

# Assessing Sample Matrix Effects

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# NELAC Chapter 5, Appendix D.1.1.3

- The laboratory must document procedures for determining the effect of the sample matrix on method performance.
- These procedures are designed as data quality indicators for a specific sample using the designated test method.
- These procedures relate to the analysis of quality system matrix specific QC samples (matrix spikes, matrix spike duplicates, matrix duplicates, surrogate spikes)

# Quality System Matrices

- Aqueous (surface water, groundwater, effluents, TCLP extracts)
- Drinking Water (potable water or potential potable water source)
- Saline/Estuarine (ocean, estuary, inland saltwater lake)
- Biological Tissue (fish tissue, shellfish, plant matter, grouped according to origin)
- Solids (soils, sediments, sludges, matrices with >15% settleable solids)
- Chemical Waste (product or by-product of industrial process)
- Air & Emissions (whole gas samples, vapors, or extracted concentrates from gases or vapors collected in sorbents, impingers, filters, etc.)

# Questions

- Is DI water an adequate clean matrix for:
  - Saline/Estuarine?
  - Particular lakes, rivers, aquifers?
- Should separate QC criteria & frequency be devised for:
  - Sands?
  - Silts?
  - Clays?
  - Loams?
  - Sludges?
- Should test method SOP account for differing procedures for:
  - Tedlar bags?
  - SUMMA canisters?
  - TENAX or XAD cartridges?
  - Filter Particulates?
  - Impinger solutions?
  - Soxhlet Extraction / Sonication Extraction / Solid-Phase Extraction?

# Analysis of Sample Matrix Spikes & Matrix Duplicates

- Frequency of analysis is determined:
  - By systematic planning process
  - As specified in the test method
- Components to be spiked are:
  - Specified by the test method
  - Any permit-specified analytes, as specified by regulation
  - Client-requested analytes
  - All chemistries and elution patterns of components are represented
  - All targeted components included over a 2-year period
  - 100% - 80% - >16 components

# Common Laboratory Observations

- Clients provide insufficient sample (particularly, aqueous) to perform sample matrix spikes and/or matrix duplicates.
- Matrix spikes are really “blank spikes” or control samples.
- “Randomly-selected” samples for spikes are really from dedicated sources (playground sand, backyard monitoring wells).
- Test methods are treated as “guidance”; procedures are followed but QC is ignored.
- Analyzing sample matrix spikes and matrix duplicates are a lot of work that does not make money for the laboratory. The client won't pay for QC.

# Selection of Test Methods

- The laboratory shall use methods for environmental testing which meet the needs of the client & are appropriate for the environmental tests it undertakes.
- Methods published in international, regional, or national standards shall preferably be used.
- When the use of specific methods are mandated (e.g., by regulation) or requested (e.g., by the client), only those methods shall be used.

# Review of Requests, Tenders, & Contracts

- The policies & procedures leading to a contract for environmental testing shall ensure that requirements, including methods to be used, are adequately defined, documented, & understood.
- The appropriate environmental test method is selected and capable of meeting the client's requirements.
- Any differences between the request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the client.
- Records of reviews, including any significant changes, shall be maintained.
- The client shall be informed of any deviation from the contract.



# Other Considerations

- The sample acceptance policy shall include the following areas of concern:
  - Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests.
- Test reports shall, where necessary for the interpretation of the test results, include the following:
  - Deviations from, additions to, or exclusions from the test method.
  - Where quality system requirements are not met, a statement of compliance / noncompliance with requirements and/or specifications, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements.

# Conclusions

- If the clients wants samples analyzed by EPA 8270, and the laboratory agrees, the laboratory is obligated to perform sample matrix spikes.
- Do not claim to be running any EPA Methods, such as 8270, when there is no intention on the part of the laboratory to perform sample matrix spikes.
- If the test method specifies that 10% of samples must be spiked and analyzed, then a matrix spike & matrix spike duplicate on the same sample in a 20-sample batch does NOT meet that requirement.

# Disclaimers

- If matrix spikes are not performed, ALL of the following must be documented on file at the laboratory:
  - Laboratory records for the client must indicate choice of test method other than EPA 8270, or client-authorized deviation from EPA 8270 requirements.
  - Laboratory-authorized departure from NELAC quality system and test method SOP requirements.
  - Sample receipt records must document non-conformance with sample acceptance requirements, and document client authorization to proceed with analysis despite those shortcomings.
  - EPA 8270 analysis benchsheets must document departure from sample matrix spike requirements, when EPA 8270 is documented as the test method.
  - Test reports for ALL clients with samples within the affected batch must document departure from EPA 8270 requirements, with statement of test results being invalid for regulatory compliance as applicable.



SO... WHAT  
DO YOU  
THINK?