# A CASE STUDY USING A COMPARATIVE TIERED VALIDATION SCHEME

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## ABSTRACT

While data validation is an important step in ensuring data quality and usability, many project managers are reluctant to include validation due to the perceived cost and time requirements. When validation is not performed, the issue is sometimes forced by the regulatory oversight agencies, or by potential litigation. The 'after-the fact' validation effort is often more expensive and time consuming, as the laboratory has moved on to other samples, and the validator must gather and understand all project requirements prior to validating the data. This can have a significant impact on project budgets and deadlines, especially for large projects.

This paper describes such a situation, and details the tiered validation scheme used to quickly review all of the data, while the most intense efforts were focused on data with potential quality or usability issues. The tiered approach was a combination of limited and full review, using both electronic and manual validation. The tiered validation satisfied the regulatory requirements, while providing the client with a rapid turnaround at a reduced cost. An additional benefit was that the electronic validation process created a database of validated data that was used for trend analysis, risk assessment calculations, and to identify data gaps for the Phase II investigation.

## INTRODUCTION

Working on a base realignment and closure (BRAC) investigation, a project manager inherited a large site that had already undergone several years of investigation using the US Army Environmental Center's (USAEC, formerly the US Army Toxic and Hazardous Materials Agency, or USATHAMA) quality assurance program. All analytical data were generated using USAEC methods. Oversight responsibility for the project had passed from the USAEC to state and federal agencies, including the US Environmental Protection Agency (USEPA). The next step of the project was a risk assessment, however, since the data were not produced using USEPA recognized analytical methods or reporting procedures, the oversight regulatory agencies required that prior to acceptance of the risk assessment, a full validation be performed on 100 percent of the data, demonstrating data quality and comparability to data produced using USEPA protocols.

The potential cost implications to the project were significant—there was a large volume of data (over 300 data packages), some of which were several years old. Many of the data packages were archived, and there was very limited backup documentation (or technical assistance) available from the laboratory. The validation was also made difficult by the different approach used by the USAEC quality assurance program. The USAEC methods use a statistical approach to quality control (QC) that monitors quality on a laboratory specific basis (one set of methods for each laboratory if a laboratory passes certification and meets certain general QC requirements, all sample results are assumed acceptable), rather than the USEPA methods which control quality on a more global, program basis (one set of methods for all laboratories sample quality monitored through various sample or batch specific QC elements). Finally, the data packages (in the USAEC format) did not include any summary forms, such as surrogate recoveries, spike recoveries, blank association, etc. Most of the data were presented 'as is' from the instruments.

## DATA COMPARABILITY

The first step was to determine the comparability of the data produced by the two different quality assurance systems. Methods from both systems (USAEC and USEPA) were broken down into QC elements, such as calibration, precision, accuracy, contamination, etc. A table listing the QC elements (and the organization responsible for determining the accuracy of the reported information) was created, and is presented as Appendix A. Using methods from both systems, cross reference tables were created that indicated how the methods addressed each QC element, producing data of known and defensible quality. An example cross reference table is included as Appendix B to this report.

As can be seen by the tables, a comparison of several USEPA methods to the USAEC methods indicated that the same QC elements were addressed by each method. The same fundamental building blocks were used by both programs, and both programs set up a framework that is used to produce data of known quality. The

differences were in the initial approach, the reporting style, and some of the specific acceptance criteria. To fully assess the quality of the data, it was decided to validate the data against the requirements of each program, and then compare the data quality (as determined by volume of qualified or non-useable data). The final determination of data quality was based on whichever program used a more conservative approach for a given QC element.

The findings of this review were submitted to the regulatory oversight agencies, both as a technical memorandum and as an oral presentation. The agencies agreed to wave the requirement for 100 percent full validation in favor of the tiered validation approach proposed for this project.

## TIERED DATA VALIDATION APPROACH

To mitigate the cost and time impacts on the project, a two tiered validation scheme was developed. Tier 1 was a combination of electronic and manual review, which produced a validation level of effort equivalent to a USEPA Level 3 review. The electronic review utilized the electronic data deliverable (EDD) required by the USAEC for all analyses, and a Data Quality Screening Tool (DQST) program. One hundred percent of the data were evaluated (electronically and manually) for these quantitative QC elements (when appropriate for a method):

- Sample index
- Holding times
- Blank contamination
- Reporting limit verification
- Blank spike percent recovery
- Surrogate percent recovery
- MS/MSD percent recovery
- MS/MSD RPD values
- Field duplicate RPD values
- Laboratory duplicate RPD values
- Target analyte list verification

Initial and continuing calibration results, instrument tuning (GCMS) and internal standard areas (GCMS) were not provided on the EDD from the laboratory therefore, these QC elements were evaluated manually, using the hard-copy data package. The Tier 1 provided a rapid review of all of the data to identify any potential problem areas.

