duplicates are required per 20 samples (or fewer) where adequate sample volume exists to do so, but not per analytical batch.

Comment #6(A):
Commenter #6 commented on Method 8260D, page 1 (disclaimer, paragraph 2). The following language is included in the disclaimer: "The performance data referenced in this method are for guidance purposes only and are not intended to be and must not be used as absolute quality control (QC) acceptance criteria for purposes of laboratory accreditation." The term "must not be used" should be removed as this should be a state decision on QC required for laboratory accreditation or specific state projects.

Response #6(A):
The Agency thanks the commenter for their input. The SW-846 methods are guidance and are performance based. The laboratories and data users should determine their project's DQOs and requirements. If they wish to use more stringent criteria for their project's needs, they are welcome to do so. Those requirements can be stipulated in the project's QC documents.

Comment #6(B):
Commenter #6 commented on Method 8260D, Analyte Table Sec. 1 and Appendix A. There is an inconsistency between the revision 2, Appendix A and the analyte table in Section 1. The revision states, "Trichlorotrifluoroethane was split into two isomers: 1,1,2-Trichlorotrifluoroethane and 1,1,1-Trichlorotrifluoroethane." The table in Section 1 lists: 1,1,2-Trichloro-1,2,2-Trifluoroethane and 1,1,1-Trichlorotrifluoroethane. Either the revision history or the table should be updated for consistency.

Response #6(B):
The Agency thanks the commenter for their input. The currently released version of 8260D is Revision 4, dated June 2017. The Agency agrees with the commenter that the nomenclature is incorrect in Table 1. The reported compounds should be: 1,1,1-Trichlorotrifluoroethane and 1,1,2-Trichlorotrifluoroethane. Table 1 has been corrected. The compounds were listed correctly in Appendix A.

Comment #6(C):
Commenter #6 commented on Method 8260D, Sec. 7.6: The six-month holding time for stock standards (once opened) was removed. However, an expiration date was not specified. An expiration date for stock standards should be specified to provide this guidance to the laboratory and to prevent the use of degraded standards.

Response #6(C):
The Agency thanks the commenter for their input. The Agency recommends that the laboratory follow the manufacturer's recommendations on standard expiration. The following section has been added to Method 8260D, Sec. 7.6.1: "Certified solutions purchased from a vendor must be replaced per the manufacturer's recommended expiration date. Stock standard solutions prepared in-house must be replaced after one year, or sooner if comparison with QC check samples indicates a problem. When solutions are mixed together, regardless of the source, they must be replaced after the manufacturer's expiration date or one year (whichever occurs first) or sooner if problems are indicated. The assigned expiration date of the mixed standard should correspond to that of the stock that expires the earliest."

Comment #6(D):
Commenter #6 commented on Method 8260D, Sec. 7.11.3. This section includes a statement, "The standard should contain all calibrated target analytes that will be reported for the project, if readily available." Sections 9.3.2 and 11.3.6 also include the term "if readily available". The term "if readily available" needs to be removed or add additional language to explain what the language "if readily available" means.

Response #6(D):
The Agency thanks the commenter for their input. This method is used for a wide variety of analyses. As such, many standards are commercially available for common analyte lists. The term "if readily available" is meant to address analytes for which a second source standard cannot be easily found from a commercial supplier. The intention of the method is to have a full-list ICV standard, if possible, while addressing the possibility that some, less common analytes may not have more than one source available. The best practice is to have a full list ICV to verify the calibration source. The following text has been added to Sec. 7.11.3: "A second lot number from the same manufacturer may be adequate to meet this requirement."

Comment #6(E):
Commenter #6 commented on Method 8260D, Sec. 11.3.4.1. There is language included in Section 11.3.4.1 of EPA 8270D that states, "Meeting the minimum RF criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity." This language should be added to Section 11.3.4.2, Page 22 of EPA 8260D since this discourages laboratories from attempting to calibrate lower on instruments that cannot meet the desired sensitivity.

Response #6(E):
The Agency thanks the commenter for their input but disagrees with the commenter. The commenter is referencing the previous version of the Method (8270D). The current Method 8270E now states in Sec. 11.3.4.2: "The RSD should be ≤20% for each target analyte (see Sec. 11.3.5). 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. The laboratory should establish procedures in its SOP (e.g., laboratory established minimum RFs, signal-to-noise (S/N) checks, etc.) to ensure that the instrument is working properly and that calibration standards were correctly prepared." Method 8260D has similar language to the new 8270E for the same reason. Additional detail on this topic can be found in Method 8260D, Secs. 11.3.5.4 and 11.6.1.

