

Connecticut Department of Environmental Protection
Responses to Public Comments on the Reasonable Confidence Protocols
Comment Period July 29, 2005 – December 28, 2005
Published June 13, 2006

The following are comments received on the Reasonable Confidence Protocols and proposed responses:

From: STL-Westfield

1. Comment: Why do the RCPs not mention SW-846 method 3546, Microwave Extraction for pesticides/PCBs by Methods 8081 and 8082.

Response: The microwave procedure will be allowed. Air-drying of samples will not be allowed except for PCBs.

From: Connecticut DPH Laboratory

2. Comment: Method 8260, In Table 1A, item 4, the response action regarding internal standards is vague. The current version reads, “If reanalysis performed outside of holding time and IS are in *out* of criteria, report both sets of data, note in narrative.”

The response should read: If reanalysis performed outside of holding time and IS are in criteria, report both sets of data, note in narrative.

Response: Commenter is correct, we will change the wording.

3. Comment: Method 8260, Section 1.8.2, instructs the user to substitute the value of V_t into equation 11.10.1 of Method 8000C. The value of V_t should be substituted into equation **11.10.2**, which is the soil equation (11.10.1 is the aqueous equation).

Response: Commenter is correct, we will change the wording.

4. Comment: Method 8260, the method requires equal volumes of soil/methanol (e.g. 1 gr soil to 1 mL methanol). We suggest using enough methanol to cover the soil sample.

Response: We agree that the methanol must cover the soil completely, or target analytes may be lost, but we also feel the ratio of soil to methanol should be approximately 1:1, in order to not have higher reporting limits than needed. This table will be changed to follow the new DEP *Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006.*

5. Comment: Method 8260, the requirement that surrogates be added to the methanol prior to sample collection is too burdensome on the laboratory. The current generation of purge and trap instruments automatically add surrogates and internal standards to samples just prior to analysis. Additionally we request the requirement to perform a multi-point calibration for surrogates be removed. Correspondence with the SW846 authors via MICE indicates this requirement will be dropped in the next release of this method.

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Additionally other methods, such as 524.2, do not require a multi-point calibration for surrogates.

Response: The requirements for surrogates to be added to the methanol prior to the sample will be removed. The QA/QC Workgroup believes a multi-point calibration should be performed for surrogates to obtain accurate recovery data.

6. Comment: ETPH Method, Item 3 of Section 1.4.2 makes reference to Section 1.7 of the method. The method has no Section 1.7, the reporting formats are in Section 1.6.

Response: Commenter is correct, we will change the wording.

From: AMRO

7. Comment: The target compound lists contain a few “odd-ball” compounds not part of any current standardized state-required lists:

Method 8081: Alaclor

Method 8260: trans-1,4-Dichloro-2-butene

Method 8270: Pentachloronitrobenzene and 1,2,4,5-Tetrachlorobenzene

Response: Alachlor is a Connecticut RSR compound and is the second most widely used herbicide for control of annual grasses and broadleaf weeds. Alachlor has been used in Connecticut and the DEP is interested in determining the prevalence of this compound in the environment.

Trans-1,4-Dichloro-2-butene, Pentachloronitrobenzene and 1,2,4,5-Tetrachlorobenzene are also Connecticut RSR compounds. As such the concentration of these compounds must be determined in order for environmental professionals to make decisions on a given site.

From Northeast Utilities, Method 8082 for PCBs

8. Comment: NU suggests the need for additional QA/QC procedures for their laboratory is not needed due to their specialized focus.

Response: The QA/QC Workgroup notes that the RCPs are voluntary. If an entity feels their own program meets or exceeds the requirements of their needs, it is their decision to adopt the RCPs or not. If the entity is regulated by other Federal or State agencies, and their analytical program is tailored to meet these other agency needs, it is up to the entity to adopt the RCPs or not.

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9. Comment: The enhanced QA/QC protocols jeopardize the quick and efficient PCB analysis required by NU to meet regulatory requirements.

Response: See 8 above.

10. Comment: The types of analyses performed by NU's laboratory are distinguishable from those used to make remediation decisions.

Response: See 8 above.

11. Comment: Site-specific Matrix Spikes/Matrix Spike Duplicates are not always necessary for each sampling event.

Response: The RCP guidance recommends site specific MS/MSDs in lieu of batch MS/MSDs. The point of the site specific QC is to characterize the site-specific matrix effects on analyte recoveries. The guidance recommends a site specific MS/MSD every 20 samples, not every sampling event. The commenter also maintained spiking with Aroclors 1216 and 1260 (we assume they meant 1016 and 1260), as these aroclors contain most of the individual PCB congeners. We agree that the PCB-1016/1260 is adequate for MS/MSDs, however in cases where it is known different PCBs are present, we recommend using the actual PCBs in lieu of the 1016/1260 in order to simplify quantitation of the aroclors.

12. Comment: Sonication should be allowed for soil samples for PCBs.

Response: The QA/QC Workgroup does not agree that sonication is a robust enough extraction procedure for PCBs in all soil matrices. Studies have shown that clays and other absorbing matrices are not efficiently extracted by sonication. The commenter states that there would be no need to use pressurized fluid extraction techniques. We note that the RCPs allow for other extraction techniques such as the Soxhlet and automated Soxhlet procedures.

13. Comment: The use of a 1:1 mixture of a hydrophilic solvent mixture should be left up to the laboratory.

Response: Again the RCPs are guidance only. The laboratory may use whatever solvent mixture they feel is adequate for their needs. If different solvents are used it should be noted in the narrative if the laboratory is certifying the data is in compliance with the RCPs.

14. Comment: The use of amber glass jars may lead to errors in PCB analysis.

Response: The word amber will be removed from the containers for transformer and waste oils.

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15. Comment: The use of TMX and DCB as surrogates across the GC run time will lead to problems with TMX interfering with the early eluting peaks.

Response: As adoption of the RCPs is voluntary, it is up to the laboratory to determine what surrogates to add. Again deviations from the RCPs should be noted in the narrative if the laboratory is certifying the data is in compliance with the RCPs. The QA/QC Workgroup notes that it is standard in the environmental testing laboratory industry to use TMX and DCB as pesticide/PCB surrogates.

16. Comment: Please clarify the reporting limit for PCBs.

Response: A laboratory is required to be calibrated at or below the reporting limit. We would assume the reporting limit would be at or below any regulatory limit the data is intended to satisfy.