Tier 2 was defined as a full validation, equivalent to an USEPA full data validation, as defined by National Functional Guidelines. The Tier 2 validation included all of the Tier 1 elements and a complete evaluation of all raw data, including:

- Completeness of laboratory documentation for sample receipt, sample analysis, and sample result reporting.
- Overall documentation practices.
- Presence and completeness of chain-of-custody documentation.
- Instrument performance, and tuning.
- Compound identification and quantification.
- Review of calculations
- Transcription check (from raw data to final results)

The Tier 2 review also included a verification of the electronic results (raw data was compared to results generated by the DQST, and to a percentage of the reported sample results in the database).

Several criteria determined which data packages were subjected to a Tier 2 review. The first criterion was the projected end-use of the data. The second criterion was that a sufficient percentage of all data packages, representative of the entire project, were selected for Tier 2. The third criterion was that any data package identified as 'critical' by the Tier 1 process was included in the Tier 2 review. A 'critical' package was defined as any data package that had more than 5 percent of the QC elements (such as surrogate/spike recoveries, relative percent difference [RPD] values, blank contamination, etc.) outside the control limits. This ensured that any package that would potentially result in a large number of qualified (or rejected) data points was subjected to the most thorough scrutiny by the validation chemists.

## DATA QUALITY SCREENING TOOL (DQST)

The DQST is an electronic validation tool developed by EcoChem, Inc., and is similar to other data evaluation programs (such as CADRE). Translation modules were developed to accept EDD specific to the USAEC IRDMIS (Installation Restoration Data Management Information System) format. The DQST performed a validation level-of effort similar to an EPA Level III validation, with the exception that instrument calibration and internal standard areas are not reviewed by the DQST because this information was not specified in the IRDMIS EDD (transfer file). The DQST compared the data to both the USAEC and USEPA acceptance criteria, and identified data points that are non-compliant. Modules containing criteria from additional project or agency QA programs can also be run.

The DQST accepted information downloaded from IRDMIS or from the IRDMIS transfer files, and converts the data into a form usable by the DQST. The data were then sorted according to QC elements, and each QC element is run through a subroutine that compared the reported data points to lists of previously input criteria. The DQST created a sample index and holding times table, tabulated all blank contamination, and reported obvious transcription errors (incorrect analyte names, a reported concentration with a 'less than' designation, etc.). The DQST calculated and tabulated surrogate and spike recoveries, and also duplicate analysis RPD values. The DQST used QC codes to identify duplicates and differentiated between field duplicates and other duplicates.

Once the DQST completed all the subroutines, the results were printed out as a series of tables and suggested qualifiers were listed on an Electronic Qualifier Action Table. A flow chart of the DQST process is included as Appendix C. The DQST results, qualifiers, and additional manual validation elements were reviewed by a qualified chemist to complete the Tier 1 review. The chemist then determined if the data set required an additional tier of review.

After all validation was complete, quality assessment reports were generated by each chemist. The final data qualifiers were added to the database, and all work was peer-reviewed. Deliverables to the client included the final database, validation reports specific to each study area at the site, and tables of qualified data sorted by method and study area.

## CLIENT BENEFITS

The tiered validation scheme provided the following benefits to the client:

- Rapid review of a large volume of data
- Reduced project costs
- Focused review of each method to determine if systematic errors existed
- Focused review of data with potential usability or quality issues
- Comparison of two quality assurance systems

There were also additional benefits that became apparent as the project progressed beyond the data validation. During the electronic validation, the DQST created a database of all sample results. This database was updated after completion of the validation, providing the client with a fully validated, qualified set of all site data. This database was used to perform in-depth analysis of any data trends (both site-wide or specific to a certain study area), a comparison of positive results to risk based levels, and also to perform some of the calculations required for the risk assessment. Since the risk assessment identified several potential data gaps, a Phase 2 investigation was begun by the client. The tiered validation approach was included in the Phase 2 Quality Assurance Project Plan.

