

Quality Assurance and Data Usability

David E. Splichal,
ERDC-EL-ECB-Omaha



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Environmental Chemistry Branch Resources

- Personnel: 28 Federal Government employees, 5 contract employees , 12 contract students
- Equipment: \$ 5.2 M including all major routing analytical equipment plus specialized equipment such as ICP/MS, LC/MS, etc.
- Locations: Vicksburg (22,000 sq ft) and Omaha (44,000 sq ft)
- One Branch Chief in Omaha, three team leaders in Omaha, and one team leader in Vicksburg



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Questions

- Is it possible to transfer prescribed fixed laboratory Quality Control procedures (ensuring data of high quality?) to LTM field test procedures?
- Can the end user get adequate data quality to meet the goals of the data gathering activity?



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Laboratory Methods

- Office of Solid Waste, SW-846.
- Contract Laboratory Program (CLP)
- Drinking Water (500-series).
- Performance Based Measurement Systems (PBMS)



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Why SW-846?

- Comprehensive for various media and chemical parameters.
- Adaptable to individual project specific requirements.
- Use of “Shell for Analytical Chemistry Requirements” (EM 200-1-3, Appendix I) – baseline implementation.



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PBMS

- This approach empowers the analytical service (data) provider with the flexibility to vary aspects of the analytical system and protocols as long as the demonstrated method performance meets the requirements established by the data user.
- Most likely the best approach for the LTM program (work with all parties involved to produce data of known quantity and quality). Need comprehensive SOP's.



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Analytes of Concern

- Lab – long lists of analytes per test method.
VOA – 66
BNA - 77
Pesticides – 21
Explosives – 14
- LTM – Very short list (1-2 “TARGET”) analytes.



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Quality Control – SW846 Chapter 1

- It is the goal of the USEPA's Quality Assurance program to ensure that all data be scientifically valid, defensible and of known precision and accuracy. The data should be of sufficient known quality to withstand scientific and legal challenge relative to the use for which the data are obtained.
- The data acquired from QC procedures are used to estimate the quality of analytical data, to determine the need for corrective action in response to identified deficiencies and to interpret results after corrective action procedures are implemented.



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QC Components

- Calibration
- Method Detection Limits
- Blanks
- Matrix/Field Duplicates
- Matrix Spikes/Matrix Spike Duplicates
- Control Spikes
- Surrogates (organics)



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Calibration

- 5 Point Initial Calibration Curve
 - checks sensitivity and linearity.
 - lowest standard set at Reporting Limit (for field this should be set at 0.5 to 0.1-times less than an action limit).
- Independent Calibration Verification check
 - checks accuracy of primary standards.
 - ensures no dilution errors in preparation of primary standards.



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Method Detection Limits

- “The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte”, (normally 7-replicates).
- MDL check – spiked at approximately 2X the MDL – verifies that sensitivity is still adequate.
- DO WE REALLY NEED THIS FOR THE LTM PROGRAM?



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Method Blank

- “Document contamination resulting from the analytical process”.
- Should be <<<< site action limits.
- Can lead to false positives.



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Duplicates

- Matrix – Intralaboratory split sample which is used to document the precision of a method in a given sample matrix.
- Field – Independent samples which are collected as close as possible to the same point in time and space. Analyzed independently. Useful in documenting the precision of the sampling process.



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Spikes

- Matrix Spike – aliquot of sample spiked with a known concentration of target analyte(s). Documents the bias of a method in a given sample matrix.
- Matrix Spike Duplicate – Intralaboratory split sample spiked with identical concentrations of the target analyte(s). Documents both the bias and precision of a method in a given sample matrix.



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Control Spikes

- A known matrix (preferably “clean”, with no interferences) spiked with the analyte(s) of interest. Used to document laboratory performance.
- Should get very good recoveries.



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Surrogates

- An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in the environment.
- Common – isotopes of target analytes. For example, toluene and toluene-d8.
- Serves as a “quick” check on process (sample specific).



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Data Review

- Analyst: Compare to SOP's and DQO's.
- Independent: Compare to SOP's and DQO's.
- Other guidance (National Functional Guidelines, etc.)
- QA split data comparison.



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Data Usability

- Do the procedures used (from “where” sampling points are located to the final number/result produced) answer the following question:

IS THE DATA ANY GOOD??



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Answer

- Based upon some set of technical specifications derived from the specific objectives of the project, analytical performance can be “demonstrated”

at the Concentrations of Concern
for the Analytes of Concern
in the Environmental Media of Concern.



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Standard Operating Procedures (Shell I.4.5)

- Fully detail the actual procedures and documentation used to implement performance-based (and guidance based) methods.
- Includes approximately 20 sections ranging from summary to health-and-safety issues. Example calculations also included.
- Must be signed (and read!!) by appropriate personnel.
- Updated as needed (yearly recommended).



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Summary of Lab Analyses

- Fixed laboratory analyses are complicated requiring chemists with working knowledge of the methods (and a sense of humor!).
- Lots of methods in use, many lists of compounds – makes it hard for the analyst to look for the right thing sometimes!
- New techniques are always being presented – which are money makers?



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Discussion Points

- Fixed laboratory QC procedures are many. Begg the question “what happens if something fails”? How are correct acceptance limits set?
- How many QC parameters are REALLY needed to satisfy the end users of LTM data? How many are practical?
- Do LTM projects need a percentage of samples to also be analyzed in a fixed lab?



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