Comment #6(F):
Commenter #6 commented on Method 8260D, regarding the second note in Sec. 11.3.5.1. This section states, "NOTE: Forcing the calibration model through the origin (for analytes that are consistently detected in the laboratory reagent blanks) allows for a better estimate of the background level of blank contaminants. An accurate estimate of background contamination is necessary to set method reporting limits for method analytes when blank levels are problematic." The analyst should not alter the calibration model to compensate for laboratory contamination. The source of the contamination should be eliminated from the analytical process. We recommend this note be removed from the method.

Response #6(F):
The Agency thanks the commenter for their input. The intention of this note is for the laboratories to see what contamination is present, not to mask it. If low level contamination is present and the calibration model is not forced through zero, the intercept can mask low level values by "calibrating it out". The Agency strongly encourages laboratories to find ways to lower the levels of contamination in their process or raise the LLOQ to an appropriately higher level. The use of any calibration model listed is permitted, but the method does not require use for any specific analyte. This practice is used in other EPA test methods including the volatile drinking water Method, 524.4 (reference found in Sec. 10.1.9). It is up to the laboratory and the data user to determine what reporting limits and contamination mean to their data's use.

Comment #6(G):
Commenter #6 commented on Method 8260D, Sec. 11.3.5.3. This section states, "In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable LLOQ." All target analytes in the ICAL should pass in order to generate reportable data, even non-detects, to prevent the possibility of reporting false negatives. Additional language should be added to this section requiring the laboratory to verify with the regulatory authority the contaminants of concern for a particular project to ensure that valid calibration data is obtained.

Response #6(G):
The Agency thanks the commenter for their input. The following language now appears in Sec. 11.3.5.2: "If more than 10% of the compounds included with the ICAL (or more than 10% of those that will be reported) exceed the 20% RSD limit and do not meet the minimum correlation criteria ($r^2 \geq 0.99$ or relative standard error (RSE) ≤20%) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Correct the source of the problem; then repeat the calibration procedure beginning with Sec. 11.3. If compounds fail to meet these criteria, the associated concentrations may still be determined but they must be reported as estimated. In order to report non-detects, it must be demonstrated that there is sufficient accuracy to detect the failed compounds at the applicable LLOQ (see Secs. 11.3.5.4 for refitting standards and 11.4.3.2 for CCV). Refer to Method 8000 for further discussion of RSE. Example RSE calculations can be found in Reference 16." The Method does not require that 100% compounds pass criteria in order to report data. It allows for the reporting of flagged data when certain conditions and limits are met. If a data user, including a State Regulatory Agency (SRA), wishes to use more stringent QC criteria they are welcome to do so, but would have to specify those criteria in their DQOs and project documents.

Comment #6(H):
Commenter #6 commented on Method 8260D, Sec. 11.4. This section states, "NOTE: Tune checks (Sec. 11.3.1) are only required prior to ICAL." The significance of removing this requirement was not included in the revision history. We would like clarification as to why the

daily (every 12 hours) BFB tune check requirement was removed from the method. The tune requirement is still included in the approved methods for drinking water and wastewater. We suggest the daily tune requirement be added back to the method to provide added QC for the instrument.

Response #6(H):
The Agency thanks the commenter for their input. This practice is used in other EPA test methods including the volatile drinking water Method, 524.4 (reference found in Sec. 9.3.4). The MS tune check defines the MS as working properly prior to the ICAL. If the CCVs are still within criteria, that sufficiently demonstrates that the detector is still operating within control as determined by this method and the laboratory's SOP. Laboratories or data users, including SRAs that wish to use more stringent criteria, such as requiring tune checks each 12-hour clock cycle, are welcome to do so.

Comment #6(I):
Commenter #6 commented on Method 8260D, Sec. 11.3. This section allows the failure of 20% of the target analytes in the CCV. This section states, "the affected target analytes may still be reported as non-detects in field samples if it can be demonstrated that there was adequate sensitivity to detect the compounds at the applicable quantitation limit." Should "quantitation limit" be changed to "LLOQ"? The procedure should also document how adequate sensitivity is to be determined. Should this require the analysis of an LLOQ check standard for all target analytes to verify sensitivity?

Response #6(I)
The Agency thanks the commenter for their input. The intent of the SW-846 is to be guidance for performance based environmental testing. The term quantitation limit used here can mean LLOQ, but the user could specify some other limit in their DQOs. The individual laboratory and data users must decide what quantitation limit (LLOQ is recommended) best meets their needs for use of the data. Some poorly performing compounds will have higher LLOQs (such as MEK) due to poor purge efficiency, overall lower stability or poor detector response. In these situations, the analyst must use professional judgment and set DQOs and LLOQs at reasonable levels for the project's needs. If the data user needs to use more stringent QC criteria, they are welcome to do so in their DQOs and project documents. An LLOQ check standard can be a helpful tool in verifying that sensitivity is adequate.

