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Environment, Safety, Health Directorate

OIO-DO

Technical Procedure

Routine Validation of Organochlorine Pesticide and Polychlorinated Biphenyl Analytical Data

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Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 2 of 14
	Revision: 0.1	Effective Date: 8/19/2015

REVISION HISTORY

Document Number and Revision <i>[Include revision number, beginning with Revision 0]</i>	Effective Date <i>[Document Control Coordinator inserts effective date]</i>	Description of Changes <i>[List specific changes made since the previous revision]</i>
OIO-TP-5163 R.0.1	8/19/2015	Periodic Review. Minor revision, changed document type and organization.

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 3 of 14
	Revision: 0.1	Effective Date: 8/19/2015

Table of Contents

Technical Procedure	1
Revision History	2
Table of Contents.....	3
1.0 Purpose And Scope	4
2.0 Background And Precautions.....	4
2.1 Background	4
2.2 Precautions.....	4
3.0 Equipment And Tools.....	4
4.0 Step-by-Step Process Description.....	4
4.1 Qualifications for Data Validators.....	4
4.2 Records.....	5
5.0 Process Flow Chart.....	5
6.0 Attachments or Appendices	5
Attachment 1 – 5163-1 Data Validation Cover Sheet.....	6
Attachment 2 – 5163-2 Organochlorine Pesticide (PEST) And Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist	7
Attachment 3 – 5163-3 Guidelines for the Qualifier and Reason Code Application.....	11

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 4 of 14
	Revision: 0.1	Effective Date: 8/19/2015

1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine pesticide/polychlorinated biphenyl (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 U.S. 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 National Nuclear Security Agency (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

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 experience in data validation.
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.

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 5 of 14
	Revision: 0.1	Effective Date: 8/19/2015

4.2 Records

1. Submits 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 Checklist.

5.0 PROCESS FLOW CHART

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

6.0 ATTACHMENTS OR APPENDICES

Attachment 1: *5163-1 Data Validation Cover Sheet*

Attachment 2: *5163-2 Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist*

Attachment 3: *5163-3 Guidance for the Qualifier and Reason Code Application*

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 6 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 1 – 5163-1 DATA VALIDATION COVER SHEET

Page 1 of 1

5161-3	Records Use only
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	<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 Safety & Health
	(Attach additional comment sheets as necessary)

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 7 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 2 – 5163-2 ORGANOCHLORINE PESTICIDE (PEST) AND POLYCHLORINATED BIPHENYL (PCB) ANALYTICAL DATA VALIDATION CHECKLIST

Page 1 of 4

5163-2	Records Use only
Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data Validation Checklist	

Yes	No	N/A		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 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. 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 Sample Management Office (SMO) or external laboratory for information.	R, P7f	R, P7f

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 8 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 2 – 5163-2 ORGANOCHLORINE PESTICIDE (PEST) AND POLYCHLORINATED BIPHENYL (PCB) ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

Page 2 of 4

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 ≤ 5 times 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 5 times.	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.	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.	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 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

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 9 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 2 – 5163-2 ORGANOCHLORINE PESTICIDE (PEST) AND POLYCHLORINATED BIPHENYL (PCB) ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

Page 3 of 4

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 percent recovery (%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"/>	21. The surrogate is < the lower acceptance limit (LAL) but ≥10 %R. Follow the external laboratory limits located within the associated data package.	N/A	J+, P3b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	22. The surrogate %R value is > the upper acceptance limit (UAL). Follow the external laboratory limits located within the associated data package.	UJ, P3c	J, P3c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	23 At least one surrogate is > the UAL and one surrogate is < the LAL. Follow the external laboratory limits located within the associated data package.	R, P3d	R, P3d
<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, P12	J-, P12
<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.	UJ, P12a	J-, P12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	26. The laboratory control sample (LCS) %R was < the LAL but >10%. Follow the external laboratory limits located within the associated data package.	N/A	J+, P12b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. The LCS %R was > the UAL. Follow the external laboratory limits located within the associated data package.	R, P12c	R, P12c
<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.	N/A	R, P8
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. The analyte was not confirmed on a second dissimilar column.	UJ, P3a	J-, P3a
<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

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 10 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 2 – 5163-2 – 5163-2 ORGANOCHLORINE PESTICIDE (PEST) AND POLYCHLORINATED BIPHENYL (PCB) ANALYTICAL DATA VALIDATION CHECKLIST (CONT.)