## SUMMARY

The use of a tiered validation approach can allow for a rapid review of all data, with a focus on any data or areas of concern, while controlling project costs. The use of electronic data validation is integral to this effort, and can provide additional benefits due to increased access and control of the analytical data.

## APPENDIX A

## CONTENT AND FORMAT FOR TYPICAL DATA QUALITY ASSESSMENT REPORT (QC ELEMENTS)

| Criteria Reference | Element (From USAEC QA or Method) | Reviewed or Verified |
|---|---|---|
| PAM 11-41[a] Sec. 10.5 | Data package completeness & document control | Lab/Validator |
| PAM 11-41 Sec. 7.5 | Chain-of-Custody—Transcription of Field ID and Audit Trail | Lab/Validator |
| PAM 11-41, Sec. 11.5.1 & Method | Holding Time Verification | Lab/Validator |
| NFG[b] & QAPP | Field QC Sample Evaluation<br>• Field Duplicate<br>• Rinsate (Decon) Blanks<br>• Field Blanks<br>• Trip Blanks | Validator |
| Method | Mass Calibration/Tuning Evaluation | Lab/Validator |
| PAM 11-41, pp. 71 & 77-79 | Initial Calibration and Calibration Checking Standards | Lab/Validator |
| PAM 11-41, pp 76-77 | Daily Calibration | Lab/Validator |
| NFG | Internal Standards (if appropriate) | Lab/Validator |
| NFG & Method | Method Blank Evaluation | Lab/Validator |
| NFG & Method | System Monitoring Compounds (surrogate spikes if appropriate) | Lab/Validator |
| PAM 11-41, Sec. 11.5 | Evaluation of Precision and Accuracy and Subsequent Non-Conformances<br>• Control Charts | Lab/USAEC/Validator |
| NFG & Method | Matrix Effects Evaluation (MS/MSD) | Validator |
| NFG | Compound Identification | Validator |
| PAM 11-41 & Method | Compound Quantitation and Certified Reporting Limits (CRL) | Lab/Validator |
| NFG & Method | Tentatively Identified Compounds (TIC) Evaluation | Lab/Validator |
| PAM 11-41, Sec. 10.8 | Transcription—10% within each lot<br>• Worksheets/Notebooks to Instrument Printouts<br>• Standard & Sample preparation & injection records to inst. output to ensure that each output is associated with correct sample<br>• Worksheets/notebook pages must be initiated, dated and explanation for changes<br>• Transfer File (Level I results) to record and group check results to analysis results | <br>Lab/Validator<br>Lab/Validator<br><br><br>Lab/Validator<br><br>Lab |
| PAM 11-41, Sec. 10.8 | Calculation Verification<br>• Field sample and QG sample results (10% unless problem noted, then 100% until resolved)<br>• Spike recovery and %RSD calculations | Lab/Validator |
| QAPP | Evaluation of System Process Control (control charts) and relation of "lot" control to site DQO (Data Quality Objectives).<br>NOTE: USAEC/CLIENT/Lab look for "valid" data points (data entry) on a lot by lot basis. | Validator |

**Note: Not all of the above QC elements will be appropriate to every type of analysis.**

[a] *US Army Toxic and Hazardous Material Agency Quality Assurance Program* (1/90)
[b] *USEPA National Functional Guidelines* (2/94)

