

C205c – APPENDIX CHECKLIST: ENVIRONMENTAL CHEMISTRY TESTING LABORATORIES

May 2006

The following pages present the criteria from the NELAC Appendix D requirements in a checklist format as they relate to chemical testing. The laboratory's policies and procedures must meet these requirements. Quality system documentation and supporting records must be available for the assessor's review.

Requirement	Reference	{RESERVED FOR ASSESSORS ONLY}			
		Compliance			Comments
		Y	N	NA	
D.1.1 Positive and Negative Controls					
a) Negative Control - Method Performance Purpose: The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures shall be in place to determine if a method blank is contaminated. Any affected samples associated with a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.					
Frequency: The method blank shall be analyzed at a minimum of 1 per preparation batch. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.					

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		Compliance			Comments
		Y	N	NA	
Composition: The method blank shall consist of a matrix that is similar to the associated samples and is known to be free of the analytes of interest.					
Evaluation Criteria and Corrective Action: While the goal is to have no detectable contaminants, each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:					
1. The concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation, AND is greater than 1/10 of the amount measured in any sample.					
2. The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.					
3. When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem. Samples associated with a contaminated					
b) Positive Control - Method Performance					
1) Laboratory Control Sample (LCS)					

Requirement	Reference	{RESERVED FOR ASSESSORS ONLY}			
		Compliance			Comments
		Y	N	NA	
Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is “out of control”. Any affected samples associated with an out of control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes.					
Frequency: The LCS shall be analyzed at a minimum of 1 per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.					
Composition: The LCS is a controlled matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. NOTE: the matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods. The following shall be					

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		Compliance			Comments
		Y	N	NA	
used in choosing components for the spike mixtures:					
The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:					
For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.					
For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.					
a) For methods that include 1-10 targets, spike all components;					
b) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;					
c) For methods with more than 20 targets, spike at least 16 components.					
Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery. The laboratory shall document the calculation for percent recovery.					

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		Y	N	NA	
The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.					
A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be “out of control” should be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.					
If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A ME is defined as being beyond the LCS control limit (3 standard deviation), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.					
The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.					

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The number of allowable marginal exceedence is as follows: Analytes in LCS- Analytes in ME of LCS limit >90 5 71-90 4 51-70 3 31-50 2 11-30 1 <11 0					
c) Sample Specific Controls					
The laboratory must document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of matrix specific Quality Control (QC) samples and are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge laboratory performance.					
Examples of matrix specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); sample duplicates; and surrogate spikes. The laboratory shall have procedures in place for tracking, managing, and handling matrix specific QC criteria including spiking appropriate components at appropriate concentrations, calculating recoveries and relative percent difference, evaluating and reporting results based on performance of the QC samples.					
Matrix Spike; Matrix Spike Duplicates:					

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		Compliance			Comments
		Y	N	NA	
Purpose: Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.					
Frequency: The frequency of the analysis of matrix specific samples shall be determined as part of a systematic planning process (e.g. Data Quality Objectives) or as specified by the required mandated test method.					
Composition: The components to be spiked shall be as specified by the mandated test method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following:					
For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.					
For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2 year period.					

Requirement	Reference	{RESERVED FOR ASSESSORS ONLY}			
		Compliance			Comments
		Y	N	NA	
a) For methods that include 1-10 targets, spike all components; b) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater; c) For methods with more than 20 targets, spike at least 16 components.					
Evaluation Criteria and Corrective Action: The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). The laboratory shall document the calculation for relative percent difference.					
Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.					
d) Matrix Duplicates:					
Purpose: Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.					

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		Y	N	NA	
Frequency: The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e.g. Data Quality Objectives) or as specified by the mandated test method.					
Composition: Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.					
Evaluation Criteria and Corrective Action: The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments.					
Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix duplicates results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.					
e) Surrogate Spikes:					
Purpose: Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix.					

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		Y	N	NA	
Frequency: Except where the matrix precludes its use or when not available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.					
Composition: Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.					
Evaluation Criteria and Corrective Action: The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results.					
The appropriate corrective action may be guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria should include appropriate data qualifiers.					
D.1.2 Limit of Detection and Limit of Quantitation					
All procedures used must be documented. Documentation must include the quality system matrix type. All supporting data must be retained.					
D.1.2.1 Limit of Detection (LOD)					

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		Compliance			Comments
		Y	N	NA	
The laboratory shall utilize a test method that provides an LOD that is appropriate and relevant for the intended use of the data. An LOD is not required for a test method when test results are not reported outside of the calibration range. LOD's shall be determined by the protocol in the mandated test method or applicable regulation. If the protocol for determining LOD's is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.					
a) The LOD shall be initially determined for the compounds of interest in each test method in a quality system matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the LOD must be determined in the quality system matrix of interest.					
b) LODs must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.					
c) The laboratory must have established a procedures to relate LOD with LOQ.					
d) The LOD must be verified annually for each quality system matrix, method and analyte according to the procedure specified in C.3.					
D.1.2.2 Limit of Quantitation (LOQ)					
Any established LOQ must be above the LOD.					
The LOQ must be verified annually for each quality system					

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		Compliance			Comments
		Y	N	NA	
matrix, method and analyte according to the procedure specified in C.3. Alternatively, the annual LOQ verification is not required if the LOD is reevaluated or verified according to D.1.2.d above.					
D.1.3 Data Reduction					
The procedures for data reduction, such as use of linear regression, shall be documented.					
D.1.4 Quality of Standards and Reagents					
a) The source of standards shall comply with 5.5.6.2.2.2.					
b) Reagent Quality, Water Quality and Checks:					
1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.					
2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.					
3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.					

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		Compliance			Comments
		Y	N	NA	
D.1.5 Selectivity					
a) The laboratory shall evaluate by following the checks established within the method, which may include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical absorption or fluorescence profiles, coprecipitation evaluations, and electrode response factors.					
b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.					
c) The laboratory shall document acceptance criteria for mass spectral tuning.					
D.1.6 Constant and Consistent Test Conditions					
a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.					

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		Compliance			Comments
		Y	N	NA	
b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method. Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.					