Comment #7:
Commenter #7 commented on Method 8260D, Sec. 11.4.5 and Table 7. In 8260D, the commenter thought that it would be useful to express results of sludge samples in g/kg for standardization. The commenter also thought that the suggested upper acceptance criteria for ISs for CCVs was too large (Table 7 or point 11.4.5, "standard responses are within 50% to 200% of mid-point of ICAL").

Response #7:
The Agency thanks the commenter for their input. The IS acceptance criteria of 50-200% are standardized across many EPA methods in SW-846, as well as, the drinking water and wastewater methods and have been for many years. The laboratories are free to report samples in whatever units supports their DQOs and project's needs. If the data user needs to use more stringent QC criteria for ISs, they are welcome to do so in their DQOs and project documents.

METHOD 8270E

Comment #1(A):
Commenter #1 made a comment on Method 8270E, Section 11.6. The section on analyte identification might benefit from a more general statement about professional judgment in cases where the objective of the analysis is to determine whether the presence of a compound can be ruled out (i.e., to demonstrate that it meets lower detection limits), especially in difficult matrices. A failure to strictly meet all the criteria for positive identification shouldn't be taken to prove the constituent's absence, because in rare cases results may be more ambiguous and a conservative approach should be taken. I have encountered a different interpretation and it would be helpful to have the point clarified.

Response #1(A):
The Agency thanks the commenter for their input. The guidance in this method is intended to be general. More proscriptive restrictions on professional judgment are not provided because the acceptance criteria for a project should be defined in its DQOs and QC documents.

Comment #1(B):
Commenter # 1 commented on Method 8270E, Section 9.7. Additional guidance on the usefulness of surrogates for DQOs would be welcomed. Hazardous waste that has been treated for organic constituents may contain residual reagent and will very quickly "treat" any surrogates that come into contact with the sample. Very low and/or divergent recoveries may result; the amount of recovery tells you more about how quickly an analyst can add solvent to the spiked sample than anything helpful for assessing data quality. Attempting to recover surrogates may also be the only reason a concentration step is required as part of an extraction procedure, since final extract volume in some methods is too large for an affordable amount of surrogate to be detectable. Skipping the concentration step can improve recovery of the more volatile analytes. It may not be the case that DQOs are in all cases well served by the requirement that surrogates be used in every sample.

Response #1(B):
Surrogates are a requirement to monitor the performance of the extraction process as a whole. Method 8270E allows for many different preparation procedures to be used. The method is performance based. If an individual laboratory wanted to modify their own extraction procedure by extracting in such a way as to not require concentration, they may do so. Most applications will require concentration to meet reporting limits.

Comment #1(C):
Commenter #1 made a comment on Method 8270E, Section 11.4.4.2. This section says that corrective action must be taken if more than 20% of calibrated compounds (or more than 20% of compounds to be reported) show greater than 20% difference or drift from the ICAL. Some flexibility would help here. Each analyte IS pair has its own curve. If every compound being reported passes the %D criteria, and these are the only compounds that need to be reported for the particular project's DQOs, a corrective action will not improve data quality, and should not be required. One can imagine an analyst lamenting, "if only I hadn't calibrated for the nitrosamine compounds on this instrument, my PNA data would be perfect!".

This requirement is actually a disincentive to creating calibration curves for more "difficult" compounds that are not required by the project, but which may be useful to have calibrated for some other reason (researching remediation methods, screening, observing breakdown products, identifying interferences, or for the purpose of judging instrument inertness over

time). It is as though the worst compounds in the calibration are treated like continuing check compounds (CCCs) that an analyst can choose not to run. I suppose one could add compounds with good stability to the method to increase the overall number and reduce the fraction of failing compounds.

I suggest eliminating this requirement or replacing it with something that does not use the number of extraneous compounds in a calibration as a determining factor in how data quality for constituents of concern is assessed. If a 20% rule is unavoidable, please consider broadening the pass range for compounds that aren't being analyzed for or reported on, as was the case with the CCC requirements in 8260B (section 7.3.6). As discussed in the next section, the IS can also be helpful in tracking overall system stability.

