Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use

This page left intentionally blank.

OSWER No. 9200.1-85
EPA 540-R-08-005
13 January 2009
www.epa.gov

Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use

U.S. Environmental Protection Agency
Office of Solid Waste and Emergency Response
Washington, DC 20460

This page left intentionally blank.

NOTICE

The policies and procedures set forth here are intended solely as guidance to the United States Environmental Protection Agency (EPA), other government employees and contractors. This guidance does not constitute rulemaking by the EPA, and cannot be relied upon to create a substantive or procedural right enforceable by any party in litigation with the United States. EPA may take action that is at variance with the policies and procedures in this guidance and may change them at any time without public notice.

This page left intentionally blank.

Table of Contents

Page

1.0 Background **1**

2.0 Scope of this Guidance **1**

3.0 Context for Superfund Laboratory Analytical Data Verification and Validation **3**

4.0 Recommended Approach to Laboratory Analytical Data Verification and Validation **4**

5.0 Describing the Stages and Processes Used to Verify and Validate Laboratory Analytical Data **5**

6.0 Communicating the Stages and Process Used for Laboratory Analytical Data Verification and Validation **6**

7.0 Conclusion **7**

8.0 References **7**

Appendix A: Laboratory Analytical Data Verification and Validation Stages and Checks: Description, Order, and Labeling of Validated Laboratory Analytical Data Packages **8**

Appendix B: Recommended Terminology and Labels for Communicating the Stages and Processes Used for Laboratory Analytical Data Verification and Validation **14**

Appendix C: Suggested Narrative(s) for Laboratory Analytical Data Package Verification and Validation "Summary" Section(s) **15**

Appendix D: Example Laboratory Analytical Results Electronic Spreadsheet with Analytical Data Package Validation Stage Column **16**

Appendix E: Glossary of Terms **17**

This page left intentionally blank.

ED_002345B_00034353-00008

1.0 Background

Each year, well over $25 million are spent through Superfund contracts to analyze soils, water, sediments, and other media for the presence of contaminants of potential concern. Additional resources are often used by the U.S. Environmental Protection Agency (EPA) to review (i.e., verify and validate) the resulting laboratory analytical data packages. These reviews are conducted in part to ensure that data produced in support of EPA's environmental decision making are of adequate quality and usability for their intended purpose (see CIO 2105.0). Since there are often different procedures used to evaluate laboratory data quality in different EPA organizations and through different EPA contracts, the manner in which the results of these reviews are communicated to decision-makers may also vary. Such variability may create problems when data sets developed by different organizations or contractors are evaluated together in support of a particular site activity (e.g., when data are gathered over long periods of time; or when data are gathered quickly by multiple groups in support of a time-critical response action). Because of this potential variability, and because of the complex nature of commonly used analytical data verification and validation procedures, it is important to minimize ambiguity in communicating the nature of the procedures used for laboratory data reviews to data users. This guidance is designed to help increase national consistency and improve communication and understanding about the nature of verification and validation conducted on laboratory analytical data for Superfund use.

The attached guidance recommends the use of consistent terminology by external data reviewers in describing the scope and content of verification and validation conducted on laboratory analytical data packages developed in support of Superfund site activities. Through the use of this guidance, EPA decision makers should be readily able to determine which analytical data verification or validation procedures have been performed on each laboratory analytical data package regardless of which region, program office, or contractor provided the review.

Note: Analytical *data verification* by an external party generally consists of a completeness check to confirm that all data requested from the laboratory have been received and comply with specified requirements. Analytical *data validation* by an external party generally consists of an analyte and sample specific process for evaluating compliance of the laboratory data received with methods, procedures or contract requirements. Definitions of terms (including verification and validation) used in this guidance are given in the Glossary (Appendix E). Generally, this guidance uses terms consistent with other EPA documents and guidance.

2.0 Scope of this Guidance

This guidance focuses on the data verification and validation of chemical and radiochemical laboratory analytical data by external parties. For the purposes of this guidance, external parties are defined as organizations (including Governmental entities, contractors, or vendors) that conduct analytical data review, verification, and validation activities and that are not part of the immediate laboratory that generates the subject analytical data (but that are part of the overall project-specific data review process).

ED_002345B_00034353-00009

This guidance draws on generally accepted procedures used in the verification and validation of laboratory analytical data generated for chemical and radiochemical parameters. EPA Regions are encouraged to use this guidance in conjunction with the EPA Contract Laboratory Program National Functional Guidelines (NFGs) for Data Review, EPA Regional guidance, contract, or method-specific data validation documents (e.g., Multi-Agency Radiological Laboratory Analytical Protocols Manual). The types of review addressed by this guidance correspond to the analysis verification and validation portion of the data review steps given in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) Manual (IDQTF 2005). The most appropriate approach for verifying and validating a particular analytical data set typically will depend on many factors, including the goals of the specific data collection activity, the nature and completeness of the data package received (hard copy and/or electronic), and available program resources (time and/or personnel).

