

REVISION HISTORY

Document No./Revision No.	Issue Date	Action	Description
OIO-TP-5163 R.0.1	8/19/2015	Major Revision	Periodic Review. Minor revision, changed document type and organization.
ER-AP-20311, R0	4/21/2017	Major Revision	Revised to reflect the National Functional Guidelines for Organic Methods Data Review, January 2017 (EPA-540-R-2017-002) holding time requirements and remove NNSA Model Validation

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1. PURPOSE

This procedure represents the minimum standards for evaluating pesticide / polychlorinated biphenyl (PCB) analytical data.

2. SCOPE

This document is intended to assist in the technical review of analytical data generated by environmental laboratories. Qualification of data is the product of data validation, analytical laboratory analysis, and focused validation that describe validation anomalies and their consequences. This procedure is for validation of pesticide and PCB analytical data generated using dual capillary columns with electron capture detectors or electrolytic conductivity detectors.

3. BACKGROUND

Data qualifiers and reason codes are assigned to analytical results from pesticide and PCB organic compound analyses according to the specifications in this method-specific procedure. These guidelines are developed using the EPA method-specific data quality criteria and/or National Functional Guidelines for Organic Data Review.

4. 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 the 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.

5. PREREQUISITE ACTIONS

Data Validators must:

- 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 or two (2) years of experience in data validation.
- Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist, during data validation.

6. PERFORMANCE

6.1 Validation Process

EIM applies a subset of qualifiers described in this procedure to analytical data using auto-validation subroutines. EIM auto-validation applies qualification to analytical records using tests listed in Attachment 2 that have a Valid Reason Description containing “(AV)”. When the project leader requests a focused validation the assigned data validator completes the following steps to assess all potential analytical data qualification:

- [1] **REVIEW** the qualifiers assigned during EIM auto-validation to verify that qualifiers were assigned consistently with this procedure. If auto-validation qualification is found to be inconsistent with this procedure then the validator initiates a change request using ER-AP-20304, Change Control for Data in the Environmental Information Management (EIM) Database.
- [2] **PRINT** Attachment 1 and **REVIEW** the data package for potential qualification using Attachment 2.
- [3] **NOTE** conditions causing recommendation for qualification and options for qualification.
- [4] **COMPLETE** Attachment 1 and **FORWARD** to the project leader with conditions and options.

The project leader is the responsible party for making the decision of record if validation qualifiers should be assigned and EIM validation records updated. This record of decision is added to comments section of Attachment 1.

Once the decision of record has been made, Attachment 1 is sent to the Sample Management Office (SMO) staff. The SMO staff re-print the data validation record from EIM and add Attachment 1 that includes the record of decision to the final records package.

6.2 Analyte Quantitation

The assignment of the detection status to analytical measurements is the first step of analytical data validation. Most validation qualifiers and validation reason codes are applied based on the measurement's initial detection status. Results that are less than the report method detection limit (RMDL) are qualified as nondetect with the U validation qualifier. Results greater than or equal to the RMDL and less than the report detection limit (RDL) are qualified as detected and estimated with the J validation qualifier. Results greater than or equal to the RDL are qualified as detected with the NQ validation qualifier.

Criteria	Validation Qualifier	Validation Reason Code
Target analyte result is < RMDL; a nondetect	U	U_LAB
Target analyte result is \geq RMDL and < RDL; a detect	J	J_LAB
Target analyte result is \geq RDL; a detect	NQ	NQ

Since a result can have only one validation qualifier and one validation reason code the sequencing of validation steps is important. Analyte quantitation occurs first, then analyte identification, because most other validation functions depend on the correct identification and quantitation of the analytical parameter. When two or more qualifiers can be applied to a record, the qualifier representing the more severe consequence to data usability supersedes the qualifier with less severe consequence. The R validation qualifier has the greatest impact on data usability and supersedes other validation qualifiers.