17. Comment: What is the ultimate fate of the RCPs – guidance or regulation.

Response: The RCPs will be added as an Appendix to the current draft site guidance. At some time we would expect the draft guidance to become final.

From Complete Environmental Testing

18. Comment: We would like to see the PCB reporting limit changes to 0.25 ppm in lieu of the 0.10 ppm.

Response: There are no reporting limits specified in the RCPs, except for the low level volatile method. RSR limits are not part of the RCPs.

19. Comment: Sonication should be allowed as an option for Methods 8081/ 8082.

Response: See 12 above.

20. Comment: For PCBs, the sulfuric acid/permanganate cleanup procedure should be optional, not a requirement.

Response: The QA/QC Workgroup feels the use of the cleanup procedure is needed for soil samples, in general. However, we agree that it is not always needed for aqueous samples. We will require the cleanup for soils, and leave it as an option for aqueous samples. In any case if the laboratory cannot meet the needed detection limit, cleanups are required prior to claiming matrix interference.

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21. Comment: Method 8081/8082/8151, can a 3rd party cross-tested source standard be used for the LCS and matrix spike (i.e. Absolute Standards 3rd party RDAR Program Standards).

Response: Yes, RDAR type standards are acceptable as 3rd party standards.

22. Comment: Can the accelerated solvent extraction procedure, Method 3545, be used for Method 8151.

Response: Yes, Method 3545 will be allowed. Air-drying of samples will not be allowed for herbicides.

23. Comment: Method 8151, Can tetrabutylammonium hydroxide/methyl iodide be used as the derivatizing agent in place of diazomethane?

Response: Method 8151 allows the use of diazomethane or pentafluorobenzyl bromide as derivatizing agents. As we have no data on the effectiveness of the tetrabutylammonium hydroxide/methyl iodide reagent outside of drinking water samples, we cannot support its use at the present time.

24. Comment: Method 8151, More specific instructions are needed concerning when the reanalyze the LCS.

Response: The batch of samples should, in general, be re-extracted and reanalyzed if the LCS fails. Recognizing that some herbicide analytes are problematic, laboratories may be able to identify those compounds that routinely fail the 40-140% recovery criteria. In those instances wider compound recovery windows may be appropriate. This should be documented and noted in the narrative.

25. Comment: There were two comments made regarding reporting limits for 2,4-D.

Response: There are no reporting limits specified in the RCPs, except for the low level volatile method. RSR limits are not part of the RCPs.

26. Comment: We would like to raise the reporting limit for ETPH from 0.10 mg/L to 0.25 mg/L.

Response: There are no reporting limits specified in the RCPs, except for the low level volatile method. RSR limits are not part of the RCPs.

27. Comment: Does the metals digestion procedure refer to all aqueous samples, including drinking water with a turbidity <1 NTU?

Response: The QA/QC Workgroup is in agreement that waters with a turbidity <1 NTU do not require digestion. The method will be modified accordingly.

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28. Comment: The reporting limit standard has a $\pm 30\%$ limit for Methods 6010/6020. This limit seems to be too stringent for the technology. For instance the EPA Contract Laboratory Protocols have the following criteria for the CRI standard:

Control Limit 70-130% with the following exceptions:
ICP-AES – Antimony, Lead, and Thallium: 50-150%.
ICP-MS – Cobalt, Manganese, and Zinc: 50-150%.

Response: The QA/QC Workgroup notes that a majority of the metals have a RSR lower limit above the CLP Contract Required Quantitation Limit (CRQL) which is the concentration of the CRI for ICP-AES. However we note that several metals have RSR limits significantly below the ICP-AES CRQL, notably antimony, arsenic, cobalt, and thallium. We will modify the control limits for these metals to 50-150% for the Method 6010.

The CRQL limits for the ICP-MS method are all significantly below the lower RSR limits. Laboratories can therefore select a reporting limit above the CRQL and should be able to meet the 70-130% criteria. The 70-130% criteria will remain for Method 6020.

29. Comment: Methods 6010/6020, shouldn't the control limits for the LCS and site-specific matrix spikes be based upon laboratory derived limits.

Response: The QA/QC Workgroup feels the limits specified are reasonable and adequate. Labs may use their own limits as long as they are within the described limits.

30. Comment: There is no reference to the ICSA/ICSAB solution for Method 6020.

Response: There is a reference to the ICSA/ICSAB in Table 1A of Method 6020. The requirement is the found concentration must be within 20% of the true value. We will modify the requirement to take into account elements not present in solution.

31. Comment: Methods 6010/6020, the control limits for LCS samples are listed as within the 95% confidence limits for water and soils. The matrix spike recovery limits are listed as 75-125%. Should these be switched?

Response: The control limits for the aqueous LCS is within 20% of the true value, soil LCS samples, usually supplied by commercial vendors, have various control limits. Laboratories must use the 95% confidence limits for these. The matrix spike recovery limits of 75-125% were deemed adequate and reasonable for the method.

EPA method 8260:

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32. We would like to allow one surrogate to fail for concrete samples. The recovery for dibromofluoromethane is very low for concrete chip samples.

Response: Any failed surrogate for this method will require reanalysis to verify matrix interference.

33. For the LCS and CC, we would like to use actual laboratory control limits (even if they are less than 70% recovery) for some of the “difficult” compounds (water soluble compounds and high boilers).

Response: Individual laboratories must identify and document problem analytes that routinely fall outside the 70-130% limit. Any exceedances must be noted in narrative. Data to support laboratory problem compounds kept on file at lab for review during audit.

34. 2-Isopropyltoluene is listed as a compound in this method. It is not listed in SW846 methods 8260 or 8021, and is not on EPA method 524.2. We checked several vendors and could not find this compound as part of any volatile mixture solution. We could only find this compound in the neat form. We would like to see this eliminated as a compound in the method.

Response: This compound will be deleted from the target list.

From ConTest

35. Comment: The laboratory LIMS system does not allow the use of report qualifiers (J, B, E, P). What provisions are available for laboratories in this predicament?

Response: The DEP feels that reporting formats need to be standardized. Laboratories are required to follow the reporting format.

36. Comment: The QA/QC certification form requires an explanation of the acceptability of samples received outside temperature receipt requirements. What guidance is there for this?

Response: The acceptability of analyses conducted on samples received outside of temperature requirements is dependent upon the specific circumstances and analytes. For example most samples for metals only would be acceptable, whereas most samples for volatile organics would not. Other factors to consider would be the temperature at which the samples were collected vs. how much time has elapsed since they were received at the laboratory. Laboratories and data users should discuss the specific circumstances and document why and how the decision was

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made. In cases where no reasonable decision can be made, please contact the DEP for guidance or resample.