This guidance does not address or discuss the evaluation of the sampling portion or overall usability (e.g., data usability assessments outlined in the UFP-QAPP Manual) of laboratory analytical data sets. Nor does it attempt to address the verification and validation of all types of analytical data (e.g., this guidance does not address data collected from field sampling measurements like pH and conductivity, or microbiological methods). However, if the appropriate quality control documentation and information is provided and defined, this guidance may be useful in labeling the scope and content of verification and validation performed on analytical data generated by less traditional means such as field analytical methods (e.g., X-ray fluorescence, image analysis, immunoassay methods, direct sensing, etc.).

In addition, this guidance does not address the following:

- Specifications and types of Quality Assurance Project Plans (QAPPs), method, procedural, or contract requirements;

- Specific actions taken to determine analytical data quality based on comparing the laboratory reported data to any QAPP, method, procedural, or contract requirements;

- Validation qualifiers used to qualify the analytical data;

- Exact procedures used to carry out these analytical data verification and validation checks;

- The appropriate "independence" of the external parties carrying out the analytical data validation;

- Reporting requirements (hardcopy and/or electronic);

- Appropriate scope and content (e.g., levels, tiers, or stages) of analytical data verification or validation required for specific site decisions; or

ED_002345B_00034353-00010

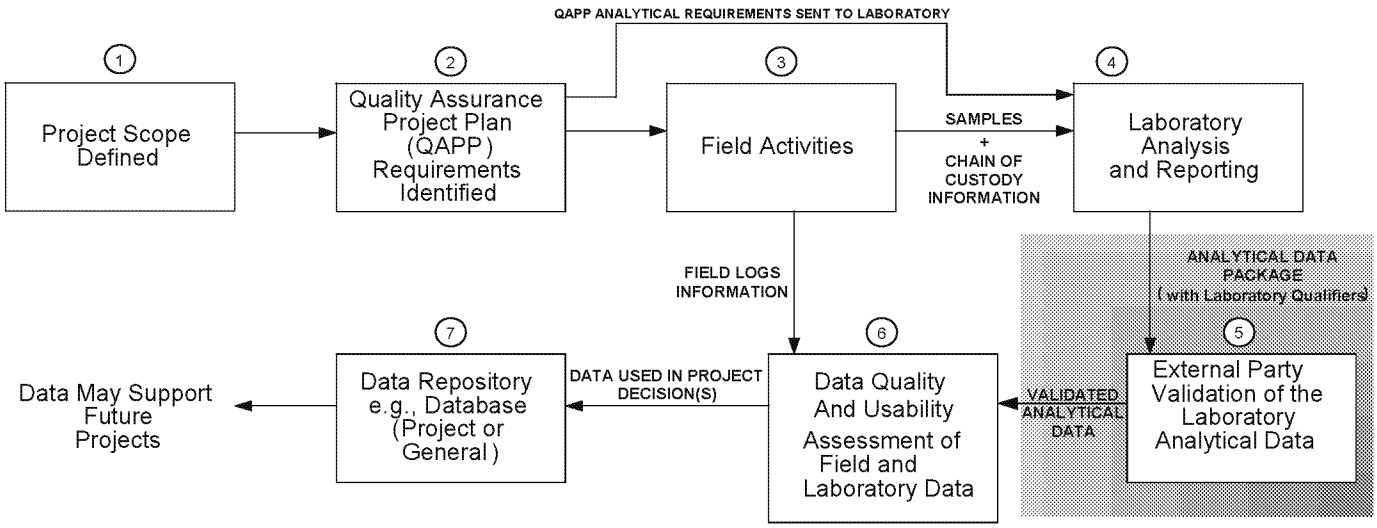
- Routine internal analytical data verification, validation, or review procedures used by laboratories that generate analytical data.

3.0 Context for Superfund Laboratory Analytical Data Verification and Validation

Figure 1 presents the broader context in which analytical data verification and validation by external parties typically occurs. The shaded area depicts the part of the process that is the focus of this guidance.

Since the collection and review of environmental information is often expensive, data are generated typically only when a specific decision requires it (e.g., to define the nature and extent of contamination; to determine the need for emergency or remedial action; to evaluate the effectiveness of cleanup technologies, or progress in the remediation of contaminated media). Therefore, the first recommended step in this process is for the anticipated data users to scope the nature of the decisions of interest and the related data requirements (Figure 1, Step 1). A project-specific QAPP should be developed to document the procedures for the collection, review, and use of the environmental data that reflect project scoping decisions (Step 2). A project-specific QAPP may provide many instructions and project requirements pertaining to (but not limited to) the contaminants of potential concern, field sampling locations, field sampling procedures, sample preservation techniques and analytical data validation criteria. The QAPP may also contain specific information regarding the analyses to be performed by a laboratory that includes (but is not limited to) target analyte and/or parameter lists, the analytical and field Quality Control (QC) criteria, along with the hardcopy and electronic reporting requirements. All portions of the QAPP relevant to sample analysis and reporting should be communicated to the laboratory before samples are sent to the laboratory.

