UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX
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LABORATORY DOCUMENTATION REQUIREMENTS

FOR DATA VALIDATION

Document Control Number 9QA-07-90

January, 1990

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INTRODUCTION

In all hazardous site investigations, it is essential to know the quality of the data used for decision-making purposes. The process of generating data of known quality begins in the planning stages when data quality objectives (DQOs) are established, continues during sample collection activities and laboratory analysis, and is completed by validating the analytical data. This document was created to identify the specific laboratory documentation requirements necessary for data validation.

Validation of data requires that appropriate QA/QC and documentation steps be performed in both the lab and the field. Professionals trained in data validation procedures review this information, "flag" data with qualifiers when QA/QC criteria are not met, and prepare the data validation report.

The "P.K. Memo" and ICF/ESAT documents, which have previously addressed non-CLP documentation requirements, have been incorporated into this document. The general requirements are discussed here, but for ease of use it has been formatted into two (2) sections, pertaining to the organic and inorganic analyses. In addition to the documentation requirements, a new and separate section for non-CLP QA/QC requirements was created.

The documentation provided by the laboratory in conjunction with the sample results, allows for the evaluation of the following indicators of data quality:

- Integrity and stability of the samples
- Instrument performance during sample analysis
- Possibility of sample contamination
- Identification and quantitation of analytes
- Precision
- Accuracy of the analytical results

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I. ORGANIC ANALYSES

I.A. Documentation

The data package submitted for EPA data validation will consist of five (5) sections:

- Case narrative
- Chain-of-Custody documentation
- Summary of results for environmental samples (including quantitation limits)
- Summary of QA/QC results
- Raw data

I.B. Case Narrative

The case narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or his/her designee. The Case Narrative will consist of the following information:

- Client's sample identification and the corresponding laboratory identification
- Parameters analyzed for each sample and the methodology used: when applicable, cite EPA method numbers
- Whether the holding times were met or exceeded
- Detailed description of all problems encountered
- Discussion of possible reasons for any QA/QC criteria outside acceptance limits
- Observations regarding any occurrences which may affect sample integrity or data quality

I.C. Chain-of-Custody Documentation

Legible copies of Chain-of-Custody forms for each sample shall be submitted in the data package. The date of receipt and the observed sample condition at the time of receipt must be described on the Chain-of-Custody form. Any internal laboratory tracking document should be included.

I.D. Summary of Environmental Results

The following information is to be included in the summary of results for each environmental sample. The summary should follow the CLP format if possible, but other formats are acceptable provided that all necessary information is included.

- Client's sample identification and the corresponding laboratory identification
- Sample matrix
- Date of sample extraction, as applicable
- Date and time of analysis
- Identification of the instrument used for analysis
- GC column and detector specifications
- Weight or volume of sample used for analysis/extraction
- Dilution or concentration factor for the samples
- Percentage of moisture in the soil samples
- Method detection limits (MDL) or sample quantitation limits
- Definitions for any data qualifiers used
- Analytical results

I.E. Summary of QA/QC Results

The following QA/QC results will be presented in a summary. These summaries should follow the CLP format, if possible. Other formats may be acceptable provided that all necessary information is included and the summary is easy to follow. These summaries will require to have all the information stated in Section I.D.

I.E.1. Instrument Calibration (for each instrument used)

Initial Calibration

Report the concentrations of the initial calibration standards and the date and time of analysis. List the response factor (RF), percent relative standard deviation (%RSD), and retention time (for GC analyses) for each analyte.

Daily Calibration and Mid-level Standard

Report the concentration of the calibration standard used for the daily calibration and for the mid-level standard, and the date and time of analysis. List the response factor (RF), percent difference (%D), and retention time (for GC analyses) for each analyte.

I.E.2. Method Blank Analysis

List the environmental samples and QC analyses associated with each method blank. Report the concentrations of any analytes found in the method blanks.

I.E.3. Surrogate Standard Recovery

Report the name and concentration of each surrogate compound added. List the percent recoveries of all surrogates in the samples, method blanks, matrix spike/matrix spike duplicates and other QC analyses.

I.E.4. Precision and Accuracy

- Matrix spike/matrix spike duplicate (MS/MSD) analysis

Report the name and concentration of each spiking compound. Samples are to be spiked with all specified compounds of interest. List the sample results, spiked sample results, percent recovery and the relative percent difference (RPD)

- Laboratory duplicate analysis, as applicable.

Report the relative percent difference (RPD) between duplicate analyses.