37. Comment: If multiple dilutions are made on a sample, and only one is in the calibration range, why should the laboratory report additional dilutions.

Response: The dilution of a sample to bring the highest analyte concentration within the calibration curve will undoubtedly result in other analytes being not detected, with reporting limits above the regulatory criteria. By reporting a lesser dilution, it is hoped to minimize this number of compounds, so as to maximize the usefulness of the data.

38. Comment: Method 8260, the compound 2-Isopropyltoluene does not appear on any compound lists, nor does any chemical supplier offer it. Should this compound be listed as an 8260 or 8021 analyte.

Response: This compound will be removed from the target analyte list.

39. Comment: Method 8260, low-level option: The method references 525.4 – should this be 524.2.

Response: The reviewer is correct. This typo will be corrected.

40. Comment: Method 8260, there are several compounds with reporting limits below the ability of our instrumentation. Can the laboratory report a higher reporting limit or is it preferable to report non-detects below the lowest standard in the calibration curve.

Response: It is the intent of the RCPs, in general, to have the lowest standard in the calibration curve at or below the laboratory's reporting limit (e.g. less than value). If a laboratory reports compounds with RLs above the limits specified in the Low Detection Limit modification, the laboratory should note this in the narrative. If the RLs were below the regulatory level, there would be no significant impact on the data. A laboratory cannot report a non-detect below the lowest calibration standard, unless a reporting limit standard is analyzed per the method (e.g. 6010, 6020, etc.).

41. Comment: Method 8260, the preservation table requires surrogates to be added to the methanol prior to addition of sample. This is not in accord with current laboratory practices.

Response: See 5 above.

42. Comment: Method 8270, the compounds Carbazole, pentachloronitrobenzene and 1,2,4,5-tetrachlorobenzene do not appear on any of our laboratory compound lists. Are these compounds required?

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Response: These compounds are required.

43. Comment: The regulatory levels for several compounds are below what our instruments can see. Can the laboratory report a higher reporting limit or is it preferable to report non-detects below the lowest standard in the calibration curve.

Response: It is the intent of the RCPs, in general, to have the lowest standard in the calibration curve at or below the laboratory's reporting limit (e.g. less than value). If a laboratory reports compounds with RLs above the limits specified in the Low Detection Limit modification, the laboratory should note this in the narrative. It is expected that laboratories will have to use SIM to calibrate for some compounds. See comment #40.

44. Comment: Method 8081, the compounds alachlor and endrin ketone do not appear on our laboratory compound lists. Are they required?

Response: Alachlor is a Connecticut RSR compound and is the second most widely used herbicide for control of annual grasses and broadleaf weeds. Alachlor has been used in Connecticut and the DEP is interested in determining the prevalence of this compound in the environment. Endrin ketone is a common breakdown product of endrin, is also a RSR compound, and is required.

45. Comment: Method 8081, our reporting limit for dieldrin is above the RSR limit.

Response: It is anticipated that laboratories will not be able to achieve the RSR limit for dieldrin. Laboratories should report to their lowest calibration standard.

46. Comment: Method 7000, the preparation method table should read "HNO3" not "HNO".

Response: Thank you , we will correct the typo.

47. Comment: Method 7000 for graphite furnace, the method requires all samples for graphite furnace analysis be spiked at the instrument. This seems excessive. Could this be changed to spike a percentage of samples of each matrix type?

Response: Due to the uncertainty of different matrices and the very real possibility of interferences in GFAAS, spiking of all samples at the instrument is a requirement. We note that method 7010 requires this.

From AccuTest

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48. Comment: Several methods prohibit forcing the calibration curve through the origin for linear calibrations. SW-846 Method 8000C allows for some curves to be forced through the origin. Can this requirement be removed?

Response: Calibration curves cannot be forced through the origin.

From York Analytical Laboratories

Comments on DEP Laboratory QA/QC Certification Form

49. For Item 6 on the Form “ For each analytical method referenced in this laboratory report package, were results reported for all constituents identified in the method-specific analyte lists presented in the Reasonable Confidence Protocol documents?”

York Analytical takes issue with the language of this representation. Some of the compound lists as presented in the RCPs contain analytes that cannot be measured at the RSR limits or are not typically measured by the specific method.

For example, under the Volatile Organics by Method 8260, SW846 version 1.0 July, 2005, two specific compounds, EBD and DBCP are noted to be analyzed by EPA method 504.1 to achieve the RSR limits. This being the case, perhaps they should be removed from the list in the method to eliminate confusion that the RSR can and should be met for these compounds by 8260. In addition, the DEP has recently listed additional contaminants in the RSR including acrolein, ethanol, 1,4-Dichlorobutene, ethyl acetate, isopropanol, tert-butyl alcohol and vinyl acetate. None of these are on the 8260 list in the method as written. If these compounds are indeed listed in the RSRs they should be addressed in the methods for the RCPs.

Response: EDB and 1,2-DBCP were specifically included in the method to address soil samples, where the RSR limits are such that Method 8260 is a viable option. While some compounds may not have achievable RSR limits of detection, such as dieldrin in GA samples, by far the majority of analytes have achievable RSR limits using the RCP methods. Also with the exception of the low level volatile method, there are no specified reporting limits in the RCPs. We feel the language on the form is consistent with USEPA and other state programs.

The additional compounds may be added to these RCP methods at some future date, or new methods may be added. DEP expects to add new compounds to the RSRs as they are needed.

50. In the signature section, the language regarding “...and based upon my personal inquiry of those responsible for providing the information contained in this analytical report, such information is accurate and complete.” Needs to be reworded.

Under advice of counsel, a signature supporting these representations would require a personal communication with each and every employee that was involved in any way

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with the samples in the report. This is grossly impractical and I don't think that this is the spirit of this representation.

Many laboratory narratives for EPA CLP and NYSDEC CLP have a representations paragraph that is more practical and less onerous than the one proposed. Such a simple representation such as :

“We certify that these data are in compliance with the CT RCP requirements both technically and for completeness for other than the conditions stated in the above narrative. Release of the data contained in the hard copy report has been authorized by the Laboratory Manager as verified by the following signature.

Approved by: _____ Date: ____/____/____
Laboratory Director

Response: We disagree. The laboratory QA/QC program should be such that the laboratory management is confident the analysts in the laboratory are following the SOP's and the laboratory's QA/QC plan. This should be documented in the laboratory, making a personnel inquiry unnecessary.