Response #1(C):
The user is not required to run all compounds in the method. If the poly-nuclear aromatic (PNA) compounds are all that are of interest, those are all you would need to run. If you wished to run a full list and report a shorter list from it, the 20% criteria only applies to the list you were reporting or the data should be flagged. The following text has modified in Sec. 11.4.4.2 to clarify: "If the criterion is not met (i.e., greater than ±20%D or drift) for more than 20% of the compounds included in the ICAL (or more than 20% of those that will be reported), then corrective action must be taken prior to the analysis of samples."

Comment #1(D):
Commenter #1 made a comment on Section 8270E, Sections 11.4.4 and 11.4.5. The move away from relative retention time (RRT) to a straightforward time-based limit is helpful. The IS response recovery range of 50% to 200% seems generous, but I'm sure the range reflects different projects where it may be appropriate. When monitoring IS response as a way to track instrument sensitivity and stability, comparing the ISs to each other may be useful as well. As an example, consider two cases: In the first, both 1,4-Dichlorobenzene-d4 and Perylene-d12 are recovering at 55% of the expected value. In the second, 1,4-Dichlorobenzene-d4 is also recovering at 55%, but Perylene-d12 is recovering at 160%. In both cases the IS recovery meets the requirements described in the method, but I think most analysts would be concerned about the second.

Response #1(D):
The Agency thanks the commenter for their input. The IS acceptance criteria are intended to be general guidance. If a laboratory wished to use more stringent guidelines they are welcome to do so. 50% - 200% is also consistent with other EPA test methods.

Comment #3:
Commenter #3 commented on Methods 8260D and 8270E, saying they appreciated the opportunity to provide comments on the proposed methods. Commenter #3 agrees with all proposed changes to these methods and commends EPA for making the following improvements:

- Allowing the use of more modern technology which has become available since the last publication of these methods to facilitate more flexibility and sensitivity.
- Revising tune verification procedures to reduce frequency for increased analytical efficiency.
- Adding language to make minimum RFs in Table 4 (Guidance Response Factor Criteria for Initial Calibration) guidance only rather than requirements to more accurately represent the intent of this table.

Response #3:
The Agency thanks the commenter for their input.

Comment #4(J):
Commenter #4 commented on Method 8270E, Section 1.4.4. Commenter quotes the method saying that "N-Nitrosodimethylamine is difficult to separate from the solvent peak under the chromatographic conditions described". Commenter has not observed this to be a problem.

Suggested text: Delete the section.

Response #4(J)
The Agency thanks the commenter for their input. We agree that the separation may not always be difficult. Therefore, the language in Sec. 1.4.4 has been changed to "may be difficult", rather than "is".

Comment#4(K):
Commenter #4 commented on Method 8270E, Section 7.7.5. Storing at -10 ºC can cause some analytes to fall out of solution. Commenter does not think it is a good recommendation.

Suggested text:
Each 1-mL aliquot of calibration standard should be spiked with 10 µL of the IS solution prior to analysis. All standards should be stored away from any light source at ≤6 °C when not in use, and should be freshly prepared once a year, or sooner if check standards indicate a problem. The ICV and CCV standards should be prepared, as necessary, and stored at ≤6 °C.

Response #4(K):
The Agency thanks the commenter for their input. The storage of standards at -10 ºC is not a requirement, merely a recommendation in the method. It is also recommended that all standards be brought up to room temperature before use.

Comment #4(L):
Commenter #4 commented on Method 8270E, Sec. 9.6.3. Since this is an instrument batch and not a preparation batch, an instrument blank should also be acceptable (in part to be consistent with Section 9.5.1 and 11.4.3).

Suggested text:
A MB or instrument blank must be included with each analytical batch. MBs consist of an aliquot of clean (control) matrix similar to the sample and of a similar weight or volume. Other types of blanks (e.g., equipment rinsates, storage blanks, etc.) should be included when appropriate but are distinct from MBs.

Response #4(L):
The Agency thanks the commenter for their input and agrees with the commenter. The suggested text was added to Sec. 9.6.3 of 8270E.

Comment #4(M):
Commenter #4 commented on Method 8270D, Sec. 9.9.1.2. In the commenter's opinion, the allowance to verify the LLOQ on a particular instrument only once every three years is a bit minimal. Suggest that the LLOQ should be verified at least once per year on each instrument.

Suggested language:
The LLOQ verification is prepared by spiking a clean control material with the analyte(s) of interest at 0.5 - 2 times the LLOQ concentration level(s). Alternatively, a representative sample matrix free of targets may be spiked with the analytes of interest at 0.5 - 2 times the LLOQ concentration levels. The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples. At a minimum, the laboratory should perform the LLOQ verification on each instrument once per year.

Response #4(M):
The Agency thanks the commenter for their input. The method requirements are a minimum level of acceptable QC. Individual laboratories and data users are welcome to use more stringent criteria where needed.