- Laboratory QC check sample analysis

Report the percent recovery for each analyte in the laboratory QC check sample. List the acceptable control limits.

I.E.5. Other QC Criteria

- Retention time windows determination (GC)

Report the retention time window for each analyte, for both primary and confirmation analyses.

Retention time windows are established by performing 3 analyses of standards for all analytes being measured throughout the course of a 72-hour period. The retention time window is defined as plus or minus 3 times the standard deviation of the absolute retention time. Retention time windows are to be updated daily.

- Compound identification (GC)

Report the retention times and the concentrations of each analyte detected in the samples for both primary and confirmation analyses.

- Method detection limits (MDL) determination

List the method detection limits.

Method detection limits are determined by performing at least 7 analyses of standards for all analytes measured at 2-5 times the required detection limit concentrations. The method detection limits are calculated as 3 times the standard deviation of the measured values. Refer to 40 CFR Part 136 Appendix B.

I.F. Raw Data

I.F.1. GC Analyses

This section shall include legible copies of the raw data for the following:

- Environmental samples (arranged in increasing client's sample number order).

The raw data for both the primary and confirmation analyses are to be included.

- Instrument calibrations

- QC analyses

- Sample extraction and clean-up logs
- Instrument analysis logs for each instrument used
- GC/MS confirmation, as applicable

The raw data for each analysis shall include the following:

- Chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names)
- Area print-outs or quantitation reports

I.F.2. GC/MS Analyses

This section shall include legible copies of the raw data for the following:

- Environmental samples (arranged in increasing client's sample number order)
- Mass and spectrometer tuning and mass calibration (BFB: DFTPP)
- Initial and continuing instrument calibrations
- QC analyses
- Sample extraction and clean-up logs
- Instrument analysis logs for each instrument used

The raw data for each analysis shall include the following:

- Chromatograms (label all analyte peaks, internal standards and surrogate standards with chemical names)
- Enhanced spectra of target analytes and tentatively identified compounds (TICs), with the associated best-match spectra
- Quantitation reports

Legible copies of the raw data shall be organized systematically, and each page shall be numbered. The raw data for compound identification and quantitation must be sufficient to verify each result presented in Sections I.D. and I.E.

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I.G. SUMMARY OF DOCUMENTATION REQUIREMENTS

Organic Data

Section I. Case Narrative

Section II. Chain-of-Custody Documentation

1. Chain-of-Custody forms
2. Internal tracking documents, as applicable

Section III. Summary of Results - Forms for the following:

1. Environmental samples, with quantitation limits (include dilutions and re-analyses)

Section IV. QA/QC Results Summaries

1. Initial calibration
2. Continuing calibration
3. Method blanks
4. Surrogate recoveries
5. Matrix spike (MS)
6. Laboratory duplicate or matrix spike duplicate (MSD)
7. Laboratory QC check sample, if applicable
8. Retention time windows
9. Method detection limits (MDL)

Section V. Raw Data - chromatograms and area/quantitation reports

1. Environmental samples (include dilutions and re-analyses)
2. Instrument tuning, for mass spectrometry (GC/MS) analyses
3. Initial calibration
4. Continuing calibration
5. Method blanks
6. Surrogate recoveries
7. Matrix spike (MS)
8. Laboratory duplicate or matrix spike duplicate (MSD)
9. Laboratory QC check sample, as applicable
10. Retention time windows
11. Percent moisture for soil samples
12. Sample extraction and clean-up logs
13. Instrument analysis log for each instrument used

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II. INORGANIC ANALYSES

II.A. Documentation

The data package submitted for EPA data validation will consist of five (5) sections:

- Case narrative
- Chain-of-Custody documentation
- Summary of results for environmental samples (including quantitation limits)
- Summary of QA/QC results
- Raw data

II.B. Case Narrative

The case narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or his/her designee. The Case Narrative will consist of the following information:

- Client's sample identification and the corresponding laboratory identification
- Parameters analyzed for each sample and the methodology used: when applicable, cite EPA method numbers
- Whether the holding times were met or exceeded
- Detailed description of all problems encountered
- Discussion of possible reasons for any QA/QC criteria outside acceptance limits
- Observations regarding any occurrences which may affect sample integrity or data quality

II.C. Chain-of-Custody Documentation

Legible copies of Chain-of-Custody forms for each sample shall be submitted in the data package. The date of receipt and the observed sample condition at the time of receipt must be described on the Chain-of-Custody form.