FIGURE 1: TYPICAL SUPERFUND DATA GENERATION AND REVIEW PROCESS



Generally, samples are then taken in the field based on the QAPP procedures and requirements (Step 3). There are typically two data flows created as a result of the field sampling activities:

(1) Samples and chain-of-custody information can be sent to a laboratory or laboratories; and,

(2) Field data, including sample location information, can be sent to the Project Manager and support staff.

The laboratory (or laboratories) then analyzes the samples and reports the results according to the protocols specified in the contract, or analytical method(s) (Step 4). The laboratory analytical data package (with any qualifiers noted by the laboratory itself) may then be sent to an external party (data validator) who can verify and validate the analytical data (or some percentage of it) using guidelines and/or requirements identified in specific national or regional analytical data validation guidance, analytical method(s) or contract (Step 5). The validated laboratory analytical data (now with external party validation qualifiers) then may be merged with information from the field logs (including sample location data). Generally, the overall data quality and usability is then assessed based on the review of field procedures and conditions during sampling (Step 6). Finally, any ultimate decisions made that rely on this data may be documented and stored in a data repository along with supporting field and laboratory data (Step 7). When stored appropriately, these data may be re-used or considered in future site decisions.

This guidance addresses only those verification and validation procedures that are provided by external parties for laboratory analytical data (see shaded area in Figure 1). In context of the UFP-QAPP Manual (IDQTF 2005), these procedures would be part of the UFP-QAPP Table 8 data review steps I (completeness) and IIa (check compliance with method, procedure, and contract requirements) for analysis.

4.0 Recommended Approach to Laboratory Analytical Data Verification and Validation

When called for by the QAPP, a laboratory analytical data package (electronic and/or hardcopy) developed for the Superfund program may be verified and validated by an external party (Figure 1, Step 5) as follows:

(1) First, the analytical data package should be checked or verified for *completeness* to ensure that all data requested is actually present in the data deliverables. The reporting requirements for the laboratory analytical data package should be specified by the project, contract or method. This is a critical step as analytical data validation (given in the following sub sections) may not be possible if any part of the requested laboratory data deliverable is not present.

(2) This completeness check should be followed by a *compliance* check to compare the documented sample receipt conditions and analytical QC results in the analytical data package to

ED_002345B_00034353-00012

the acceptance criteria, requirements or guidelines present in national or regional data validation documents, analytical method(s) or contract. The analytical QC results generally consist of two parts: (1) sample-related QC; and, (2) instrument-related QC.

(3) The completeness and compliance checks may be followed by *recalculation* checks. The laboratory reported values (e.g., sample results, instrument calibration results) can be checked by recalculating them using the data from instrument outputs reported by the laboratory to ensure that the laboratory used proper procedures to determine the final reported values. This may be done on a fraction or percentage of results or on all results reported by the laboratory. When a fraction or percentage of results is checked, additional results should be recalculated and checked if any issues are noted with the first recalculated values.

(4) Finally, the *actual instrument outputs* may be checked to ensure that the laboratory-reported analytes have been correctly identified and quantitated. This also generally may be done on a fraction or percentage of instrument outputs. Additional outputs should be checked if any issues are noted with the first fraction or percentage reviewed.

External party reviewers should document their findings by adding appropriate validation qualifiers (as necessary) to the sample results in the laboratory data packages based on the compliance, recalculations, and instrument output checks.

The process by which analytical data verification and validation should be carried out depends on the type of the laboratory analytical data package received by the external party. The laboratory analytical data package may be a hard copy data package, an electronic data deliverable, or both. The external party may verify and validate the analytical data using wholly manual, wholly electronic or a combination of manual and electronic processes, depending on the type of laboratory analytical data package and review instructions received. The nature and extent of verification and validation of analytical data needed to meet project goals should be specifically identified in the QAPP. The verified and validated analytical data may be sent either as a hard copy or an electronic file to the data recipient or EPA customer.

5.0 Describing the Stages and Processes Used to Verify and Validate Laboratory Analytical Data

For the purposes of this guidance, the following terminology is recommended for use by external parties to describe the stages (extent) and processes used to validate laboratory analytical data packages.

5.1 Analytical Data Verification and Validation Stages

(1) A verification and validation based only on completeness and compliance of sample receipt condition checks should be called a Stage 1 Validation.

(2) A verification and validation based on completeness and compliance checks of sample receipt conditions and ONLY sample-related QC results should be called a Stage 2A Validation.

ED_002345B_00034353-00013

(3) A verification and validation based on completeness and compliance checks of sample receipt conditions and BOTH sample-related and instrument-related QC results should be called a Stage 2B Validation.

(4) A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, AND recalculation checks should be called a Stage 3 Validation.

(5) A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, AND the review of actual instrument outputs should be called a Stage 4 Validation.

The recommended minimum baseline checks conducted for each stage of analytical data verification and validation are described in more detail in Appendix A.

Note: Using higher stages of analytical verification and validation does not typically result in higher data quality. However, the quality of the analytical data becomes more transparent as more stages of verification and validation are conducted. As a result, the usability of the analytical data for its intended use becomes more apparent.