Comment #4(N):
Commenter #4 commented on Method 8270D, regarding the note in Sec. 11.3.1.2. In the commenter's opinion, decafluorotriphenylphosphine (DFTPP) analysis is not useful for SIM analysis (since it is not performed in SIM mode). There should be a requirement for demonstrating mass accuracy and resolution with PFTBA, similar to the requirements for tandem MS and CI. We suggest merging the SIM requirement with the second note.

Suggested text:
DFTPP tune checks are not appropriate for SIM analysis, CI analysis or tandem MS analysis using SRM. However, the laboratory must demonstrate, prior to the ICAL, that the MS system achieves mass accuracy and mass resolution criteria specified by the instrument manufacturer for the perfluorotributylamine (PFTBA) internal calibrant or another appropriate chemical.

Response #4(N):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and the following language was added to the notes in Sec. 11.3.1.2: "NOTE: All subsequent standards, field samples, and QC samples associated with a DFTPP analysis must use identical MS instrument conditions with the exception of SIM analysis. DFTPP may be analyzed in full scan mode while standards, samples, and QC are analyzed in SIM. As an alternative to DFTPP for SIM analysis, the laboratory may use an alternate detector verification, such as PFTBA, or the manufacturer's recommended detector check.

NOTE: DFTPP tune checks are not appropriate for CI analysis or tandem MS analysis using SRM. However, the laboratory must demonstrate, prior to the ICAL, that the MS system achieves mass accuracy and mass resolution criteria specified by the instrument manufacturer for the perfluorotributylamine (PFTBA) internal calibrant or another appropriate chemical. "

Comment #4(O):
Commenter #4 commented on Method 8270D, Sec. 11.3.7. The commenter suggests standardizing on the language in 8260D with the text listed below.

Suggested text:
Sec. 11.3.7 ICV – Prior to analyzing samples, verify the ICAL using a standard obtained from a second source to the calibration standard, if possible, such as a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration, if readily available. This standard should be prepared in the same clean control matrix as that used for ICAL standards. Suggested acceptance criteria for the analyte concentrations in this standard are 70 - 130% of the expected analyte concentration(s). Alternative criteria may be appropriate

based on project-specific DQOs. Quantitative sample analyses should not proceed for those analytes that do not meet the ICAL verification criteria. However, analyses may continue for those analytes that do not meet the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values.

Response #4(O):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and Sec. 11.3.7 now reads: "Prior to analyzing samples, verify the ICAL using a standard obtained from a second source to the calibration standard, if possible, such as a second manufacturer or a manufacturer's batch prepared independently from the batch used for calibration, if readily available. Suggested acceptance criteria for the analyte concentrations in this standard are 70 - 130% of the expected analyte concentration(s). Alternative criteria may be appropriate based on project-specific DQOs. Quantitative sample analyses should not proceed for those analytes that do not meet the ICAL verification criteria. However, analyses may continue for those analytes that do not meet the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values."

The Agency has made an effort to standardize the text between Methods 8260D and 8270E where the Work Group agreed it was practical to do so.

Comment #4(P):
Commenter #4 commented on Method 8270D, Sec. 11.4.4.2. This is a valuable and sensible allowance, but it would be useful to include some direction on how adequate sensitivity is to be demonstrated.

Suggested text:
In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. Adequate sensitivity may be demonstrated by including analysis of a standard spiked at or below the LLOQ in the analytical batch. Sufficient sensitivity is demonstrated for analytes that meet all applicable qualitative identification criteria.

Response #4(P):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group. Sec. 11.4.4.2 was revised to read as follows: "If the %D or percent drift for a compound is ≤20%, then the ICAL for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. The analyst should strive to place more emphasis on meeting the CCV criteria for those compounds that are critical to the project. If the criterion is not met (i.e., greater than ±20%D or drift) for more than 20% of the compounds included in the ICAL (or more than 20% of those that will be reported), then corrective action must be taken prior to the analysis of samples. Target analytes that do not meet the CCV criteria and are reported in the associated samples must be qualified to indicate the reported concentrations are potentially estimated or biased values. In cases where compounds fail low, they may be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the LLOQ or project specific level of interest (e.g., by calibrating below the established LLOQ to confirm the non-detect, or by analyzing a standard near that level to confirm the analyte could be qualitatively identified if it were present [See Sec. 11.7 of Method 8000]). Alternatively, the non-detect could be qualified or the LLOQ raised to a higher level. In

cases where compounds fail high in the CCV and are not found in the associated field samples, they may be reported without qualification."