Comments on Volatile Organics by Method 8260, SW-846

51. Table 1.3 GC/MS Tune criteria for BFB states that alternative BFB criteria such as 524.2 criteria can be applied. The intention of SW846-8260B is to have the laboratory tune the mass spectrometer in accordance with manufacturers instructions to a reference compound (Perfluorotributylamine). The resulting tune profile of a properly operating ion source will yield mass 69=100%, mass 131 = 40-45% and mass 219 = 40-45% with 0.5 amu (+/- 0.05) peak widths. On HP 5971 and 5972 mass spectrometers mass 75 is often slightly above 60 %. This is a characteristic of HP electron impact ionization systems. The BFB criteria developed in the mid 1980s was done so by EPA in Ohio using mostly Finnigan brand mass spectrometers. Finnigans fragment somewhat differently biasing mass 75 to the low side of the 30-60% range.

It is our suggestion that the criteria for mass 75 be widened to 30-66% (524.2) due to this fact. Our SOP reflects this allowance and this alternative criteria has passed validation scrutiny. As stated the approach cannot vary from day to day to meet the ranges.

Response: The RCP allows for alternate accepted GC/MS tuning criteria (i.e. The New York ASP criteria - laboratories may not invent their own criteria), as long as all samples, standards, blanks, etc. are analyzed using the same tune criteria. Method 524.2 has a range of 30-80% for amu 75. We feel laboratories that adopt alternate tune criteria must use all criteria from the alternate method. Laboratories may not pick and choose the criteria they wish to employ from various methods.

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52. We also have a concern with how the spectrum for BFB is obtained. By limiting it to the apex and one scan on each side of the apex. With the fast capillary analyses occurring today and with advances being made to decrease analysis times, the width of a capillary GC peak is becoming narrower and narrower. Even with very fast amplifier

electronics, defining a GC and MS peak by three scans is not prudent. Since the electronics must scan from say 35-260 amu (225 amu) in a very small amount of time, the mass peaks will be skewed. The best approach would be to average a minimum of 2 peaks on either side of the apex to minimize the mass spectral skew seen during fast capillary peaks. Perhaps this could be offered as an alternative to accommodate newer capillary technology.

Response: The recommended procedure for obtaining a BFB spectra is just that, a recommendation. Alternate standardized procedures may be used by the laboratory. The purpose is to halt the practice of hunting through several scans or combination thereof, to obtain a compliant spectra when in reality the mass spectrometer is in need of service. If a laboratory uses an alternate procedure, this must be noted in the SOP's and the report narrative. The practice of searching for the right combination of scans to add and background subtract to yield a passing tune is not allowed.

Initial Calibration

53. Item no. 3 requires all compounds having RSDs of $\leq 15\%$. This is not practical given the number of compounds to be determined and the characteristics of certain species with regard to purge and trap. The grand mean of the RSD of all the compounds of 15% as dictated in SW846-8260B is based upon the practical limitations of the method. Due to this fact/limitation with the methodology, more times than not laboratories will be citing a number of compounds in the narrative creating many questions on data use by the consultants/LEPs.

Response: According to Method 8000C of SW-846 (not yet promulgated), the use of the grand mean will be withdrawn. The criteria specified in the RCP method allows for 20% of the compounds to be outside of the 15% RSD criteria. Alternatively laboratories may use regression for quantitation. The QA/QC Workgroup deemed these criteria fair and routinely achievable. We also feel that data users will need to be aware when compounds are out of calibration criteria that may lead to biased results.

General Reporting Issues

54. The RCP says the laboratory should only report concentrations detected above the sample specific RL. Then it goes on to say (in 2) that concentrations below the RL are reported as "ND". However, in the "Analytical Response Action" column it states "(1) Qualification of results reported below the RL is required."

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This is confusing. Do we or can we report concentrations less than the reporting limit down to the MDL as “J” –estimated or not?

Response: The DEP expects most analytes detected below the sample specific reporting limit to be reported as not detected. However, in certain instances the data user may wish the laboratory to report detections below the RL. In those limited cases, the results would be report and the data (“J” values) must be qualified.

55. In Item 4) if a dilution is performed, the highest detected analyte must be in the upper 60% of the calibration curve.... This is an impractical requirement and implies that the bottom 40% of the curve is inaccurate, which is not the case. There is defined accuracy from the low standard to the high standard. Therefore this requirement does not have any technical basis.

Response: The requirement for analytes to be in the upper 60% of the curve is to prevent laboratories from over diluting a sample and reporting target analytes as non-detected at high reporting limits. This type of result is of limited value to the data user, especially if the reporting limits are above the applicable regulatory limit. The QA/QC Workgroup does not feel this requirement is either impractical or unreasonable.

TICs -Appendix A-2 Mass Spectral Criteria -8260

56. A-2.2. The spectral library match must be $\geq 85\%$ for a tentative identification to be made. In our opinion this will exclude many positive TIC identifications. This spectral match, very often is totally incorrect. In addition, many of the spectra in the NIST mass spectral database are incomplete, incorrect or “tilted” based upon their origin (i.e. electron impact ionization vs. others). This should not be a major consideration in mass spectral identification.

A-2.4 The relative intensities of the major ions must agree within 20%. This again is too restrictive for the same reasons above: In our opinion this will exclude many positive TIC identifications. This spectral match, very often is totally incorrect. In addition, many of the spectra in the NIST mass spectral database are incomplete, incorrect or “tilted” based upon their origin (i.e. electron impact ionization vs. others).

York understands the impact of a false positive (e.g. aldol condensation products mis-identified as TICs, etc.), however, by making these rules so restrictive many labs will not make any attempt at tentative identification (i.e. mass spectral interpretation) and report many unknowns with or without a class of compound. Leave some chemistry to the chemists.

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Response: The QA/QC Workgroup is concerned that significant decisions on sites might be made on TIC data, which could be inaccurate. Laboratories will be allowed to report TIC compounds that do not meet the criteria specified as long as it

is narrated in the report. The DEP guidance will recommend that prior to any site decisions being made on the basis of TICs, the compounds be verified by use of a calibrated standard and a demonstration made by the laboratory that the compound can be determined by the analytical method used.

57. A-3.1 The laboratory must evaluate the spectra for any compound which exhibits a characteristic chlorine or bromine spectral pattern. There is no further direction on how to handle or report such data, even though it is < 10% of the nearest ISTD.