5.2 Analytical Data Verification and Validation Processes

(1) A verification and validation based only on an electronic deliverable received from the laboratory and conducted using automated electronic data review tools should be described as Electronic Validation.

(2) A verification and validation based on an electronic deliverable as well as a hardcopy data package received from the laboratory using electronic data review tools and manual procedures should be described as Electronic and Manual Validation.

(3) A verification and validation based on only a hardcopy data package received from the laboratory using manual procedures should be described as Manual Validation.

Recommended summary terminology and labels that communicate both the stages of analytical data verification and validation and the processes used for conducting the verification and validation are given in Appendix B.

6.0 Communicating the Stages and Processes Used for Laboratory Analytical Data Verification and Validation

External parties should use one of the labels given in Appendix B to describe the stages and processes used to verify and validate particular laboratory analytical data packages (e.g., Sample Delivery Group). The verified and validated laboratory analytical data packages (with the appropriate labels) can then be sent to the data recipient for further data quality assessment (Figure 1, Step 6).

ED_002345B_00034353-00014

Suggested narratives for external parties to summarize the stage and manner of verification and validation for each verified and validated laboratory analytical data package are provided in Appendix C. It is recommended that this type of language be included in the accompanying transmittal memorandum in a section entitled "Analytical Data Package Validation Stage Summary."

If the external party delivers verified and validated laboratory analytical data in an electronic data deliverable (e.g., spreadsheet, a text file, an eXtensible Markup Language [XML] file, etc.), it is recommended that one of the labels given in Appendix B be appended to each analyte or parameter (by analytical method) in a column or data element named "Analytical Data Package Validation Stage." In this case the label appended to each analyte or parameter should correspond to the stage and process used by the external party to verify and validate the overall laboratory analytical data package (hardcopy and electronic). An example of an electronic spreadsheet format linking analyte-specific information with a recommended label is given in Appendix D.

Note: It is recommended that the external party label laboratory analytical data (within a validated laboratory analytical data package) that have not been verified and validated by them as "Not Validated".

7.0 Conclusion

The recommended stages used for verifying and validating laboratory analytical data addressed in this guidance constitute an important part of the overall laboratory analytical data quality assessment and usability assessment processes. Use of these labels by external parties should provide a consistent mechanism to communicate the nature and extent of their analytical data verification and validation procedures to their clients. The labels also should assist data recipients and future data users in merging verified and validated laboratory analytical data sets from different sources.

Note: In order to assess the *usability* of the laboratory analytical data, additional information should be considered including (but not limited to) the procedures used to collect the samples, field parameters, field location measurements, comparison of results with QAPP measurement performance criteria, and overall project quality objectives.

8.0 References

CIO 2105.0 (May 2000). *Policy and Program Requirements for the Mandatory Agency-wide Quality System*, U.S. Environmental Protection Agency, Washington, DC, http://www.epa.gov/quality/qa_docs.html

IDQTF. 2005. (March 2005), *Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs*, Intergovernmental Data Quality Task Force, EPA-505-B-04-900A. March. http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf

ED_002345B_00034353-00015

APPENDIX A

Laboratory Analytical Data Verification and Validation Stages and Checks: Description, Order, and Labeling of Validated Laboratory Analytical Data Packages

1.0 Recommended Minimum Baseline Checks Used in the Stages of Laboratory Analytical Data Verification and Validation.

A recommended sequence of laboratory analytical data verification (completeness) and validation (compliance, recalculations, and instrument output evaluations) checks is summarized below (including the minimum baseline checks for each validation stage). Specific checks will depend on the analytical method being verified and validated and the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract. Each higher stage of verification and validation should include all the relevant checks defined in the next lower stage or stages. For example, there are 23 checks listed as part of a Stage 2B verification and validation (these include check numbers 1-9 noted in Stage 1, numbers 10-16 noted in Stage 2A, and numbers 17-23 noted in Stage 2B). The recommended checks discussed below are not a complete list of all the checks that can be done for a particular laboratory analytical data package as these will vary depending on the method(s) used to generate the data. However, the list of checks given in each stage covers a majority of checks conducted during the verification and validation of Superfund laboratory analytical data and represents a recommended baseline level of checks that can be performed during the verification and validation process (as appropriate for the method).

1.1 Recommended Stage 1 Verification and Validation Checks

Stage 1 validation of the laboratory analytical data package consists of verification and validation checks for the compliance of sample receipt conditions, sample characteristics (e.g., percent moisture), and analytical results (with associated information). It is recommended that the following minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 1 validation label:

(1) Documentation identifies the laboratory receiving and conducting analyses, and includes documentation for all samples submitted by the project or requester for analyses.

(2) Requested analytical methods were performed and the analysis dates are present.

(3) Requested target analyte results are reported along with the original laboratory data qualifiers and data qualifier definitions for each reported result (and the uncertainty of each result and clear indication of the type of uncertainty reported if required, e.g., for radiochemical analyses).

(4) Requested target analyte result units are reported (along with their associated uncertainty units if required, e.g., for radiochemical analyses).

ED_002345B_00034353-00016

(5) Requested reporting limits for all samples are present and results at and below the requested (required) reporting limits are clearly identified (including sample detection limits if required).