# APPENDIX B

## METHOD CROSS REFERENCE COMPARISON TABLES
## VOLATILE ORGANIC ANALYSIS METHODS

| PROCEDURE | METHOD 524 | METHOD 624 | METHOD 8240/8260 | EPA CLP SOW 3/90 OLM01.0 | WATER: METHOD UM21 12/22/92 VERSION 4A | SOIL: METHOD LM23 12/22/91 VERSION 3A | METHODS UM 21 AND LM 23 (ADDITIONAL LAB PROCEDURES) |
|---|---|---|---|---|---|---|---|
| **Holding Time** | 14 days (Preserved) | 14 days | 14 days (Preserved) 7 days (Unpreserved) | 10 days from VTSR | 14 days (Preserved) 7 days (Unpreserved) | 7 days from sampling Analysis hold time 14 days from sampling | |
| **Tuning** | | | | | | | |
| • Requirement | 50 ng BFB | 50 ng BFB | 50 ng BFB | 50 ng BFB | 50 ng BFB | 50 ng BFB | |
| • Frequency | 8 hours | Daily | 12 hours | 12 hours | 12 hours | 12 hours | |
| • Criteria | Table 3 | Table 3 | Table 3 | Table 3 | same as EPA 624 | same as EPA 8240 | |
| **Initial Calibration (IC)** | | | | | | | |
| • Requirement | All target analytes | All PP analytes | All target analytes | All TCL analytes | Target analytes and surrogates | Target analytes and surrogates | |
| • Levels | 3-5: near DL-upper end | 5: near DL-upper end | 5: near DL-upper end | 5: 10, 20, 50, 100, 200 µ/L | 5: 10.0, 20.0, 50.0, 100.0, 150.0 µg/L | 5: 10.0, 20.0, 50.0, 100.0, 150.0 µg/L | |
| • Frequency | Initially, or when CC fails | Initially | Initially, or when CC fails | Initally, after major instrument maintenance, or when CC fails | Initally (berfore certification or analysis of field samples), or Instrument startup, or different analytes, or daily calibration fails | Initally (berfore certification or analysis of field samples), or Instrument startup, or different analytes, or daily calibration fails | |
| • Criteria RRF | NS | NS | 5 SPCC > 0.300; Bromoform > 0.250 | All > 0.01; Most > Min Value | NS | NS | |
| %RSD | All < 35% | All <35% | 6 CCC < 30% | Most < 20.5% | NS | NS | |
| | or generate second or third order linear regression curve | or plot a calibration curve | | | Curve linearity determined by lack-of-fit; zero intercept tests, and least squares linear regression, 2/3 analytes must pass calibration | Curve linearity determined by lack-of-fit; zero intercept tests, and least squares linear regression, 2/3 analytes must pass calibration | |
| **Continuing Calibration (CC)** | | | | | | | |
| • Requirement | Mid-level standard | 20 µg/L QC Check | Midpoint standard | 50 µg/L standard | 50.0 µg/L | 50.0 µg/L | |
| • Includes | All target analytes | All PP analytes | All target analytes | All TCL analytes | Target analytes and surrogates | Target analytes and surrogates | |
| • Frequency | 8 hours | Daily (usually 12 hours) | 12 hours | 12 hours | Before and after 12-hr. sample analyses | Before and after 12-hr. sample analyses | |
| • Criteria RRF | NS | NS | 5 SPCC > 0.300; Bromoform > 0.25 | All > 0.01; Most > Min Value | NS | NS | |
| %D | < ± 30% | Method QC limits | 6 CCC < ± 25% | Most < ± 25.0% | 2/3 analytes < ± 25% of IC 50.0 µg/L std. | 2/3 analytes < ± 25% of IC 50.0 µg/L std. | |
| IS area | ± 30% of last CC or ± 50% of IC | NS | -50% to +100% of last CC | -50% to +100% of last CC | NS | NS | |
| IS RT | NS | NS | ± 30 sec of last CC | ± 30 sec of last CC | NS | NS | |
| **Method Blank** | | | | | Standard matrix (ASTM Type I) method blank | Standard matrix (site background soil) method blank | |
| • Frequency | Daily | Daily | 12 hours | 12 hours | 1 per lot (20 samples/lot) | 1 per lot (20 samples/lot) | |
| • Criteria | Analytes < MDL | Interference free | Interference free | $CH_2Cl_2$, acetone, MEK < 5x CRQL; All others < CRQL | Interference free < CRL | Interence free < CRL | |
| **Spike** | Blank spike--All target compounds and surrogates | Matrix spike using EPA QC check solution | Matrix spike--5 compounds plus surrogates | Matrix spike--5 compounds plus surrogates | Standard matrix (ASTM Type I) method blank/spike--all surrogates spiked | Standard matrix (site background soil) method blank/spike--all surrogates spiked | Additionally, lab performs standard EPA SOW MS/MSD for Army work |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| • Frequency | Daily or 5% samples | 5% samples | 5% samples | 5% samples or once per SDG | 1 per lot (20 samples/lot) | 1 per lot (20 samples/lot) | NS |