Response: Laboratories must report any unknown peak that exhibits a characteristic chlorine or bromine pattern. The methods will be modified so that peaks less than 10% of the nearest IS need not be evaluated.

Comments on Semi-Volatile Organics by Method 8270, SW-846

58. Section 1.2.1 addresses extraction techniques for Method 8270 SVOCs. It states that sonication may only be used for extraction of highly contaminated (free product) non-soil/sediments(debris). Any other use of ultrasonic extraction is not allowed.

The method is essentially saying that other techniques must be used when indeed sonication (EPA SW846-3550C) is an approved time-tested procedure. The extraction efficiency is not only a function of the sonication, but also, and equally as important, is the preparation and the concentration steps. York has demonstrated equivalent recoveries with sonication for many soil types. It is important (critical) that the samples be mixed with anhydrous sodium sulfate to yield a free flowing powder to increase the surface area available for sonic extraction. In this manner recoveries rival those of other methodologies. There is no mention of the concentration options which can “lose” up to 50% of some of the analytes.

This method requiring the use of other extraction methods, when most laboratories (especially smaller ones) have invested in sonication devices is not warranted and unfair. For a laboratory to invest in PFE (or ASE - accelerated solvent extraction) for instance would be a \$40,000 expenditure. This requirement should not be taken lightly and should be reconsidered.

Response: The QA/QC workgroup is concerned that the extraction efficiency of sonication for certain matrices is not adequate for some site decisions. In order to evaluate if sonication is viable for a specific matrix, a side-by-side comparison would be required for each matrix at each site. As this is impractical, the use of sonication is restricted. Also see comment #12.

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59. Table 1C GC/MS Tune criteria for DFTPP states that alternative criteria such as 525 or CLP criteria can be applied. The intention of SW846-8270B is to have the laboratory tune the mass spectrometer in accordance with manufacturers instructions to a reference

compound (Perfluorotributylamine). The resulting tune profile of a properly operating ion source will yield mass 69=100%, mass 219 = 40-45% and mass 502 = 1-3% with 0.5 amu (+/- 0.05) peak widths. On HP 5971 and 5972 mass spectrometers mass 127 is often slightly below the 40-60% window in Table 1C. This is a characteristic of HP electron impact ionization systems. The DFTPP criteria developed in the mid 1980s was done so by EPA in Ohio using mostly Finnigan brand mass spectrometers. Finnigans fragment somewhat differently biasing mass 127 to the high side of the 40-60% range.

It is our suggestion that the criteria for mass 127 be widened to 30-60% due to this fact. Our SOP reflects this allowance and this alternative criteria has passed validation scrutiny. As stated the approach cannot vary from day to day to meet the ranges.

Response: See comment #51.

60. We also have a concern with how the spectrum for DFTPP is obtained. By limiting it to the apex and one scan on each side of the apex. With the fast capillary analyses occurring today and with advances being made to decrease analysis times, the width of a capillary GC peak is becoming narrower and narrower. Even with very fast amplifier electronics, defining a GC and MS peak by three scans is not prudent. Since the electronics must scan from say 40-450 amu (410 amu) in a very small amount of time, the mass peaks will be skewed. The best approach would be to average a minimum of 2 peaks on either side of the apex to minimize the mass spectral skew seen during fast capillary peaks. Perhaps this could be offered as an alternative to accommodate newer capillary technology.

Response: See comment #52.

Initial Calibration

61. Table 1A requires all compounds having RSDs of $\leq 15\%$. This is not practical given the number of compounds to be determined and the characteristics of certain species with regard to active sites and chromatographic characteristics. The grand mean of the RSD of all the compounds of 15% as dictated in SW846-8260B is based upon the practical limitations of the method. Due to this fact/limitation with the methodology, more times than not laboratories will be citing a number of compounds in the narrative creating many questions on data use by the consultants/LEPs.

Response: See comment #53.

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62. **Table 1A the LCS** (4) must contain all target analytes. There presently is no provider of soil LCS for all the target compounds in the RCP. An alternative must be offered whereby the laboratory purchases (from a different source than that used for GC/MS calibration) a standard or series of standards in solvent containing all the target BNAs. This is then injected onto a clean soil matrix and processed as the LCS.

Response: Laboratories may spike blank soil and use this as an LCS. The method will be modified to reflect this for 8260 and 8270.

General Reporting Issues-8270

63. The RCP says the laboratory should only report concentrations detected above the sample specific RL. Then it goes on to say (in 2))that concentrations below the RL are reported as “ND”. However, in the “ Analytical Response Action” column it states “(1) Qualification of results reported below the RL is required.”

This is confusing. Do we or can we report concentrations less than the reporting limit down to the MDL as “J” –estimated or not?

Response: See comment #54.

64. In Item 4) if a dilution is performed, the highest detected analyte must be in the upper 60% of the calibration curve.... This is an impractical requirement and implies that the bottom 40% of the curve is inaccurate, which is not the case. There is defined accuracy from the low standard to the high standard. Therefore this requirement does not have any technical basis.

Response: See comment #55.

TICs -Appendix A-2 Mass Spectral Criteria -8270

65. A-2.2. The spectral library match must be $\geq 85\%$ for a tentative identification to be made. In our opinion this will exclude many positive TIC identifications. This spectral match, very often is totally incorrect. In addition, many of the spectra in the NIST mass spectral database are incomplete, incorrect or “tilted” based upon their origin (i.e. electron impact ionization vs. others). This should not be a major consideration in mass spectral identification.

A-2.4 The relative intensities of the major ions must agree within 20%. This again is too restrictive for the same reasons above: In our opinion this will exclude many positive TIC identifications. This spectral match, very often is totally incorrect. In addition, many of the spectra in the NIST mass spectral database are incomplete, incorrect or “tilted” based upon their origin (i.e. electron impact ionization vs. others).

York understands the impact of a false positive (e.g. aldol condensation products mis-identified as TICs, etc.), however, by making these rules so restrictive many labs will not

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make any attempt at tentative identification (i.e. mass spectral interpretation) and report many unknowns with or without a class of compound. Leave some chemistry to the chemists.

Response: See comment #56.

66. A-3.1 The laboratory must evaluate the spectra for any compound which exhibits a characteristic chlorine or bromine spectral pattern. There is no further direction on how to handle or report such data, even though it is < 10% of the nearest ISTD.

Response: See comment #57.

Comments on Pesticides by Method 8081, SW-846

67. Section 1.2.1 addresses extraction techniques for Method 8081 Pesticides. It states that sonication may only be used for extraction of highly contaminated (free product) non-soil/sediments(debris). Any other use of ultrasonic extraction is not allowed.