(6) Sampling dates (including times if needed), date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory (including preservation, pH and temperature) are documented.

(7) For radiochemical analyses, the sample-specific critical values (sometimes called "critical level," "decision level" or "detection threshold") and sample specific minimum detectable value, activity or concentration for all samples are reported and results at and below the requested (required) critical values are clearly identified.

(8) For radiochemical analyses, the chemical yield (if applicable to the method) and reference date and time (especially for short lived isotopes) is reported for all samples (as appropriate).

(9) Sample results are evaluated by comparing sample conditions upon receipt at the laboratory (e.g., preservation checks) and sample characteristics (e.g., percent moisture) to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

1.2 Recommended Stage 2A Verification and Validation Checks

Stage 2A validation builds on the validation conducted in Stage 1. Stage 2A validation of the laboratory analytical data package consists of the Stage 1 validation plus the verification and validation checks for the compliance of sample-related QC. It is recommended that the following additional minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 2A Validation label:

(10) Requested methods (handling, preparation, cleanup, and analytical) are performed.

(11) Method dates (including dates, times and duration of analysis for radiation counting measurements and other methods, if needed) for handling (e.g., Toxicity Characteristic Leaching Procedure), preparation, cleanup and analysis are present, as appropriate.

(12) Sample-related QC data and QC acceptance criteria (e.g., method blanks, surrogate recoveries, deuterated monitoring compounds (DMC) recoveries, laboratory control sample (LCS) recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries, serial dilutions, post digestion spikes, standard reference materials) are provided and linked to the reported field samples (including the field quality control samples such as trip and equipment blanks).

(13) Requested spike analytes or compounds (e.g., surrogate, DMCs, LCS spikes, post digestion spikes) have been added, as appropriate.

ED_002345B_00034353-00017

(14) Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated.

(15) Frequency of QC samples is checked for appropriateness (e.g., one LCS per twenty samples in a preparation batch).

(16) Sample results are evaluated by comparing holding times and sample-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

1.3 Recommended Stage 2B Verification and Validation Checks

Stage 2B validation builds on the validation conducted in Stage 2A. Stage 2B validation of the laboratory analytical data package consists of the Stage 2A validation plus the verification and validation checks for the compliance of instrument-related QC. It is recommended that the following additional minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 2B Validation label:

(17) Initial calibration data (e.g., initial calibration standards, initial calibration verification [ICV] standards, initial calibration blanks [ICBs]) are provided for all requested analytes and linked to field samples reported. For each initial calibration, the calibration type used is present along with the initial calibration equation used including any weighting factor(s) applied and the associated correlation coefficients, as appropriate. Recalculations of the standard concentrations using the initial calibration curve are present, along with their associated percent recoveries, as appropriate (e.g., if required by the project, method, or contract). For the ICV standard, the associated percent recovery (or percent difference, as appropriate) is present.

(18) Appropriate number and concentration of initial calibration standards are present.

(19) Continuing calibration data (e.g., continuing calibration verification [CCV] standards and continuing calibration blanks [CCBs]) are provided for all requested analytes and linked to field samples reported, as appropriate. For the CCV standard(s), the associated percent recoveries (or percent differences, as appropriate) are present.

(20) Reported samples are bracketed by CCV standards and CCBs standards as appropriate.

(21) Method specific instrument performance checks are present as appropriate (e.g., tunes for mass spectrometry methods, DDT/Endrin breakdown checks for pesticides and aroclors, instrument blanks and interference checks for ICP methods).

(22) Frequency of instrument QC samples is checked for appropriateness (e.g., gas chromatography-mass spectroscopy [GC-MS] tunes have been run every 12 hours).

(23) Sample results are evaluated by comparing instrument-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

ED_002345B_00034353-00018

1.4 Recommended Stage 3 Verification and Validation Checks

Stage 3 validation builds on the validation conducted in Stage 2B. Stage 3 validation of the laboratory analytical data package consists of the Stage 2B validation plus the recalculation of instrument and sample results from the laboratory instrument responses, and comparison of recalculated results to laboratory reported results. It is recommended that the following additional minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 3 Validation label:

(24) Instrument response data (e.g., GC peak areas, ICP corrected intensities) are reported for requested analytes, surrogates, internal standards, and DMCs for all requested field samples, matrix spikes, matrix spike duplicates, LCS, and method blanks as well as calibration data and instrument QC checks (e.g., tunes, DDT/Endrin breakdowns, interelement correction factors, and Florisil cartridge checks).

(25) Reported target analyte instrument responses are associated with appropriate internal standard analyte(s) for each (or selected) analyte(s) (for methods using internal standard for calibration).

(26) Fit and appropriateness of the initial calibration curve used or required (e.g., mean calibration factor, regression analysis [linear or non-linear, with or without weighting factors, with or without forcing]) is checked with recalculation of the initial calibration curve for each (or selected) analyte(s) from the instrument response.

(27) Comparison of instrument response to the minimum response requirements for each (or selected) analyte(s).