| • Concentration | 0.2-5 µg/L | 20 µg/L or 1-5 x MDL | 20 µg/L or 1-5 x MDL or 10 x PQL | 50 µg/L for 5 analytes | Approx. 10 x CRL (surr. cmpds. only) | Approx. 10 x CRL (surr. cmpds. only) | EPA CLP SOW levels |
| • Criteria | 80-120% recovery | Method % Rec limits | Method % Rec limits | Method % Rec limits | Accuracy is control chart dependent using Dixon's outlier test | Accuracy is control chart dependent using Dixon's outlier test | EPA CLP SOW criteria |
| **Duplicate** | Blank spike dup | Not required | Matrix spike dup | Matrix spike dup | | | Matrix spike dup |
| • Frequency | Quarterly | Not required | 5% samples | 5% samples or once per SDG | Not required | Not required | NS |
| • Criteria | % Rec 80-120%; RSD < 20% | Not required | Method QC limits | Method % Rec and RPD limits | NS | NS | EPA CLP SOW criteria |
| **Sample Analysis** | | | | | | | |
| • Qualitative ID | RT within ± 30 sec of standard RT | RT within ± 30 sec of standard RT | RRT within ± 0.06 RRT units of standard RRT | RRT within ± 0.06 RRT units of standard RRT | RRT within RT windows (± 3 x standard deviation of average RRT in calibration standards) | RRT within RT windows (± 3 x standard deviation of average RRT in calibration standards) | |
| • | 3 characteristic ions in std. Present in sample within ± 20% relative intensity | 3 characteristic ions in std. Present in sample within ± 20% relative intensity | Ions > 10% in std. Present in sample within ± 20% of ion abundance in std. | Ions > 10% in std. Present in sample within ± 20% of ion abundance in std. | Characteristic ion | Characteristic ion | Lab uses EPA CLP SOW identification criteria |
| • IS area | NS | NS | NS | -50 to +100% of CC area | NS | NS | -50 to +100% of CC area (no action for outliers for Army) |
| • IS RRT | NS | NS | NS | ± 30 sec of CC RT | RRT within RT windows (± 3 x std. dev. of average RRT in calibration standards) | RRT within RT windows (± 3 x std. dev. of average RRT in calibration standards) | |
| • Surrogate Criteria | 80-120% recovery | Statistically generated from laboratory results | Method % Rec limits | Method % Rec limits | Accuracy is control chart dependent using Dixon's outlier test | Accuracy is control chart dependent using Dixon's outlier test | Method % Rec limits (no action for outliers for Army) |
| • Quantitative | Within calibration range | Within calibration range | Within calibration range | Within calibration range | Within calibration range. Upper limits in Sec. II.C. | Within calibration range. Upper limits in Sec. II.C. | |
| **QC Check Sample** | External source | 20 µg/L check standard | Laboratory control sample | Performance evaluation sample | None (other than blank/spike) | None (other than blank/spike) | |
| • Frequency | Quarterly | 5% sample | Each sample batch | Each sample delivery group | (1 per lot) | (1 per lot) | |
| • Criteria | Specified QC limits | Method QC limits | Specified QC limits | EPA QC limits | (Accuracy is control chart dependent using Dixon's outlier test) | (Accuracy is control chart dependent using Dixon's outlier test) | |
| **Initial Demonstration of Competency** | | | | | | | |
| • Requirement | 4-7 replicate spikes at 0.2-5 µg/L | 4 replicate spikes at 20 µg/L | 4 replicate spikes at 20 µg/L | Performance evaluation samples | Certification | Certification | |
| • Frequency | Initial, one-time | Initial, one-time | Initial, one-time | Pre-award | Initial, one-time | Initial, one-time | |
| • Criteria | % Rec 80-120%, RSD < 20% | Method % Rec and SD limits | Method % Rec and SD limits | EPA QC limits | Certification, performance sample, and QA/QC plan results acceptable (PAM Fig. 5-1) | Certification, performance sample, and QA/QC plan results acceptable (PAM Fig. 5-1) | |
| **Method Detection Limit Determination** | Required | Required | May be required for specific matrices | Not Required | Determined during certification | Determined during certification | |
| **Other QC** | Field blanks | Field duplicates | Equipment blanks, trip blanks, field duplicates | Trip blanks, storage blanks, field duplicates | Field blanks, trip blanks, rinse blanks, field duplicates as per Project Workplan. 1 per 20 or 1 per lot (whichever is greater). | Field blanks, trip blanks, rinse blanks, field duplicates as per Project Workplan. 1 per 20 or 1 per lot (whichever is greater). | |

## APPENDIX C

## DATA QUALITY SCREENING TOOL PROCESS FLOW