The method is essentially saying that other techniques must be used when indeed sonication (EPA SW846-3550C) is an approved time-tested procedure. The extraction efficiency is not only a function of the sonication, but also, and equally as important, is the preparation and the concentration steps. York has demonstrated equivalent recoveries with sonication for most soil types. It is important (critical) that the samples be properly mixed with anhydrous sodium sulfate to yield a free flowing powder to increase the surface area available for sonic extraction. In this manner recoveries rival those of other methodologies. There is no mention of the concentration options which can “lose” up to 50% of some of the analytes. Some labs use K-D concentration while others use Turbo-Vap or like technologies. Others still yet finish with a simple nitrogen blowdown. These steps affect recoveries dramatically.

This method requiring the use of other extraction methods, when most laboratories (especially smaller ones) have invested in sonication devices is not warranted and unfair. For a laboratory to invest in PFE (or ASE - accelerated solvent extraction) for instance would be a \$40,000 expenditure. This requirement should not be taken lightly and should be reconsidered.

Response: See comments #12 and 58.

Comments on PCBs and Congeners by Method 8082, SW-846

68. Section 1.2.1 addresses extraction techniques for Method 8082 PCBs and specific congeners. It states that sonication may only be used for extraction of highly contaminated (free product) non-soil/sediments(debris). Any other use of ultrasonic extraction is not allowed.

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mixed with anhydrous sodium sulfate to yield a free flowing powder to increase the surface area available for sonic extraction. In this manner recoveries rival those of other methodologies. There is no mention of the concentration options which can “lose” up to 50% of some of the analytes. Some labs use K-D concentration while others use Turbo-Vap or like technologies. Others still yet finish with a simple nitrogen blowdown. These steps affect recoveries dramatically.

This method requiring the use of other extraction methods, when most laboratories (especially smaller ones) have invested in sonication devices is not warranted and unfair. For a laboratory to invest in PFE (or ASE - accelerated solvent extraction) for instance would be a \$40,000 expenditure. This requirement should not be taken lightly and should be reconsidered.

Response: See comments #12 and 58.

From the Center for Environmental Engineering and Excellence

69. Comment: The reporting limit (RL), except for the volatiles method, is actually dependent upon the extraction/digestion procedure as well as the lowest calibration standard. We suggest analyzing a low standard carried through the entire analytical procedure to verify the RL.

Response: We agree the sample preparation procedure has an influence on the ability of the method to accurately quantify analytes, especially at the low end of the calibration curve. However, we do not feel it is advisable to require laboratories to verify the RL by analyzing a low standard put through the entire analytical process every time the analytical system is calibrated.

70. Comment: Reporting of detections below the reporting limit may be of use to the data user.

Response: See The DEP expects most analytes detected below the sample specific reporting limit to be reported as not detected. However, in certain instances the data

user may wish the laboratory to report detections below the RL. In those limited cases, the results must be qualified. See comment #54.

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71. Comment: In Methods 8260 and 8270 there is no initial calibration verification standard. A second source standard should be analyzed to verify the initial calibration. Methods 8081, 8082, 8151, and ETPH all have this requirement..

Response: The QA/QC Group agrees. A second source standard will be added to the method to verify the initial calibration. This may also be used as the calibration check standard.

72. Comment: The LCS should not be a second source standard. It should be the same source as the calibration and matrix spike solutions and used to evaluate the analyst's technique and verify any matrix spike problems are sample related and not technique related.

Response: The methods will be modified so that the LCS need not be a second source standard.

73. Comment: The State of Connecticut should allow performance-based methods, as NELAC does.

Response: Thank you for the comment, but this is beyond the scope of the QA/QC Workgroup.

74. Comment: Laboratories should print out all manual integrations with the raw data and initial each page. This will help to prevent improper activities and enable data reviewers to catch such practices.

Response: Thank you for the comment, but this is beyond the scope of the QA/QC Workgroup. The QA/QC Workgroup agrees laboratories should follow this practice for all manual integrations.

75. Comment: Regarding TICs, due to the high uncertainty of TICs it would be better for laboratories to provide the mass spectra and their references. Data users could then decide on whether to further confirm the TIC or not.

Response: The QA/QC Workgroup disagrees. We feel most data users do not have the expertise to evaluate library search data.

76. Comment: On page 6 the document states "Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is not allowed". This requirement is not reasonable. The sonication extraction (method 3550) is the main method used for SVOC analysis for soil

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samples. In fact, this is the only extraction method allowed by EPA CLP methodology for SVOC analysis in soil samples. The reporting limits of the method 8270 in RRCP are the same as the contract required quantitation limit (CRQL) of CLP method, and the CLP has

the highest QC requirements. Method 3550 should be acceptable for soil analysis in the State of Connecticut.

Response: See comments #12 and 58.

77. Comment: Method 8270, on page 11 the document states that for phenolic compounds, “the SW-846 method 3510 may not be suitable for sample extraction because of known low recoveries (<25%)”. This statement is not proper. Theoretically, calculations show that at pH2 extraction for three times (sample size 1L, methylene chloride 60 mL) the extraction recoveries for all phenolic compounds in the list are higher than 95%. The lower recoveries observed by some laboratories are due to the analysts overlooking some other technical details.

Response: The QA/QC Workgroup agrees that Method 3510 can generate good data, but for certain type samples it may not be appropriate. Examples would be samples that form a severe emulsion, or otherwise cause problems with the separatory funnel procedure. In those cases alternate methods should be used.

78. Comment: On page 11 the document states that the surrogate recovery limits must be within 50-150% on both columns. For ETPH analysis no second column is necessary.

Response: Thank you for your comment. The method will be corrected.

79. Comment: Method 8260, The BFB tune criteria should be allowed to follow EPA CLP method as in method 8270. (page 18)

Response: The method allows for accepted alternate tune criteria as long as all samples, standards, blanks, etc. are analyzed under the same conditions. See item #51.

80. Comment, Method 6010, Page 14, the interference check standards (ICSA & ICSAB): The document states “2) ICSA and ICSAB containing known amounts of analytes and interferents per method”. The statement is not proper. The ICSA contains only interferents, while the ICSAB contains both interferents and the analytes. The requirement of recovery of $\pm 20\%$ of true value is applied to interference elements and analytes for ICSAB. However, for the ICSA this rule can only be applied to the interferences. The information of the false-positive or false-negative error of analytes caused by the interference elements is important, and a QC criterion should be specified for it. We suggest that for ICSA, the concentration of analytes should fall within $\pm 2 \times RL$.