(28) Recalculation of each (or selected) opening and closing CCV (and CCB) response from the peak data reported for each (or selected) analyte(s) from the instrument response, as appropriate.

(29) Compliance check of recalculated opening and/or closing CCV (and CCB) response to recalculated initial calibration response for each (or selected) analyte(s).

(30) Recalculation of percent ratios for each (or selected) tune from the instrument response, as appropriate.

(31) Compliance check of recalculated percent ratio for each (or selected) tune from the instrument response.

(32) Recalculation of each (or selected) instrument performance check (e.g., DDT/Endrin breakdown for pesticide analysis, instrument blanks, interference checks) from the instrument response.

ED_002345B_00034353-00019

(33) Recalculation and compliance check of retention time windows (for chromatographic methods) for each (or selected) analyte(s) from the laboratory reported retention times.

(34) Recalculation of reported results for each reported (or selected) target analyte(s) from the instrument response.

(35) Recalculation of each (or selected) reported spike recovery (surrogate recoveries, DMC recoveries, LCS recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries, serial dilutions, post digestion spikes, standard reference materials etc.) from the instrument response.

(36) Each (or selected) sample result(s) and spike recovery(ies) are evaluated by comparing the recalculated numbers to the laboratory reported numbers according to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

Note: Selection of analytes, spikes, and performance evaluation checks for the Stage 3 validation checks for a laboratory analytical data package being verified and validated generally will depend on many factors including (but not limited to) the type of verification and validation being performed (manual or electronic), requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract, the number of laboratories reporting the data, the number and type of analytical methods reported, the number of analytes reported in each method, and the number of detected analytes.

1.5 Recommended Stage 4 Verification and Validation Checks

Stage 4 validation builds on the validation conducted in Stage 3. Stage 4 validation of the laboratory analytical data package consists of the Stage 3 validation plus the evaluation of instrument outputs. It is recommended that the following additional minimum baseline checks (as relevant) be performed on the laboratory analytical data package received for a Stage 4 Validation label:

(37) All required instrument outputs (e.g., chromatograms, mass spectra, atomic emission spectra, instrument background corrections, and interference corrections) for evaluating sample and instrument performance are present.

(38) Sample results are evaluated by checking each (or selected) instrument output (e.g., chromatograms, mass spectra, atomic emission spectra data, instrument background corrections, interference corrections) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences).

(39) Each (or selected) instrument's output(s) is evaluated for confirmation of non-detected or tentatively identified analytes.

ED_002345B_00034353-00020

Note: Selection of instrument outputs for the Stage 4 validation checks for a laboratory analytical data package being verified and validated generally will depend on many factors including, but not limited to, the type of verification and validation being performed (electronic or manual), requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract, the number of laboratories reporting the data, the number and type of analytical methods reported, the number of analytes reported in each method, and the number of detected analytes.

2.0 Order of Stages and Checks

It is recommended that all relevant Stage 1 checks be performed first, followed by all relevant checks in the remaining validation stages in order of increment as required by the project. The checks within a Stage (used for verification and validation) need not be done in any particular order as this will vary depending on the importance of the check, type of laboratory analytical data package received (hardcopy, electronic or both), the type of laboratory analytical data reported (methods used), and the documents and tools used to verify and validate the laboratory analytical data.

3.0 Labeling of Verified and Validated Laboratory Analytical Data Packages

Generally, all relevant checks within each stage should be done and validation qualifiers added (as appropriate) to the sample results in the laboratory analytical data package (e.g., Sample Delivery Group) in order to label the laboratory analytical data package (or the reported analyte or parameter) as being verified and validated to that stage.

Note: When automated data review tools are used to review analytical data sent by laboratories in electronic data deliverables, the tools should (a) conduct all relevant checks within each stage for each reported analyte or parameter in the electronic data deliverable; (b) automatically label each analyte or parameter with the appropriate label from Appendix B, and (c) provide this label for each analyte or parameter in the tool's electronic outputs (in a column or a data element named "Analytical Data Validation Stage").

ED_002345B_00034353-00021

APPENDIX B

Recommended Terminology and Labels for Communicating the Stages and Processes Used for Laboratory Analytical Data Verification and Validation

The following labels and/or codes are recommended for use by external parties when communicating to data recipients and data users the steps as well as the manner used for laboratory analytical data verification and validation.

Label	Corresponding Label Code
Stage_1_Validation_Electronic	S1VE
Stage_1_Validation_Manual	S1VM
Stage_1_Validation_Electronic_and_Manual	S1VEM
Stage_2A_Validation_Electronic	S2AVE
Stage_2A_Validation_Manual	S2AVM
Stage_2A_Validation_Electronic_and_Manual	S2AVEM
Stage_2B_Validation_Electronic	S2BVE
Stage_2B_Validation_Manual	S2BVM
Stage_2B_Validation_Electronic_and_ Manual	S2BVEM
Stage_3_Validation_Electronic	S3VE
Stage_3_Validation_Manual	S3VM
Stage_3_Validation_Electronic_and_Manual	S3VEM
Stage_4_Validation_Electronic	S4VE
Stage_4_Validation_Manual	S4VM
Stage_4_Validation_Electronic_and_ Manual	S4VEM
Not_Validated	NV

ED_002345B_00034353-00022

APPENDIX C

Suggested Narrative(s) for Laboratory Analytical Data Package Verification and Validation "Summary" Section(s)

Section Name: Analytical Data Package Validation Stage Summary

Example 1:

Sample Delivery Groups JFS123, JFS124, and JFS125 have the following label:
Stage_4_Validation_Manual (S4VM).

