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| Effective Date: 6/17/08 | Next Review Date: 6/17/13 | |

Waste & Environmental Services

Standard Operating Procedure

for **ROUTINE VALIDATION OF ORGANOCHLORINE
PESTICIDE (PEST) AND POLYCHLORINATED
BIPHENYL (PCB) ANALYTICAL DATA**

APPROVAL SIGNATURES:

| Subject Matter Expert: | Organization | Signature | Date |
|-------------------------------|--------------|-------------------|---------|
| Bill Hardesty | WES-EDA | Signature on file | 4/21/08 |
| Quality Assurance Specialist: | Organization | Signature | Date |
| Laura Ortega | QA-IQ | Signature on file | 4/30/08 |
| Responsible Line Manager: | Organization | Signature | Date |
| Craig Eberhart | WES-EDA | Signature on file | 4/21/08 |

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1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine Pesticide/PCB analytical data. This procedure is a mandatory document and shall be implemented by all Los Alamos National Laboratory (LANL or Laboratory) personnel and contractors who evaluate routine Pesticide/PCB analytical data for the specific LANL projects.

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

This procedure conforms to the requirements of Environmental Protection Agency (EPA) Methodologies and the EPA document, "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review." LANL data validation is performed according to procedures based upon the NNSA Model Data Validation Procedure. Data qualifiers and reason codes are assigned according to the specifications in this method specific procedure.

2.2 Precautions

Nothing in this procedure precludes the data validator from going beyond the minimum requirements specified within this procedure. If additional directions are required, the data validator shall reference NNSA Model Data Validation Procedure, EPA method specific guidelines and/or National Functional Guidelines for Organic Data Review. Implementation of this procedure may be followed by a more focused and data use-specific evaluation of the data by the project chemist, especially if the implementation of this procedure indicates the data may contain technical deficiencies.

3.0 EQUIPMENT AND TOOLS

None.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Qualifications for Data Validators

| | | |
|----------------|----|--|
| Data Validator | 1. | Possess a minimum of a bachelor's degree in chemistry, or one of the physical sciences |
| | | AND |
| | | either two (2) years of experience in generating analytical data in an environmental analytical laboratory |
| | | AND |
| | | two (2) years of data validation experience. |
| | 2. | Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist, during data validation. |
| | 3. | Refer to Attachment 3, Guidance for the Qualifier and Reason Code Application, for additional guidance. |

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4.2 Records

- Data Validator 1. Submit the following records generated by this procedure to the Records Processing Facility:
- Completed Data Validation Cover Sheets; and
 - Completed Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklists.

5.0 PROCESS FLOW CHART

For specific validation criteria follow the NNSA Model for Data Validation.

6.0 ATTACHMENTS

- Attachment 1 5163-1 Data Validation Cover Sheet (1 page)
- Attachment 2 5163-2 Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist (4 pages)
- Attachment 3 5163-3 Guidance for the Qualifier and Reason Code Application (4 pages)

7.0 REVISION HISTORY

Author: Bill Hardesty

| Revision No. <i>[Enter current revision number, beginning with Rev.0]</i> | Effective Date <i>[DCC inserts effective date for revision]</i> | Description of Changes <i>[List specific changes made since the previous revision]</i> | Type of Change <i>[Technical (T) or Editorial (E)]</i> |
|--|--|---|---|
| 0 | | New Document | T |

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ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

5163-1

Example of a Data Validation Cover Sheet

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Section I.

REQUEST NUMBER: _____ VALIDATION DATE: _____ LAB CODE: _____

CONTRACT LABORATORY NAME: _____

VALIDATOR: _____ ORGANIZATION: _____

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

- | | | | |
|--|--|---|--|
| <input type="checkbox"/> TPH-GRO | <input type="checkbox"/> HIGH EXPLOSIVES | <input type="checkbox"/> DIOXIN FURANS | <input type="checkbox"/> LCMSMS PERCHLORATES |
| <input type="checkbox"/> TPH-DRO | <input type="checkbox"/> METALS | <input type="checkbox"/> PCB CONGENERS | <input type="checkbox"/> ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS |
| <input type="checkbox"/> GENERAL CHEMISTRY | <input type="checkbox"/> RADIOCHEMISTRY | <input type="checkbox"/> LCMSMS HIGH EXPLOSIVES | |

OTHER (DESCRIBE):

Section II. Completeness Check

| YES | NO | N/A | (CHECK ONE) | YES | NO | N/A | (CHECK ONE) |
|--------------------------|--------------------------|--------------------------|-----------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 1. CHAIN-OF-CUSTODY FORM(S) | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 6. RAW/BSS DATA |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 2. CASE NARRATIVE | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 7. QUALITY CONTROL FORMS |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 3. SAMPLE RESULT FORMS | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 8. QUANTITATION REPORTS |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 4. SAMPLE CHROMATOGRAMS | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 9. TICS FORMS |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 5. STANDARD CHROMATOGRAMS | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 10. TICS MASS SPECTRA |