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Response: You are correct and thank you for your comment. We will change the criteria for the method.

81. Comment: Method 6010, Page 14, the LCS requires spike standard from the second source. For the same reason explained in 61 we suggest spiking standard from the same source as of the initial calibration standards. (This comment also applies to Method 6020, Method 7470/7471, Method 7000, Method 7191 and Method 9010/9012/9014).

Response: LCSs will not be required to be second source standards, as long as a second source initial calibration verification is analyzed prior to sample analysis.

82. Comment: Method 6010, page 15, the document does not require performing the dilution test and the post-digestion spike addition test. The EPA method 6010 requires performing both these tests. These tests provide important information about the matrix effect on the accuracy of the analytical data of analytes. It is better to require these tests.

Response: SW-846 Method 6010B recommends the dilution test and post digest spike for new or unusual sample matrices. The QA/QC Workgroup would prefer the use of site-specific matrix spikes and sample duplicates in lieu of these tests. While these tests are good laboratory practice, they are not required under the RCPs.

83. Comment: Method 6010, Page 13, CCV, “3) Recovery \pm 10% of true value, must use at least two burns with RPD<5%”, “two burns” is not a proper terminology. It is better to use “two replicates”. Because there is no combustion in the ICP process.

Comment: Your comment will be incorporated in the method.

84. Comment: Method 6020, Page 11, the criterion of 3% for daily performance standard should be clarified. The method requires the stability check should have $RSD \leq 5\%$ not $\leq 3\%$. While the oxide and double charge levels should be $\leq 3\%$. In addition, the oxide and double charge check should be performed after tuning, not before the tuning.

Response: The criteria will be changed to reference that an instrument stability check be performed by analysis of the tuning solution 5x, either as five separate analyses or 5 integrations, with a RSD for all analytes in the tuning solution <5%.

85. Comment: Method 6020, Page 13, about interference check: as in comment 7.1.1, a QC criterion of false-positive or false-negative error of analytes should be set for ICSA. We suggest that the concentration of the analytes caused by interference should fall within $\pm 3 \times RL$.

Response: Thank you for your comment. The method criteria will be modified.

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86. Comment: Method 6020, Page 13, about LCS: requires “standard source different from initial calibration source”. The standards should come from the same source as matrix spike.

Response: See item 81.

87. Comment: Method 6020, Page 13, about LR: “each wavelength” should be “each mass charge ratio”.

Response: You are correct and thank you for your comment. The method will be modified.

88. Comment: Method 6020, Page 14, the document requires performing the dilution test, but does not require performing the post-digestion spike addition test. The EPA method 6020 requires performing both these test. The post-digestion spike test provides the information of the matrix effect on the analysis accuracy of all analytes. It is as important as the dilution test is, and should not be omitted.

Response: The QA/QC Workgroup would prefer the use of site-specific matrix spikes and sample duplicates in lieu of these tests. While these tests are good laboratory practice, they are not required under the RCPs.

89. Comment: Method 7470/7471 CVAA for mercury analysis
Page 10, MS/MSD: says “lab may substitute MSD in lieu of sample duplicate”, when using sample duplication the RPD requirement should be applied only when the concentration of mercury $\geq 5x$ RL.

Response: The method will be modified to reflect the following: The RPD limit is $\pm 20\%$ if the concentration of the analyte is $\geq 5x$ the RL. If the concentration is $<5x$ the RL, the RPD would be \pm the RL.

90. Comment: Method 7000 series Page 12, for ICV and CCV the percent differences from the true value are all required $\leq 10\%$. These requirements are tight. The EPA method allows $\leq 20\%$ (see 7000A 8.3). We think $\leq 15\%$ is more reasonable.

Response: SW-846 has different criteria for flame AAS and graphite furnace AAS. The QC criteria will be modified to contain criteria applicable for each analytical technique.

From: Envirite Analytical Services (EAS)

RCP Comments

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91. Laboratory Analysis QA/QC Certification Form

Item 1 covers all of the items in questions 2 – 7. It might be too broad of a question.

Item 5 What if no reporting limits were specified on the chain-of-custody? How is this question answered?

Response: The laboratory should contact the data user to determine what detection limits are needed. Laboratories and environmental professionals are encouraged to

utilize the Project Communication Form or other documentation to communicate project needs. The QA/QC Workgroup feels the language is appropriate.

92. Semi-volatile Organic Compounds by Method SW-846-8270

Table 1A “Method Blanks” 1) Extracted every 20 or every batch, whichever is greater. Should it read “*which ever is more frequent*”?

Response: You are correct, the method will be modified

93. Extractable Petroleum Hydrocarbons – CT-ETPH Method

Table 1.0 Typical Reporting Limits

The soil/sediment RL of 100 mg/kg is reasonable for most samples with a % solids value above 25%. The line below the table seems to indicate that the 100 mg/kg is the beginning.

Response: The reporting limits presented in the method are for guidance only. We would expect laboratories to set their reporting limits based on their initial calibration, sample weights/volumes extracted, etc.

94. Table 1A Surrogates Item 2 Recovery limits on “both columns”?

Response: You are correct, the method will be modified

Below are some of my comments with regard to VOC by method 8021 and 8260.

Method 8021

95. Page 3 paragraph 1.1.1 the line: "The following table lists approximate reporting limits for various matrices utilizing the standard quadrapole mass spectrometer." This is a GC/PID/Conductivity Detector method.

Response: You are correct, the method will be modified.

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96. Page 6 paragraph 1.2 "Quantitation is accomplished by using the response of a major ion..." This is used in an MS technique, this method requires either peak height or peak area.

Response: You are correct, the method will be modified.

97. Page 6 paragraph 1.3.2 "If a high sample is inadvertently analyzed, the system must be....the same autosampler position..." With the purging

procedure being carried out directly from the sample vials there is one line which is used to introduce the sample. This line moves from sample to sample. Therefore the system needs to be checked with a Lab Blank for contamination.

Response: You are correct, the method will be modified.

98. Page 9 paragraph 1.5.5 "Trip Blanks and Field Duplicates for SW-846 Method 8260 Analysis" Should read 8021.

Response: You are correct, the method will be modified.

99. Page 12 Table 1A (MS/MSD recovery limits 70-130% for all target compounds) these seems very tight. Maybe for compounds detected in the samples. Then set a lower limit of 40 - 50%. Requires a third spike to prove matrix effect. Report data for all three spikes.

