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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

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.

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. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
0		New Document	T

Using a CRYPTOCard, click here to record "self-study" training to this procedure.

If you do not possess a CRYPTOCard or encounter problems, contact the EP training specialist.

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

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Example of a Data Validation Cover Sheet

Records Use only

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Section I.

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

CONTRACT LABORATORY NAME: _____

VALIDATOR: _____ ORGANIZATION: _____

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

- ☐ **TPH-GRO**
- ☐ **TPH-DRO**
- ☐ **GENERAL CHEMISTRY**
- ☐ **HIGH EXPLOSIVES**
- ☐ **METALS**
- ☐ **RADIOCHEMISTRY**
- ☐ **DIOXIN FURANS**
- ☐ **PCB CONGENERS**
- ☐ **LCMSMS HIGH EXPLOSIVES**
- ☐ **LCMSMS PERCHLORATES**
- ☐ **ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS**
- ☐ **OTHER (DESCRIBE):** _____

Section II. Completeness Check

YES	NO	N/A	(CHECK ONE)	YES	NO	N/A	(CHECK ONE)
☐	☐	☐	1. CHAIN-OF-CUSTODY FORM(S)	☐	☐	☐	6. RAW/BSS DATA
☐	☐	☐	2. CASE NARRATIVE	☐	☐	☐	7. QUALITY CONTROL FORMS
☐	☐	☐	3. SAMPLE RESULT FORMS	☐	☐	☐	8. QUANTITATION REPORTS
☐	☐	☐	4. SAMPLE CHROMATOGRAMS	☐	☐	☐	9. TICS FORMS
☐	☐	☐	5. STANDARD CHROMATOGRAMS	☐	☐	☐	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: _____

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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

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Los Alamos NATIONAL LABORATORY EST. 1943

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	**1. The holding time was >1 and ≤2 times the applicable holding time requirement.**	**UJ, P9**	**J-, P9**
☐	☐	☐	**2. The holding time was >2 times the applicable holding time requirement.**	**R, P9a**	**J-, P9a**
☐	☐	☐	**3. The affected analytes are regarded as rejected because the analytical holding time was exceeded.**	**R, P9b**	**R, P9b**
☐	☐	☐	**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**
☐	☐	☐	**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**
☐	☐	☐	**6. The Initial Calibration Verification (ICV) and/or Continuing Calibration Verification (CCV) were recovered outside the method-specific limits.**	**UJ, P7c**	**J, P7c**
☐	☐	☐	**7. The ICV and/or CCV were not analyzed at the appropriate method frequency.**	**UJ, P7d**	**J, P7d**
☐	☐	☐	**8. The multicomponent standard was not analyzed within 72 hours of the initial analysis.**	**N/A**	**J, P7e**
☐	☐	☐	**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**

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	**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**
☐	☐	☐	**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**
☐	☐	☐	**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**
☐	☐	☐	**13. The sample result is ≤5X the concentration of the related analyte in the method blank.**	**N/A**	**U, P4**
☐	☐	☐	**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**
☐	☐	☐	**15. The sample result is ≤5X the concentration of the related analyte in the instrument blank and continuing calibration blank.**	**N/A**	**U, P4b**
☐	☐	☐	**16. The sample result is ≤5X the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank.**	**N/A**	**U, P4d**
☐	☐	☐	**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**
☐	☐	☐	**18. The analyte RT shifted by more than 0.05 minutes from the mid-level standard of the initial calibration.**	**R, P0**	**J, P0**
☐	☐	☐	**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**

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	**20. The surrogate is <10%R. Follow the external laboratory limits located within the associated data package.**	**R, P3**	**J-, P3**
☐	☐	☐	**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**
☐	☐	☐	**22. The surrogate %R value is > the UAL. Follow the external laboratory limits located within the associated data package.**	**N/A**	**J+, P3b**
☐	☐	☐	**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**
☐	☐	☐	**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**
☐	☐	☐	**25. The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package.**	**R, P12**	**J-, P12**
☐	☐	☐	**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**
☐	☐	☐	**27. The LCS percent recovery was > the UAL. Follow the external laboratory limits located within the associated data package.**	**N/A**	**J+, P12b**
☐	☐	☐	**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**
☐	☐	☐	**29. The analyte was not confirmed on a second dissimilar column.**	**N/A**	**R, P8**
☐	☐	☐	**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**
☐	☐	☐	**31. Duplicate, Dilution, or reanalysis.**	**UJ, P88**	**J, P88**

Yes	No	N/A (Check One)		Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
☐	☐	☐	**32. The affected analytes have elevated detection limits and may not meet project DQOs because the sample was diluted without any target analytees identified due to matrix interference. Qualify as Reject if the analytical laboratory cannot provide proof for matrix interference.**	**UJ, R, P15**	**R, P15**
☐	☐	☐	**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**
☐	☐	☐	**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.**

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.**

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 ≤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 ≤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 ≤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.**

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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