II.D. Summary of Environmental Results

The following information is to be included in the summary of results for each environmental sample. The summary should follow the CLP format if possible, but other formats are acceptable provided that all necessary information is included.

- Client's sample identification and the corresponding laboratory identification
- Sample matrix
- Date of sample digestion, as applicable
- Date and time of analysis
- Identification of the instrument used for analysis
- Instrument specifications
- Weight or volume of sample used for analysis/digestion
- Dilution or concentration factor for the samples
- Percentage of moisture in the soil samples
- Instrument detection limits (IDL) or method detection limits (MDL)
- Definitions for any data qualifiers used
- Analytical results

II.E. Summary of QA/QC Results

The following QA/QC results will be presented in a summary. These summaries should follow the CLP format, if possible. Other formats are acceptable provided that all necessary information is included and the summary is easy to follow. These summaries will require to have all information stated in Section II.D.

II.E.1. Instrument Calibration

The order of reporting of calibrations for each analyte must follow the temporal order in which the standards were analyzed.

Initial Calibration Verification

Report the source for the calibrations standards. Report the concentration for the true value, the concentration found, and the percent recovery for each element analyzed. Record the date and time of analysis.

Continuing Calibration Verification

Report the source for the calibrations standards. Report the concentration for the true value, the concentration found, and the percent recovery for each element analyzed. Record the date and time analysis.

Report results for (low-level) standards used to verify instrument sensitivity (that the reported detection limits can be achieved) in the manner described for continuing calibration verification.

II.E.2. Method Blank Analysis

Report analyte concentrations found in the initial calibration blank (ICB), the continuing calibration blank (CCB), and in the preparation blank. Record the date and time of analysis.

The order of reporting ICB and CCB for each analyte must follow the temporal order in which the blanks were analyzed.

II.E.3. ICP Interference Check Sample

Identify the source for the interference check sample. Report the true value, the initial and final results and the calculated percent recovery.

II.E.4. Precision and Accuracy

- Matrix spike (MS) analysis

Report the concentration of the spiked sample result, the sample result and the spiking solution added for each element in the predigestion spike. Calculate and report the percent recovery and list the control limits.

- Post Digest Spike

In addition to matrix spikes, post-digest spikes are analyzed during furnace analysis. Report the concentration of the spiked sample result, the sample result, and the spiking

solution added for each element. Calculate and report the percent recovery and list the control limits.

- Laboratory Duplicate Analysis

Report the original concentration, duplicate concentration and relative percent difference (RPD). List the control limits.

- Laboratory Control Sample

Identify the source for the laboratory control sample. Report the concentration of the spiked sample result, the sample results and the spiking solution added for each element analyzed. Calculate and report the percent recovery and list the control limits.

The laboratory control check sample is prepared in the same way as the analytical samples.

II.E.5. Other QC Criteria

- Method of Standard Additions (MSA)

This summary must be included when MSA analyses are required. Report the absorbance values with corresponding concentration values. Report the final analyte concentration and list the correlation coefficient.

- ICP serial dilution

Report the initial and serial dilution results and the percent difference.

- ICP Linear Ranges

For each instrument and wavelength used, report the date on which the linear ranges were established, the integration time, and the upper limit concentration.

- ICP Interelement Correction Factors

For each instrument and wavelength used, report the date on which the correction factors were determined. List the interelement correction factors for Al, Ca, Fe, Mg and any other element and the analytes to which they are applied.

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- Instrument detection limits (IDL) determination

List the instrument detection limits.

Instrument detection limits are determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution at a concentration 3-5 times the required detection limit concentrations, with 7 consecutive measurements per day. Refer to the 40 CFR Part 136 Appendix B.

II.F. Raw data

This section shall include legible copies of the raw data for the following:

- Environmental samples (arranged in increasing client's sample number order)
- Instrument calibrations
- QC analyses
- Sample preparation and digestion logs
- Instrument analysis logs for each instrument used
- Percent moisture in the soil samples

The raw data for each analysis shall include the following:

- Measurement print-outs and quantitation reports for each instrument used
- Absorbance, titrimetric, or other measurements for wet chemical analysis

Legible copies of the raw data shall be organized systematically, and each page shall be numbered. The raw data for compound identification and quantitation must be sufficient to verify each result presented in Sections II.D. and II.E.