Comment #4(Q):
Commenter #4 commented on Method 8270E, Sec. 11.6.1.2, that ±10 seconds is a really wide window.
Suggested text:
The retention time (RT) should be within ±2 seconds of the RT for this analyte in the CCV run at the beginning of the 12-hour period (delta RT 0.034 minute) or within ±2 seconds relative to the shift of the associated internal standard (IS) (delta RT of the IS ± 10 seconds).

Responses #4(Q):
The Agency thanks the commenter for their input. The method requirements are a minimum level of acceptable QC. Individual laboratories and data users are welcome to use more stringent criteria where needed.

Comment #4(R):
Commenter #4 commented on Method 8270E regarding the note in Sec. 11.6.1.2.
We have not observed this to be a problem and would consider a RT shift of >10 seconds relative to the CCV to be an indication of a malfunctioning instrument.

Suggested text: Remove the note.

Response #4(R):
The Agency thanks the commenter for their input. The note in Sec. 11.6.1.2 is not a requirement, merely an acknowledgment that some analyte's RT may be affected by matrix interferences which could shift RT forward. If a laboratory noticed significant RT shifts, they should investigate the cause and take appropriate corrective action.

Comment #4(S):
Commenter #4 commented on Method 8270E, Sec. 11.6.1.4. The CCV level is probably the same or at least close to the midlevel, so we are not sure what is gained by this text.

Suggested text:
Unresolved structural isomers with similar mass spectra are identified as isomeric pairs. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is ≤ 50% of the average of the two peak heights, or ([valley height]/ [average peak height] is ≥50%). At a minimum, the resolution should be verified on the mid-point concentration of the ICAL.

Response #4(S):
The Agency thanks the commenter for their input. This suggested text was discussed with the SW-846 Work Group and the following language was added to Sec. 11.6.1.4: "It is important to check the separation of structural isomers in the ICV and the daily CCV check standards to verify if the instrument performance is adequate regarding separation of compounds of interest which are structural isomers."

Comment #4(T):
Commenter #4 commented on Method 8270E, reference 19. This document (R. Burrows, Basic RSE calculator v2 and instructions, December 2016) can be obtained from The NELAC Institute (TNI) web site.

Suggested text:
Reference 19, R. Burrows, Basic RSE calculator v2 and instructions, December 2016
Available at: http://nelac-institute.org/docs/comm/emmec/Calculating%20RSE.pdf

Response #4(T):
The Agency agrees with the commenter and will make the change in the final version. The referenced spreadsheet can currently be found in the docket on regulations.gov.

Comment #5(A):
Commenter #5 commented on Method 8270E, Sec. 4. Section 4 (Interferences) is missing most of the information that was contained in Sec. 3.0 Interferences in 8260B. The older section had tips to avoid contamination, such as avoiding the use of non-PTFE thread sealants, plastic tubing and rubber components. It also contained information on how to clean contaminated glassware and was more specific about the purpose of a trip blank. 8260D refers the reader to Method 8000 but that is not specific to the unique challenges of volatiles. This is all valuable information to continue to pass on to new analysts.

Response #5(A):
The Agency thanks the commenter for their input. That information was removed in 8260D, as it pertained to older uses of the method. These interferences are not applicable to 8270E analyses. SW-846 is not intended to be a complete training manual. Laboratories must develop their own SOPs for any glassware or non-disposable materials used to ensure they are free of contamination.

Comment #6(J):
Commenter #6 commented on Method 8270E, Disclaimer (Paragraph 2). The following language is included in the second paragraph: "The performance data referenced in this method are for guidance purposes only and are not intended to be and must not be used as absolute quality control (QC) acceptance criteria for purposes of laboratory accreditation." The term "must not be used" should be removed as this should be a state decision on QC required for laboratory accreditation or specific state projects.

Response #6(J):
The Agency thanks the commenter for their input. The SW-846 methods are guidance and are performance based. The laboratories and data users should determine what their project's DQOs and requirements need to be. If they wish to use more stringent criteria for their project's needs, they are welcome to do so. Those requirements should be stipulated in the projected QC documents.

Comment #6(K):
Commenter #6 commented on Method 8270E, Section 1.2. This section states that EPA Method 3511 can be used for the extraction of semivolatiles in water. EPA Method 3511 only addresses the use of this extraction technique for Polynuclear Aromatics Hydrocarbons (PAHs). Provide the reference for the additional performance data for the other semivolatiles using EPA Method 3511 or specify in Section 1.2 that Method 3511 is for PAHs only.