6.2 Analyte Quantitation (continued)

Order Of Severity	Validation Qualifier	Description
1	R	The reported sample result is classified as rejected due to serious noncompliance regarding quality control acceptance criteria. The presence or absence of the analyte cannot be verified.
2	UJ	The analyte is classified as not detected, with an expectation that the reported result is more uncertain than usual.
3	U	The analyte is classified as not detected.
4	J	The analyte is classified as detected but the reported concentration value is expected to be more uncertain than usual.
5	NQ	No validation qualifier flag is associated with this result, and the analyte is classified as detected.

LANL project chemists may identify quality deficiencies in analytical results affecting analyte quantitation. These deficiencies can include analytical results with detection limits elevated above project data-quality objectives, concentrations above the calibration range of the instrument or method, results exhibiting carryover or detector contamination, large relative percent difference between dual-column detects, chromatographic interference from another analyte, and other quality deficiencies. The reason code of P15 or P19 are applied to affected records by the project chemist to identify these quality deficiencies when they are identified.

6.3 Analyte Identification

The identification of an analytical parameter is the second step of analytical data validation. Identification of pesticides and PCBs using GC/ECD methods depends upon the relative retention time of the compound of interest to the known retention time of the compound in the calibration standard, and simultaneous analyte detection on two dissimilar capillary columns. When relative retention time criteria are not met the P0 series of reason codes are applied to affected parameters. When the analyte is not detected from both columns the P8 series of reason codes are applied to affected parameters.

6.4 Holding Times and Sample Preservation

Sample handling requirements are specified to ensure integrity and defensibility of analytical measurements. Samples are to be prepared and analyzed within specified time limits. Samples are also preserved chemically and physically by controlling temperature and light. When sample handling requirements are not met the P9 series of reason codes are applied to affected samples.

6.5 Initial and Continuing Calibration

Calibration is performed to set the operating range of the instrument and to ensure that the instrument is performing within specifications. The initial calibration and verification is performed prior to the start of analyses. Continuing calibration checks and instrument performance samples are performed periodically during analysis to ensure the instrument is providing accurate results. When initial calibration criteria are not met the P7 series of reason codes are applied to affected analytes in all samples analyzed after the unacceptable initial calibration to the next acceptable initial calibration for that instrument. When continuing calibration criteria are not met the P7 series of reason codes are applied to affected analytes in all samples analyzed after the unacceptable continuing calibration to the next acceptable continuing calibration for that instrument.

6.6 Surrogates

Surrogates are compounds not normally found in the environment, but which have quantitation limits and retention times similar to the analytes of interest in a sample. Surrogates are added to samples, standards, and QC samples to determine the effectiveness of analyte quantitation. Sample results are not adjusted based on surrogate recoveries. When surrogate recovery criteria are not met the P3 series of reason codes are applied to affected samples.

6.7 Breakdown Standards

Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD or DDE. When excessive breakdown is observed the P13 series of reason codes are applied to affected samples.

6.8 Blanks

The Method Blank is an analyte-free matrix that is prepared and analyzed in the laboratory with the samples. The method blank determines contamination from the analytical processes.

Method blanks are prepared with every preparation batch. If more than one method blank is associated with a given sample, qualification is based upon a comparison with the associated blank having the highest concentration of the parameter. When method blank criteria are not met the P4 series of reason codes are applied affected samples.

The Field Blank is an analyte-free matrix opened to the atmosphere at the time of sample collection. Field blanks are used to determine if atmospheric conditions resulted in contamination of samples during sample collection. Samples collected the same day as the field blank that do not meet blank criteria are qualified with the P4 series of reason codes.

The Equipment Blank is an analyte-free matrix poured over or through sample collection equipment. Equipment blanks are used to determine the cleaning effectiveness of sampling equipment between samples. Samples collected using the same tools as the equipment blank that does not meet blank criteria are qualified with the P4 series of reason codes.

6.9 Matrix Spike and Laboratory Control Samples

The laboratory control sample is created by adding known amounts of parameters of interest to an aliquot of a blank matrix. The laboratory control sample is used to evaluate the effect of the analytical process of the recovery of analytes. When laboratory control sample criteria are not met the P12 series of reason codes are applied to all associated samples.

7. RECORDS

Records generated by this procedure will be submitted to the Environmental Protection Records Management Office for document management in accordance with Institutional Records Management Procedure, P1020-1 and EP-AP-10003, Records Management.