II.G. SUMMARY OF DOCUMENTATION REQUIREMENTS

Inorganic Data

Section I. Case Narrative

Section II. Chain-of-Custody Documentation

1. Chain-of-Custody forms
2. Internal tracking documents, as applicalbe

Section III. Summary of Results - Forms for the following:

1. Environmental samples, with quantitation limits (include dilutions and re-analyses)

Section IV. QA/QC Result Summaries

1. Initial and continuing calibrations
2. Method blanks, continuing calibration blanks, and prep blanks
3. ICP interference check sample
4. Matrix spike
5. Laboratory duplicate
6. Laboratory control sample
7. Method of standard additions
8. ICP serial dilution
9. Instrument detection limits
10. ICP linear range

Section V. Raw Data - sequential measurement readout records for ICP, graphite furnace AA, flame AA, cold vapor mercury, cyanide, and/or other inorganic analyses.

1. Environmental samples (including dilutions and reanalyses)
2. Initial and continuing calibrations
3. Continuing calibration and Preparation blanks
4. Matrix spikes
5. Post digest spikes
6. Method of standard additions, when applicable
7. Laboratory duplicate or matrix spike duplicates
8. ICP Serial Dilution
9. Laboratory control samples, when applicable
10. Percent moisture for soil samples
11. Sample digestion and/or sample preparation logs
12. Instrument analysis log, for each instrument used
13. Instrument tuning for ICP-MS, when applicable

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III. QC REQUIREMENTS SUMMARY

III.A. GC/MS Organic Analyses

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. Instrument Tuning

   - At the beginning of each day that samples are analyzed

2. Initial Calibration

   - At the beginning of the QC program
   - Whenever percent difference (%D) of the response factors for specified compounds of interest or calibration check compounds (CCC; a minimum of 5 compounds total) between continuing calibration and initial calibration exceeds ±25%
   - Whenever the response factors for specified compounds of interest or system performance check compounds (SPCC; a minimum of 5 compounds total) are less than 0.300 (0.250 for bromoform) for volatiles or less than 0.050 for semi-volatiles analyses
   - After installation of a new column or after maintenance service/repair of the gas chromatography/mass spectrometry (GC/MS)

3. Continuing Calibration

   - Prior to the analysis of environmental samples, on each 12-hour shift that samples are analyzed

4. Method Blank

   - Volatiles: After each continuing calibration analysis and after the analyses of unusually concentrated samples, to demonstrate that the system is free of contamination.
   - Semi-volatiles: One for each extraction batch of 20 or fewer samples, for each sample matrix. Analyze method blanks on all instruments used for sample analysis.

- Method blanks should not contain any analytes of interest and are to be free of interfering peaks.

5. Calibration Range

   - For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. Surrogate Standard

   - Surrogate standards (3 for volatiles; 3 phenolic and 3 neutral compounds for semi-volatiles) are to be added to the calibration standards, method blanks, environmental samples and QC samples.

7. Internal Standard

   - Internal standards (3 for volatiles and 6 for semi-volatiles) are to be added to the calibration standards, method blanks, environmental samples and QC samples.

   - If the extracted ion chromatogram profile (EICP) area for any of the internal standards changes by a factor of two (-50% to +100%) from the last continuing calibration, re-analysis of the samples is required after corrective action.

8. Matrix Spike (MS) Analysis

   - For each extraction/analysis batch of 20 or fewer samples, for each sample matrix

   - MS solutions are to contain all specified compounds of interest.

9. Sample Duplicate or Matrix Spike Duplicate (MSD) Analysis

   - For each extraction/analysis batch of 20 or fewer samples, for each sample matrix

10. Laboratory QC Check Sample

    - At the beginning of the QC program and as needed

11. Method Detection Limits Determination

    - At the beginning of the QC program and as needed

III. QC REQUIREMENTS SUMMARY

III.B. Pesticides/PCBs

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. Initial Calibration

   - At beginning of the QC program
   - Whenever the percent difference (%D) in calibration factors (CF) between continuing calibration and initial calibration exceeds ±15%
   - After installation of a new column or after maintenance service/repair of the gas chromatography (GC)

2. Daily Calibration

   - Prior to the analysis of environmental samples, on each day that samples are analyzed

3. Mid-level Standard

   - After each group of 10 samples
   - Report the percent breakdown for 4,4'-DDT and for endrin.

4. Method Blank

   - For each extraction batch of 20 or fewer samples, for each sample matrix. Analyze method blanks on all instruments used for sample analysis.
   - Method blanks must demonstrate that the analytical system is free of contaminants and interfering peaks.

5. Calibration Range

   - For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. Surrogate Standard

   - Surrogate standards are to be added to the calibration standards, method blanks, environmental samples and QC samples.