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

VALIDATOR'S SIGNATURE: _____ DATE: _____

SOP-5163, Revision 0.0

LOS ALAMOS
Environmental Restoration Project

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ATTACHMENT 2: ORGANOCHLORINE PESTICIDE (PEST) AND POLYCHLORINATED BIPHENYL (PCB) ANALYTICAL DATA VALIDATION CHECKLIST

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Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist

Records Use only



| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|---------------------------|--------------------------|--------------------------|--|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 1. The holding time was >1 and ≤2 times the applicable holding time requirement. | UJ, P9 | J-, P9 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 2. The holding time was >2 times the applicable holding time requirement. | R, P9a | J-, P9a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 3. The affected analytes are regarded as rejected because the analytical holding time was exceeded. | R, P9b | R, P9b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 4. The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit. | UJ, R, P7 | J, P7 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 5. The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is <0.995. | UJ, P7a | J, P7a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 6. The Initial Calibration Verification (ICV) and/or Continuing Calibration Verification (CCV) were recovered outside the method-specific limits. | UJ, P7c | J, P7c |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 7. The ICV and/or CCV were not analyzed at the appropriate method frequency. | UJ, P7d | J, P7d |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 8. The multicomponent standard was not analyzed within 72 hours of the initial analysis. | N/A | J, P7e |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 9. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information. | R, P7f | R, P7f |

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| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|---------------------------|--------------------------|--------------------------|--|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 10. The breakdown criteria have been exceeded. This can cause low bias in reported results. If compound is detected, qualify J-. If compound is not present, but breakdown products are present, qualify R. If no compounds or breakdown products are present, qualify UJ (4,4' DDT and Endrin). | UJ, R, P13 | J-, P13 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 11. The breakdown criteria have been exceeded. This can cause high bias in the reported results and potential false positive results for the breakdown products Endrin ketone, Endrin aldehyde, DDD, and DDE. | UJ, P13a | J+, P13a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 12. The breakdown documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, P13b | R, P13b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 13. The sample result is $\leq 5X$ the concentration of the related analyte in the method blank. | N/A | U, P4 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 14. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was greater than 5X. | N/A | J, P4a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 15. The sample result is $\leq 5X$ the concentration of the related analyte in the instrument blank and continuing calibration blank. | N/A | U, P4b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 16. The sample result is $\leq 5X$ the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank. | N/A | U, P4d |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 17. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, P4e | R, P4e |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 18. The analyte RT shifted by more than 0.05 minutes from the mid-level standard of the initial calibration. | R, P0 | J, P0 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 19. Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, P0b | R, P0b |

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| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|---------------------------|--------------------------|--------------------------|--|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 20. The surrogate is <10%R. Follow the external laboratory limits located within the associated data package. | R, P3 | J-, P3 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 21. The surrogate is < the Lower Acceptance Level (LAL) but ≥10%R. Follow the external laboratory limits located within the associated data package. | UJ, P3a | J-, P3a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 22. The surrogate %R value is > the UAL. Follow the external laboratory limits located within the associated data package. | N/A | J+, P3b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 23. At least one surrogate is > the Upper Acceptance Limit (UAL) and one surrogate is < the LAL. Follow the external laboratory limits located within the associated data package. | UJ, P3c | J, P3c |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 24. Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, P3d | R, P3d |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 25. The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package. | R, P12 | J-, P12 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 26. The LCS percent recovery was < the LAL but >10%. Follow the external laboratory limits located within the associated data package. | UJ, P12a | J-, P12a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 27. The LCS percent recovery was > the UAL. Follow the external laboratory limits located within the associated data package. | N/A | J+, P12b |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 28. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, P12c | R, P12c |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 29. The analyte was not confirmed on a second dissimilar column. | N/A | R, P8 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 30. The second dissimilar column documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. | R, P8a | R, P8a |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 31. Duplicate, Dilution, or reanalysis. | UJ, P88 | J, P88 |

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| Yes No N/A (Check One) | | | | Assign Qualifier Listed Below If Criterion = Yes | |
|-------------------------------|--------------------------|--------------------------|---|---|---------------------|
| | | | | Non-detected Analyte | Detected Analyte |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 32. The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytes identified due to matrix interference. Qualify as Reject if the analytical laboratory cannot provide proof for matrix interference. | UJ, R, P15 | R, P15 |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 33. Qualification of data via data validation did not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifiers found within the Form I analytical data summary sheets generated by the external laboratory. | U, U_LAB | J, J_LAB, NQ, NQ |
| <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | 34. The LANL project chemist identified quality deficiencies in the reported data that requires further qualification. This code can only be used and/or under advisement by the LANL project chemist. | UJ, R, P19 | J, R, P19 |

ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

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Guidelines for the Qualifier and Reason Code Application

Records Use only



| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|-------------------|--|
| 1 | R | J | P0 | The analyte RT shifted by >0.05 minutes from the mid-level standard of the initial calibration. |
| 2 | R | R | P0b | Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 3 | R | J- | P12 | The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package. |
| 4 | UJ | J- | P12a | The LCS percent recovery was < the Lower Acceptance Limit (LAL) but >10%. Follow the external laboratory limits located within the associated data package. |
| 5 | N/A | J+ | P12b | The LCS percent recovery was > the Upper Acceptance Limit (UAL). Follow the external laboratory limits located within the associated data package. |
| 6 | R | R | P12c | The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information located within the associated data package. |

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| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|-------------------|--|
| 7 | UJ, R | J- | P13 | The breakdown criteria have been exceeded. This can cause low bias in reported results. If compound is detected, qualify J-. If compounds not present, but breakdown products are present, qualify R. If compounds and no breakdown products are present, qualify UJ (4,4' DDT and Endrin). |
| 8 | UJ | J+ | P13a | The breakdown criteria have been exceeded. This can cause high bias in the reported results and potential false positive results for the breakdown products Endrin ketone, Endrin aldehyde, DDD, and DDE. |
| 9 | R | R | P13b | The breakdown documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 10 | UJ, R | R | P15 | The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytes identified due to matrix interference. Qualify as Reject if the analytical laboratory cannot provide proof for cleanup or matrix interference. |
| 11 | UJ, R | J, R | P19 | The project chemist identified quality deficiencies in the reported data that requires further qualification. This code can ONLY be used and/or under advisement by the project chemist. |
| 12 | R | J- | P3 | The surrogate is <10%R, which indicates the potential for a severely low bias in the results. Follow the external laboratory limits located within the associated data package. |
| 13 | UJ | J- | P3a | The surrogate is < the LAL but ≥10%R, which indicates the potential for a low bias in the results. Follow the external laboratory limits. |
| 14 | N/A | J+ | P3b | The surrogate %R value is > the UAL, which indicates a potential for a high bias in the results and a potential for false positive results. Follow the external laboratory limits located within the associated data package. |
| 15 | UJ | J | P3c | At least one surrogate is > the UAL and one surrogate is < the LAL, which indicates a > normal degree of uncertainty in the result. Follow the external laboratory limits located within the associated data package. |

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| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|-------------------|---|
| 16 | R | R | P3d | Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 17 | N/A | U | P4 | The sample result is $\leq 5X$ the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank. |
| 18 | N/A | J | P4a | The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was $>5X$. |
| 19 | N/A | U | P4b | The sample result is $\leq 5X$ the concentration of the related analyte in the instrument and CCB, which indicates the reported detection is considered indistinguishable from contamination in the blank. |
| 20 | N/A | U | P4d | The sample result is $\leq 5X$ the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank, which indicates the reported detection is considered indistinguishable from contamination in the blank. |
| 21 | R | R | P4e | Required blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 22 | UJ, R | J | P7 | The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit. |
| 23 | UJ | J | P7a | The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is <0.995 . |
| 24 | UJ | J | P7c | The ICV and/or CCV were recovered outside the method-specific limits. |
| 25 | UJ | J | P7d | The ICV and/or CCV were not analyzed at the appropriate method frequency. |
| 26 | N/A | J | P7e | The multicomponent standard was not analyzed within 72 hours of the initial analysis. |
| 27 | R | R | P7f | Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information. |

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| No. | Valid Flag Code Nondetect | Valid Flag Code Detect | Valid Reason Code | Valid Reason Description |
|-----|---------------------------|------------------------|---------------------|--|
| 28 | N/A | R | P8 | The analyte was not confirmed on a second dissimilar column. |
| 29 | UJ | J | P88 | Duplicate, dilution, or reanalysis. |
| 30. | R | R | P8a | The required dissimilar column documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information. |
| 31. | UJ | J- | P9 | The extraction/analytical holding time is exceeded by <2X the published method for holding times. |
| 32. | R | J- | P9a | The extraction/analytical holding time was exceeded by >2X the published method for holding times. |
| 33. | R | R | P9b | The affected analytes are regarded as Rejected because the analytical holding time was exceeded. |
| 34. | U | J, NQ | U_LAB, J_LAB, NQ | Qualification of data via data validation did not occur based on Quality Control requirements in this procedure. Adhere to the external laboratory qualifiers found within the Form I analytical data summary sheets generated by the external laboratory. |

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