Page 4 of 4

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"/>	31. Duplicate, dilution, or reanalysis.	UJ, P88	J, P88
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	32. The affected 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. 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 require further qualification. This code can only be used and/or under advisement by the LANL project chemist.	UJ, R, P19	J, R, P19

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 11 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 3 – 5163-3 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

Page 1 of 4

5163-3	Records Use only
Guidelines for the Qualifier and Reason Code Application	

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
1	R	J	P0	The analyte retention time 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 Sample Management Office or external laboratory for information.
3	R	J-	P12	The laboratory control sample (LCS) percent recovery (%R) was <10%. Follow the external laboratory limits located within the associated data package.
4	UJ	J-	P12a	The LCS %R 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 %R 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.
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.

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 12 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 3 – 5163-3 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)

Page 2 of 4

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
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 data-quality objectives because the sample was diluted without any target analytes identified as a result of 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 be used ONLY 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.
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 ≤5 times the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 13 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 3 – 5163-3 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)
Page 3 of 4

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
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 >5 times.
19	N/A	U	P4b	The sample result is ≤5 times the concentration of the related analyte in the instrument and continuous calibration blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
20	N/A	U	P4d	The sample result is ≤5 times 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 percent relative standard deviation criteria and/or the associated multipoint calibration correlation coefficient is <0.995.
24	UJ	J	P7c	The initial calibration verification (ICV) and/or continuing calibration verification (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.
28	N/A	R	P8	The analyte was not confirmed on a second dissimilar column.
29	UJ	J	P88	Duplicate, dilution, or reanalysis.

Title: Routine Validation of Organochlorine Pesticide (PEST) and Polychlorinated Biphenyl (PCB) Analytical Data	No: OIO-TP-5163	Page 14 of 14
	Revision: 0.1	Effective Date: 8/19/2015

ATTACHMENT 3 – 5161-3 GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION (CONT.)

Page 4 of 4

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
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 <2 times the published method for holding times.
32	R	J-	P9a	The extraction/analytical holding time was exceeded by >2 times 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.



Environment, Safety and Health

Electronic Public Reading Room - Posting of Controlled Procedures

Operations Integration Office Management Approval:

Print Name	Signature	Date
Ellena Martinez	<i>Ellena Martinez</i>	3/4/16

Derivative Classifier:

OUO
 UCNI
 Unclassified
 Classified

Print Name	Signature	Date
Larry W. Maassen	<i>Larry Maassen</i>	3/4/16

List of Controlled Documents:

Procedure No.	Title/Description
Air Monitoring (ENV)	
ENV-ES-TPP-003	Technical Project Plan for the Neighborhood Environmental Watch Network (NEWNET)
ENV-ES-TPP-007	Technical Project Plan for the Direct Penetrating Radiation Monitoring Network (DPRNET)
Data Validation (ADESH)	
OIO-TP-5161	Routine Validation of Volatile Organic Compound Analytical Data
OIO-TP-5162	Routine Validation of Semivolatile Organic Compound Analytical Data
OIO-TP-5163	Routine Validation of Organochlorine Pesticide and Polychlorinated Biphenyl Analytical Data
OIO-TP-5165	Routine Validation of Metals Analytical Data
General Field Work	
OIO-TP-222	Shipping/Receiving of Environmental Samples by the Sample Management Office (SMO)
OIO-QP-219	Sample Control and Field Documentation
Soil, Foodstuffs, and Biota Sampling (ENV)	
ENV-ES-TPP-002	Technical Project Plan for Biota Dose Assessment
ENV-ES-TP-003	Collection of Soil and Vegetation Samples for the Environmental Surveillance Program
ENV-ES-TP-004	Produce Sampling
ENV-ES-TP-007	Game Animal Sampling
ENV-ES-TP-006	Sampling Soil and Vegetation at Facility Sites
SOP-5247	Collection of Benthic Macroinvertebrates in the Rio Grande
ENV-ES-TP-008	Collection of Crawfish in the Rio Grande
Well Drilling, Construction, Development, Maintenance, and Abandonment	
ENV-RCRA-QP-010	Land Application of Groundwater