Example 2:

All sample results in the attached laboratory analytical data package Number 23 have the following label:
Stage_2B_Validation_Electronic_And_Manual (S2BVEM).

Example 3:

Sample Delivery Groups JFS126 and JFS127 have the following label:
Stage_4_Validation_Electronic_And_Manual (S4VEM).

Sample Delivery Group JFS128 has the following label:
Stage_2B_Validation_Manual (S2BVM).

Example 4:

All sample results (except those analyzed by Method 8260 Volatile Organic Analysis) in Sample Delivery Group JFS129 have the following label:

Stage_4_Validation_Manual (S4VM).

All sample results for Method 8260 in Sample Delivery Group JFS129 were not validated (per client request) and have the following label:

Not_Validated (NV).

APPENDIX D

Example Laboratory Analytical Results Electronic Spreadsheet With a Column for the Analytical Data Package Validation Stage

Lab Name	Client Sample ID	Matrix ID	Analyte Name	Client Method ID	Result	Result Units	Validation Qualifier	Analytical Data Package Validation Stage
ARM	A1231	Water	Benzene	CLPVOA	6.6	ug/L	J	S3VE
ARM	A1231	Water	Toluene	CLPVOA	<5.0	ug/L	UJ	S3VE
ARM	A1231	Water	Ethyl Benzene	CLPVOA	7.7	ug/L	J	S3VE
ARM	A1231	Water	m,p-Xylene	CLPVOA	<5.0	ug/L	UJ	S3VE
ARM	A1231	Water	Acetone	CLPVOA	<5.0	ug/L	U	S3VE
ARM	A1231	Water	Chloro methane	CLPVOA	<5.0	ug/L	U	S3VE
ARM	A1231	Water	Styrene	CLPVOA	<5.0	ug/L	U	S3VE
ARM	A1231	Water	Chloro ethane	CLPVOA	<5.0	ug/L	U	S3VE
ARM	A1231	Water	o-Xylene	CLPVOA	<5.0	ug/L	U	S3VE
ARM	A1231	Water	Vinyl Chloride	CLPVOA	<5.0	ug/L	U	S3VE
ARM	A1231	Water	Cyclo Hexane	CLPVOA	<5.0	ug/L	U	S3VE

ED_002345B_00034353-00024

APPENDIX E

GLOSSARY OF TERMS[1]

Aliquot — A measured portion of a sample taken for analysis.

Analyte — A property which is to be measured.

Analytical batch — A group of samples, including quality control samples, which are processed together using the same method, the same lots of reagents, and at the same time or in continuous, sequential time periods. Samples in each batch should be of similar composition and share common internal quality control standards.

Blank — A sample subjected to the usual analytical or measurement process to establish a zero baseline or background value; a sample that is intended to contain none of the analytes of interest. A blank is used to detect contamination during sample handling preparation and/or analysis.

Calibration — A set of operations that establish, under specific conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or value's represented by a material measure of a reference material, and the corresponding values realized by standards.

Completeness — A measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct, normal conditions.

Continuing calibration verification (CCV) — A check of the initial calibration that is performed during the course of an analytical shift at periodic intervals using a Calibration Check Standard. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. The purpose is to assess the continued capability of the measurement system to generate accurate and precise data over a period of time.

Data review — The process of examining and/or evaluating data to varying levels of detail and specificity by a variety of personnel who have different responsibilities within the data management process. It includes verification, validation, and usability assessment.

Data user — Technical and other personnel responsible for engineering, scientific, and legal evaluations that are the basis for site decisions. Data users are responsible for determining data needs required to satisfy project objectives from their perspective (remedy, risk, compliance, etc.).

---

[1] Where available, the definitions and terms were taken from the "Glossary of Quality Assurance and Related Terms" in the Intergovernmental Data Quality Task Force, (March 2005), Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP) Manual, EPA-505-B-04-900A, http://www.epa.gov/fedfac/documents/qualityassurance.htm

ED_002345B_00034353-00025

Electronic data evaluation — The use of computers and/or software to assess and process laboratory analytical data in meeting compliance with established quality control requirements.

Environmental data — Any parameters or pieces of information collected or produced from measurements, analyses, or models of environmental processes, conditions, and effects of pollutants on human health and the ecology, including results from laboratory analyses or from experimental systems representing such processes and conditions. It also includes information collected directly from measurements, produced from models, and compiled from other sources such as databases or the literature.