7. Matrix Spike (MS) Analysis

   - For each extraction batch of 20 or fewer samples, for each sample matrix

   - MS solutions are to contain all specified compounds of interest.

8. Sample Duplicate or Matrix Spike Duplicate (MSD) Analysis

   - For each extraction batch of 20 or fewer samples, for each sample matrix

9. Laboratory QC Check Sample

   - At beginning of the QC program and as needed

10. Retention Time Windows Determination

   - For each GC column, to be updated daily

11. Method Detection Limits Determination

   - At beginning of the QC program and as needed

III. QC REQUIREMENTS SUMMARY

III.C. Purgeable Organics by GC

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. Initial Calibration

   - At beginning of the QC program
   - Whenever the percent difference (%D) in calibration factors (CF) between continuing calibration and initial calibration exceeds ±15%
   - After installation of a new column or after maintenance service/repair of the gas chromatography (GC)

2. Daily Calibration

   - Prior to the analysis of environmental samples, on each day that samples are analyzed

3. Mid-level Standard

   - After each group of 10 samples

4. Method Blank

   - After each daily calibration and mid-level standard analysis and after the analyses of unusually concentrated samples, to demonstrate that the system is free of contamination.
   - Method blanks should not contain any analytes of interest and are to be free of interfering peaks.

5. Calibration Range

   - For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. Surrogate Standard

- Surrogate standards are to be added to the calibration standards, method blanks, environmental samples and QC samples.

7. Matrix Spike (MS) Analysis

- For each analysis batch of 20 or fewer samples, for each sample matrix
- MS solutions are to contain all specified compounds of interest.

8. Sample Duplicate or Matrix Spike Duplicate (MSD) Analysis

- For each analysis batch of 20 or fewer samples, for each sample matrix

9. Laboratory QC Check Sample

- At beginning of the QC program and as needed

10. Retention Time Windows Determination

- For each GC column, to be updated daily

11. Method Detection Limits Determination

- At beginning of the QC program and as needed

III. QC REQUIREMENTS SUMMARY

III.D. Metals Analyses

QC limits, unless specified below, shall be according to the analytical methods. When QC limits are not specified in the methods, good laboratory practices (GLP) are to be followed. Re-analyses may be necessary when QC limits are not met.

1. **Initial Calibration**

   - Daily and each time the instrument is set up
   - Whenever the percent difference between the initial calibration and the continuing calibration exceeds 10% (20% for mercury)
   - Whenever the percent difference between either of the ICP interference check samples and the true value exceeds 20%.
   - Blank standard required as part of initial calibration

2. **Continuing Calibration Verification Standard**

   - After every ten or fewer samples
   - Analyses are required to have calibrations with acceptable recoveries (the percent difference between the initial calibration and the continuing calibration less than 10% [20% for mercury]) before and after the sample analysis.

3. **Blanks**

   - Continuing calibration blank run immediately following continuing calibration verification standard
   - Method blank for each preparation batch of 20 or fewer samples, for each sample matrix

4. **ICP Interference Check Sample**

   - At the beginning and at the end of the analytical run
   - ICP analyses are required to have both ICP interference check samples with acceptable recoveries (the percent difference between the true value and the ICP interference check sample less than 20%).

5. Calibration Range

- For samples containing one or more analytes at concentrations above the initial calibration range, the samples are to be diluted and re-analyzed.

6. Matrix Spike (MS) Analysis

- For each preparation batch of 20 or fewer samples, for each sample matrix
- MS solutions are to contain all specified compounds of interest.

7. Sample Duplicate Analysis

- For each preparation batch of 20 or fewer samples, for each sample matrix

8. Laboratory Control Sample (LCS)

- For each preparation batch of 20 or fewer samples, for each sample matrix
- Analyses are required to have the laboratory check sample with acceptable recoveries (the percent difference between the true value and the laboratory check sample less than 20%).
- Laboratory control samples are not required for mercury or cyanide determinations.

9. Graphite Furnace Post Digest QC

- A post digest spike at 10 to 20 ug/L is required for all furnace analyses. If the result is greater than or equal to 10 ug/L in the digestate and the recovery of the spike is not within 85% to 115%, the method of standard additions is required to be used.
- If the method of standard additions correlation coefficient is less than 0.995, the method of standard additions analysis is required to be repeated once.

10. ICP Serial Dilution

- For each preparation batch of 20 or fewer samples, for each sample matrix, dilute the digestate by five and re-analyze.