Response #6(K):
The Agency thanks the commenter for their input. Method 3511 states in Sec. 1.3: “This method also may be used to extract selected volatile organic compounds (VOCs) or semivolatile organic compounds (SVOCs) which are slightly soluble or insoluble in water at neutral pH once their extraction performance has been demonstrated to be satisfactory using an appropriate analytical technique.” If a laboratory wished to use Method 3511 to extract samples and report any compounds (including PAHs or other compounds), they would be required to show they were able to adequately recover those compounds by this method. Many SW-846 extraction methods are useful for compounds not listed in the original development. In those cases, the burden of proof falls to the laboratory performing the analysis to demonstrate acceptable recovery for the project’s needs.

Comment #6(L):
Commenter #6 commented on Method 8270E, Sec. 9.3.2. This section states to verify the ICAL standards using a second source ICV standard, if readily available. The term "if readily available" needs to be removed or add additional language to explain what the language "if readily available" means.

Response #6(L):
The Agency thanks the commenter for their input. This method is used for a wide variety of analyses. As such, many standards are commercially available for common analyte lists. The term “if readily available” is meant to provide flexibility for situations for which a second source standard cannot easily be found from a commercial supplier. The intention of the method is to have a full-list ICV standard, if possible, while addressing the possibility that some, less common analytes may not have more than one source available. The best practice is to have a full list ICV to verify the calibration source. The following text was added to 7.7.3 “A second lot number from the same manufacturer may be adequate to meet this requirement.

Comment #6(M):
Commenter #6 commented on Method 8270E, Sec. 11.3.4.1. The language in Section 11.3.4.1 of EPA 8270D states, "meeting the minimum RF criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity." This language was removed from EPA 8270E and should be added back to EPA 8270E since this discourages laboratories from attempting to calibrate lower on instruments that cannot meet the desired sensitivity.

Response #6(M):
The Agency thanks the commenter for their input and the commenter is correct about the language change. The current Method 8270E now states in Sec. 11.3.4.2: “The RSD should be ≤20% for each target analyte (see Sec. 11.3.5). 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. The laboratory should establish procedures in its SOP (e.g., laboratory established minimum RFs, signal-to-noise (S/N) checks, etc.) to ensure that the instrument is working properly and that calibration standards were correctly prepared.” The RF tables were always intended as guidance and not intended to be an absolute requirement. The removal of the older language was intentional and discussed by the SW-846 Work Group.

Comment #6(N):
Commenter #6 commented on Method 8270E, Sec. 11.3.5.1. Sec. 11.3.5.1 states: "NOTE: Forcing the calibration model through the origin (for analytes that are consistently detected in the laboratory reagent blanks) allows for a better estimate of the background level of blank contaminants. An accurate estimate of background contamination is necessary to set method reporting limits for method analytes when blank levels are problematic." The analyst should not alter the calibration model to compensate for laboratory contamination. The source of the contamination should be eliminated from the analytical process. We recommend this note be removed from the method.

Response #6(N):
The Agency thanks the commenter for their input. The issue of background contamination in semivolatile calibration standards was discussed with the Work Group, who did not feel it was a significant issue in this analysis. The issue is more prominent in volatile analyses. Therefore, the note in Sec. 11.3.5.1 of Method 8270E has been removed.

Comment#6(O):
Commenter #6 commented on Method 8270E, Sec. 11.3.5.3. Sec. 11.3.5.3 states, "In order to report non-detects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable LLOQ." All target analytes in the ICAL should pass in order to generate reportable data even non-detects to prevent the possibility of reporting false negatives. Additional language should be added to this section requiring the laboratory to verify with the regulatory authority the contaminants of concern for a particular project to ensure that valid calibration data is obtained.

Response #6(O):
The Agency thanks the commenter for their input. The Method does not require that 100% compounds pass criteria in order to report data. It allows for the reporting of flagged data when certain conditions and limits are met. If a data user, including a SRA, wishes to use more stringent QC criteria they are welcome to do so, but would have to specify those criteria in their DQOs and project documents.

The text in Sec. 11.3.5.3 has been revised as follows: “If more than 10% of the compounds included with the ICAL (or more than 10% of those that will be reported) exceed the 20% RSD limit and do not meet the minimum correlation criteria ($r^2 \geq 0.99$ or relative standard error (RSE) ≤20%) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Correct the source of the problem; then repeat the calibration procedure beginning with Sec. 11.3. If compounds fail to meet these criteria, the associated concentrations may still be determined but they must be reported as estimated. In order to report non-detects, it must be demonstrated that there is sufficient accuracy to detect the failed compounds at the applicable LLOQ (see Secs. 11.3.6 for refitting standards and 11.4.4.2 for CCV). Refer to Method 8000 for further discussion of RSE. Example RSE calculations can be found in Reference 19.”