- Completed Data Validation Cover Sheets (Attachment 1)
- Completed Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist (Attachment 2)

8. REFERENCES

EP-AP-10003, Records Management

ER-AP-20304, Change Control for Data in the Environmental Information Management (EIM) Database

P1020-1, Laboratory Records Management

9. ATTACHMENTS

Attachment 1: Data Validation Cover Sheet

Attachment 2: Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist

ATTACHMENT 1

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Data Validation Cover Sheet

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 & Cyanide	<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					
<input type="checkbox"/> 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: _____			
ER-AP-20311, R0				Los Alamos Environmental Safety & Health			
				(Attach additional comment sheets as necessary)			

ATTACHMENT 2

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Yes No N/A (Check One)	Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist			Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
Holding Time and Sample Preservation					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. The preserved sample was extracted > 7-day holding time and ≤ 14 days. (AV)	UJ, P9	J-, P9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. The sample extract was analyzed > 40-day holding time and ≤ 80 days. (AV)	R, P9	J-, P9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. The sample was extracted or the extract was analyzed >2x holding time. (AV)	R, P9a	R, P9a
Calibration – Initial and Continuing					
<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 percent relative standard deviation 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. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the Sample Management Office (SMO) or external laboratory for information.	R, P7f	R, P7f
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. The multicomponent standard was not analyzed within 72 hours of the initial analysis.	N/A	J, P7e

ATTACHMENT 2

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Yes No N/A (Check One)			Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist	Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
Breakdown Criteria					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. The breakdown criteria have been exceeded. This can cause low bias in reported results for Endrin and DDT. If the target 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
Blanks					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	13. The sample result is ≤ 5 times the concentration of the related analyte in the method blank. (AV)	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 5 times. (AV)	N/A	J, P4a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. The sample result is ≤ 5 times the concentration of the related analyte in the instrument blank and continuing calibration blank. (AV)	N/A	U, P4b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	16. The sample result is ≤ 5 times the concentration of the related analyte in the trip blank, rinsate blank, or equipment blank. (AV)	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

ATTACHMENT 2

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Yes No N/A (Check One)			Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist	Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
Analyte Identification					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	18. The analyte retention time 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
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	20. The analyte was not confirmed on a second dissimilar column.	UJ, P8	J-, P8
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	21. 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
Surrogates					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	22. The surrogate is <10 percent recovery (%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"/>	23. The surrogate is < the lower acceptance limit (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"/>	24. The surrogate %R value is > the upper acceptance limit (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"/>	25. At least one surrogate is > the 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"/>	26. Required surrogate information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, P3d	R, P3d

ATTACHMENT 2

Yes No N/A (Check One)			Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist	Assign Qualifier Listed Below If Criterion = Yes	
				Non-detected Analyte	Detected Analyte
Laboratory Control Samples					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. The LCS percent recovery was <10%. Follow the external laboratory limits located within the associated data package. (AV)	R, P12	J-, P12
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	28. The laboratory control sample (LCS) %R was < the LAL but >10%. Follow the external laboratory limits located within the associated data package. (AV)	UJ, P12a	J-, P12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. The LCS %R was > the UAL. Follow the external laboratory limits located within the associated data package. (AV)	N/A	J+, P12b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	30. The LCS documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, P12c	R, P12c
Analyte Quantitation					
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	31. The non-detected analytes have elevated detection limits and may not meet project data-quality objectives because the sample was diluted without any target analytes identified as a result of matrix interference. Reject non-detected results if the analytical laboratory cannot provide proof for matrix interference.	UJ, R, P15	NA
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	32. The LANL project chemist identified quality deficiencies in the reported data that require further qualification. This code can only be used under advisement by the LANL project chemist.	UJ, R, P19	J, R, P19
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	33. Qualification of data via data validation did occur, however no data quality control requirements in this procedure were applicable. Adhere to the external laboratory qualifiers found within the Form 1 analytical data summary sheets generated by the external laboratory. (AV)	U, U_LAB	J, J_LAB NQ, NQ (No qualification)