Response: The QA/QC Workgroup has evaluated your comment and has determined that the method criteria are reasonable. MS/MSD recoveries outside the QC limits would have no impact on reasonable confidence status, but would require narration and may affect the ultimate usability of the data for its intended purpose.

100. Page 15 paragraph 1.6.1 "Additional Reporting Requirements" I agree with the statement made earlier in this procedure that said this method should be used for a "limited list of pre-selected (and known) volatile contaminants of concern at disposal sites that have been previously characterized with qualitative certainty (SW-846-Method 8260)... Therefore this section should not be in the method.

Response: This section is a general description concerning RCP requirements. The QA/QC Workgroup has determined that it should not be deleted.

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101. "In order to meet detection limits it may be necessary.....or employ selective ion monitoring." This is a conductivity/PID method not an ion detector.

Response: You are correct, the method will be modified.

102. Page 18 Table 1B Acetone, Acrylonitrile, MEK, carbon disulfide, 2-hexanone, MIBK, and THF are not amenable to this method.

Response: You are correct, the method will be modified.

Method 8260

103. When we see an impact from the bisulfate preservation the acetone and MEK are about 200 - 500 ug/kg and 50 -200 ug/kg respectively. With the RSR's at 14,000 ug/kg and 800 ug/kg are these background levels a concern?

Response: The method will be modified to reflect the new DEP guidance for soil preservation referenced in comment #4. Environmental professionals would be required to evaluate and appropriately address levels of acetone and MEK or any compound present in any sample.

104. Same comment as above for the MS/MSD recovery ranges. In this method there are more stringent criteria on the MS/MSD recoveries then the LCS. The LCS can have 10% of the TCL outside the 70 - 130% as long as they are within 40 -160%

Response: If the LCS recoveries are outside the QC requirements, the sample batch would not achieve reasonable confidence. MS/MSD recoveries outside the QC limits would have no impact on reasonable confidence status, but would require narration and may affect the ultimate usability of the data for its intended purpose.

From: Loureiro Engineering Associates

105. I noticed that the 8270 method on the EPOC website states in Table 1.2 Report Deliverables (page 19 of 28) that Internal Standards are a required deliverable for the method.

Response: Internal standards are not a required deliverable.

106. I'm not sure if this has been brought to your attention before but I just happened to notice it today so I thought I'd pass it along. The headers for all the tables in the RCP Method 9010/9012/9014 reference Pesticides by SW846 Method 8081 where it should state Determination of Total Cyanide by SW846 Methods 9010/9012/9014. This is the

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case at least for the version of this method that is posted on the EPOC website.

Response: You are correct, the methods will be modified .

From the Connecticut DEP Staff:

107. The following comments are in regard to the “Project Planning Worksheet:”

- The section of the form titled Analysis/Methods should flag the samples that are waste samples that may contain elevated concentrations of substances.

Response: The “other” box under sample matrix allows for a wide variety of materials and may include a notation if elevated concentrations are anticipated.

The communication form may be modified by the user to facilitate communication. The QA/QC Workgroup encourages all users of this form to modify the form as their needs dictate.

- The section of the form titled Analysis/Methods should note the required method and associated detection limits.

Response: It is important for the environmental professional to communicate to the laboratory what the regulatory requirements are so that the appropriate analytical methods can be used and the appropriate reporting levels achieved. Information regarding the regulatory limits are included on another section of this form.

- Do not allow detection limits to be the industrial commercial direct exposure criteria. This will not allow the environmental professional to fully define the extent of the release area.

Response: In most cases, the reporting limits achieved will be less than the regulatory criteria. This will allow the environmental professional to determine the extent of contamination.

- The section of the form titled Report Deliverables Requirements should also specify reporting formats that the DEP can use.

Response The option for report deliverables allows for many different data reporting formats.

From Lancaster Labs:

Determination of Trace Metals by SW-846 Method 6020

108. Since we check the calibration at the reporting limit (using a statistical window), mid-range (+/-10%), and at the linear range (+/-10%), is it acceptable to calibrate using a blank and a standard in the middle of the linear range as recommended by the instrument manufacturer and other agency audits?

Response: A reporting limit standard may be analyzed. The metals should be at or below the reporting limit, and the recovery would be within 30% of the true value.

109. As noted above, a statistical window is used for the evaluation of the reporting limit standard. Is this acceptable? We analyze the reporting limit standard at the beginning and end of every run.

- We'd also like to note that we analyze the linear range standard at the beginning of every run (+/- 10%). CT requires this to be performed annually.

Response: See comment 108.

Determination of Trace Metals by SW-846 Method 6010

110. A statistical window is used for the evaluation of the reporting limit standard. Is this acceptable? We analyze the reporting limit standard at the beginning and end of every run.

Response: See comment 108.

Determination of Total Cyanide by SW-846 Methods 9010/9012/9014

111. A 2nd source mid-range non-distilled standard is analyzed for the initial calibration verification and acceptance criteria of +/-10% are applied. Is this acceptable? The LCS (2nd source) is distilled and acceptance criteria of +/-10% are applied.

Response: The ICV must be distilled each day samples are distilled. When the corresponding group/batch of samples are analyzed, the ICV is analyzed. The RCP criteria is ±15%.

Determination of Hexavalent Chromium by SW-846 Method 7196

112. According to SW-846 3060A Alkaline Digestion for Hexavalent Chromium, Section 6.4 says, "Cr(VI) has also been shown to be stable in the alkaline digestate for up to 168 hours after extraction from soil." We noticed that the holding time on Table 2 for CT's version of 7196 notes "Analyze digestate within 24-hours of preparation." If this is correct, could you explain the reason for the shorter timeframe?

Response: The holding time for digestates held under the alkaline conditions as specified in the method will be 7 days.

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113. In addition, pH and ORP are listed as having a 24-hour holding time. Are these parameters being tested in the field? Due to shipping and other logistics, achieving a 24-hour hold at the lab may not always be possible. When needed, we have analyzed for these parameters at the laboratory as soon as practical. However, this is usually after the 24-hour holding time. Is this acceptable?

Response: The holding time will remain at 24 hours from collection. Holding time violations should be narrated.

Other

114. We noticed that ultrasonic extraction is only permitted in certain cases and wondered why there was a limitation as our understanding is that it is widely accepted. Is 3550B Ultrasonic Extraction acceptable?

Response: In general, sonication will not be allowed except for debris or waste samples. See comments #12 & 58.