Comment #6(P):
Commenter #6 commented on Method 8270E, Sec. 11.3.6. Sec. 11.3.6 requires the recalculating of the initial calibration standards of the ICAL. For the lowest calibration standard (LLOQ), ± 50% difference is allowed. Method 8270D was more stringent allowing only ± 30% difference from the expected value. Because this is a calibration standard and not an extracted standard, the laboratory should have no problem meeting the ± 30% acceptance criteria. The laboratories have not expressed that this criterion is too stringent.

Response #6(P):
The Agency thanks the commenter for their input. The responses at the lowest level of the calibration will generally not be as precise as the higher levels for some compounds. The 50% RSE criteria listed in Method 8270D is based upon guidance found in Method 8000D Sec.11.5.4.1. If the data user or laboratory wishes to use more stringent QC criteria, they are welcome to do so in their DQOs and project documents. This criterion is also consistent with other EPA test methods.

Comment #6(Q):

Commenter #6 commented on Method 8270E, Sec. 11.4.1. Sec. 11.4.1 states: "Daily analysis of the GC/MS tune check solution is no longer required as part of the CCV." The significance of removing this requirement was not included in the revision history. We would like clarification as to why the daily (every 12 hrs.) DFTPP tune check requirement was removed from the method. The tune requirement is still included in the approved methods for drinking water and wastewater. We suggest the daily tune requirement be added back to the method to provide added quality control for the instrument.

Response #6(Q):
The Agency thanks the commenter for their input. This practice is consistent with other EPA test methods, including the drinking water methods. The MS tune check defines the MS as working properly prior to the ICAL. If the CCVs are still within criteria, that sufficiently demonstrates that the detector is still operating within control as determined by this method and the laboratory's SOP. Laboratories or data users that wish to use more stringent criteria, such as requiring tune check each 12-hour clock cycle, are welcome to do so.

Comment #6(R):
Commenter #6 commented on Method 8270E, Sec. 11.4.4.2. Sec. 11.4.4.2 allows the failure of 20% of the target analytes in the CCV. This section states: "the affected target analytes may still be reported as non-detects in field samples if it can be demonstrated that there was adequate sensitivity to detect the compounds at the applicable quantitation limit." Should "quantitation limit" be changed to "LLOQ"? The procedure should also document how adequate sensitivity is to be determined. Should this require the analysis of an LLOQ check standard for all target analytes to verify sensitivity?

Response #6(R):
The Agency thanks the commenter for their input. The intent of the SW-846 is to be guidance for performance based environmental testing. The term quantitation limit used here can mean LLOQ, but the user could specify some other limit in their DQOs. The individual laboratory and data users must decide what quantitation limit (LLOQ is recommended) best meets their needs for use of the data. Some poorly performing compounds will have higher LLOQs due to poor extraction efficiency, overall lower stability or poor detector response. In these situations, the analyst must use professional judgment and set DQOs and LLOQs at reasonable levels for the project's needs. If the data user needs to use more stringent QC criteria, they are welcome to do so in their DQOs and project documents. An LLOQ check standard can be a helpful tool in establishing that sensitivity is adequate. Secs. 11.3.5.3 and 11.4.4.2 have been updated based on several user's comments. The full text can be found in the revised Method 8270E and earlier in this document.

Comment#6(S):
Commenter #6 commented on Method 8270E, Sec. 11.4.4.2. Sec. 11.4.4.2 states, "NOTE: Daily tailing and degradation checks are good indicators of reactivity in the system and the need for maintenance. Because these are no longer required daily, the analyst must closely monitor responses and chromatography in the CCV for signs that the system is too reactive for analysis to continue (e.g., losses of reactive analytes, unusual tailing, loss of resolution). If significant losses of target analytes/ISs occur (<50% recovery) or if significant degradation of the chromatography occurs (tailing factor >2), system maintenance must be performed or the analyst must demonstrate there is adequate sensitivity at the LLOQ." If the analyst is required to monitor responses and chromatography for signs of degradation and tailing, the best method of doing this is the degradation checks that were required in Section 11.3.1.3 of EPA 8270D. These daily checks should be added back to EPA 8270E. This allows the laboratory to monitor changing trends over time and allows the analysts to take corrective action before a problem occurs.

Response #6(S):
The Agency thanks the commenter for their input. The intent of SW-846 is to be guidance for performance based environmental testing. If the data user needs to use more stringent QC criteria (such as performing daily degradation checks), they are welcome to do so in their DQOs and project documents. It was felt that the tailing and degradation could be judged well enough in the CCV standards that the check was not needed each day.