Equipment blank — A sample of water, or other appropriate media, free of measurable contaminants poured over or through decontaminated field sampling equipment that is considered ready to collect or process an additional sample. The purpose of this blank is to assess the adequacy of the decontamination process. Also called rinse blank or rinsate blank.

External party(ies) — Organizations (including Governmental entities, contractors, or vendors) that conduct analytical data review, verification, and validation activities and that are not part of the immediate laboratory that generates the analytical data.

Extensible Markup Language (XML) — A flexible way to create common information formats and share both the format and the data on the World Wide Web, intranets, and elsewhere. XML can be used by any individual or group of individuals or companies that wants to share information in a consistent way.

Holding time — The period of time a sample may be stored prior to its required preparation and/or analysis.

Instrument quality control (QC) sample — A sample of known composition analyzed concurrently with environmental samples to verify the performance of one or more components of the analytical measurement process. Those components can include retention time, resolution, recovery, degradation, etc.

Internal standard — A standard added to a test portion of a sample in a known amount and carried through the entire determination procedure as a reference for calibrating and controlling the precision and bias of the applied analytical method.

Laboratory(ies) — A place equipped for experimental study in science or for testing and analysis. Laboratories can be found in government or industry facilities, schools and universities, or in mobile or semi-moveable structures (e.g., specialized vehicles, trailers, temporary office structures, ships, and aircraft).

Laboratory control sample (LCS) — A sample of known composition prepared using contaminant-free water or in inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples.

ED_002345B_00034353-00026

Laboratory duplicates/replicates — Two or more representative aliquots taken from one sample by the laboratory and analyzed in the same laboratory. Laboratory duplicate/replicate samples are quality control samples that are used to assess intralaboratory preparatory and analytical precision as well as sample homogeneity.

Manual data evaluation — The examination of laboratory analytical data by individuals or groups of people using standard operating procedures and guidance in meeting compliance with established quality control requirements.

Matrix — The material of which the sample is composed, such as water, soil/sediment, or other environmental medium.

Matrix spike — A sample prepared by adding a known concentration of a target analyte to an aliquot of a specific homogenized environmental sample for which an independent estimate of the target analyte concentration is available. The matrix spike is accompanied by an independent analysis of the unspiked aliquot of the environmental sample. Spiked samples are used to determine the effect of the matrix on a method's recovery efficiency.

Matrix spike duplicate — A homogeneous sample used to determine the precision of the intralaboratory analytical process for specific analytes in a sample matrix. The duplicate sample is prepared simultaneously as a split with the matrix spike sample, and each is spiked with identical, known concentrations of targeted analyte(s).

Method — A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

Method blank — A sample of a matrix similar to the batch of associated samples (when available) in which no target analytes or interferences are present at concentrations that impact the analytical results. It is processed and analyzed simultaneously with samples of similar matrix and under the same conditions as the samples.

Quality — The totality of features and characteristics of a product or service that bears on its ability to meet the stated or implied needs and expectations of the user.

Quality assurance — An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality assurance project plan (QAPP) — A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that should be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

ED_002345B_00034353-00027

Quality control (QC) — A term that may be applied in several different contexts: (1) The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; (2) operational techniques and activities that are used to fulfill requirements for quality; also, (3) the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality control (QC) sample — One of any number of samples, such as a Proficiency Test (PT) sample, intended to demonstrate that a measurement system or activity is in control.

Quality system — A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities.

Raw data — The documentation generated during sampling and analysis. This documentation includes, but is not limited to, hard copies of electronic data, magnetic tapes, untabulated sample results, QC sample results, printouts of chromatograms, instrument outputs, and handwritten notes.

Reagent blank — An aliquot of water or solvent free of measurable contaminants analyzed with the analytical batch and containing all the reagents in the same volume as used in the processing of the samples. The method blank goes through preparatory steps; the reagent blank does not.

Spike — A substance that is added to an environmental sample to increase the concentration of target analytes by known amounts. A spike is used to assess measurement accuracy (spike recovery). Spike duplicates are used to assess measurement precision.

Standard operating procedures (SOPs) — A document that details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps written for and followed by its intended user. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.

Surrogate spike or analyte — A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes (e.g., Deuterated Monitoring Compounds).

Trip blank — A clean sample of water, or other appropriate media, free of measurable contaminants that is taken to the sampling site and transported to the laboratory for analysis without having been exposed to sampling procedures. Trip blanks are analyzed to assess whether contamination was introduced during sample shipment (typically analyzed for volatile organic compounds only).

ED_002345B_00034353-00028

Usability assessment — Evaluation of data based upon the results of data validation and verification for the decisions being made. In the usability step, reviewers assess whether the process execution and resulting data meet quality objectives based on criteria established in the QAPP.

Validation — Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. For the purpose of this guidance, *data validation* consists of an analyte and sample specific process for evaluating compliance of the laboratory data received with methods, procedures or contract requirements.

Verification — Confirmation by examination and provision of objective evidence that the specified requirements (analytical) have been met. This is to be a completeness check. For the purpose of this guidance, *data verification* consists of a completeness check to confirm that all data requested from the laboratory have been received and comply with specified requirements.

ED_002345B_00034353-00029