

APPENDIX J

DATA VALIDATION REPORT (LDC)

SWHB SURVEY

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Appendix Table J-1. LDC Third Party Level IV Data Validation QA/QC Review Summary - Sediment Data

Polynuclear Aromatic Hydrocarbons - Data Qualification Summary (1504003-001)

Sample	Compound	% of PAH data affected	Flag	Reason Code (Code)	Explanation	
SWHB-07	Benzo(a)anthracene	2.1%	J (all detects)	Initial calibration (r ²) (BC)	The initial calibration (ICAL) curve did not meet method-specified criteria.	
SWHB-18	Chrysene		UJ (all non-detects)			
	Benzo(b)fluoranthene					
	Benzo(k)fluoranthene					
	Benzo(e)pyrene					
	Benzo(a)pyrene					
	Perylene					
	Indeno(1,2,3-cd)pyrene					
Dibenz(a,h)anthracene						
SWHB-07	Benzo(b)fluoranthene	0.7%	J	Initial calibration verification (%D) (LV)	Low ICV recovery. Analytical result may be biased low.	
SWHB-18	Perylene		UJ			
	Indeno(1,2,3-cd)pyrene					
SWHB-18	Benzo(b)fluoranthene	0.3%	J	Continuing calibration (%D) (CH)	High continuing calibration verification (CCV) recovery. Analytical results may be biased high.	
	Perylene					
	Indeno(1,2,3-cd)pyrene					
SWHB-07	Acenaphthene	0.2%	J, UJ	Surrogate spikes (%R) (LS)		
	Phenanthrene					
SWHB-18	1-Methylphenanthrene	0.7%	J, UJ	Duplicate sample analysis (RPD) (HD)	Potential analytical imprecision.	
	Benzo(a)pyrene					
	Benzo(b)fluoranthene					
	Benzo(e)pyrene					
	Dibenz(a,h)anthracene					
Perylene						
SWHB-07	Naphthalene	0.7%	J, UJ	Laboratory control samples (%R) (LL)	Low LCS recovery. Analytical result may be biased low.	
SWHB-18	1-Methylnaphthalene					
	2-Methylnaphthalene					
SWHB-07	Naphthalene	0.7%	J, UJ	CRM (%) (LP)	Low CRM recovery. Analytical result may be biased low.	
	SWHB-18					1-Methylnaphthalene
						2-Methylnaphthalene

Chlorinated Pesticides- Data Qualification Summary- SDG 1504003-001

Sample	Compound	% of pesticide data affected	Flag	Reason Code (Code)	Explanation
SWHB-07	4,4'-DDT	0.2%	UJ	Initial calibration (r ²) (BC)	The initial calibration (ICAL) curve did not meet method-specified criteria.
SWHB-18					
SWHB-18	beta-BHC	0.1%	UJ	Initial calibration verification (%D) (HV)	High initial calibration verification (ICV) recovery. Analytical results may be biased high.
SWHB-18	beta-BHC	0.2%	UJ	Continuing calibration (%D) (CH)	High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
	4,4'-DDD				

Metals - Data Qualification Summary - SDG 1504003-001

Sample	Compound	% of metal data affected	Flag	Reason Code (Code)	Explanation
SWHB-14	Aluminum	1.3%	J	CRM (HP)	High certified reference material (CRM) recovery. Analytical results may be biased high.
SWHB-19	Antimony				
	Iron				

**Appendix Table J-1. LDC Third Party Level IV Data Validation QA/QC Review Summary -
Sediment Data**

Polychlorinated Biphenyls as Congeners - Data Qualification Summary - SDG 1504003-001

Sample	Compound	% of PCB data affected	Flag	Reason Code (Code)	Explanation
SWHB-07	PCB-169	0.1%	J, UJ	Initial calibration (r ²) (BC)	The initial calibration (ICAL) curve did not meet method-specified criteria.
SWHB-18					
SWHB-07	PCB-003	5.6%	J, UJ	Initial calibration verification (%D) (HV)	High initial calibration verification (ICV) recovery. Analytical results may be biased high.
	PCB-008				
	PCB-018				
	PCB-031				
	PCB-028				
	PCB-033				
	PCB-052				
	PCB-049				
	PCB-044				
	PCB-037				
	PCB-074				
	PCB-070				
	PCB-066				
	PCB-095				
	PCB-056/060				
	PCB-101				
	PCB-099				
	PCB-097				
	PCB-087				
	PCB-081				
	PCB-110				
	PCB-077				
	PCB-151				
	PCB-123				
	PCB-149				
SWHB-18	PCB-118				
	PCB-114				
	PCB-153				
	PCB-168/132				
	PCB-105				
	PCB-141				
	PCB-138				
	PCB-158				
	PCB-187				
	PCB-183				
	PCB-128				
	PCB-167				
	PCB-174				
	PCB-177				
	PCB-156				
	PCB-157				
	PCB-199/200				
	PCB-180				
	PCB-169				
	PCB-170				
	PCB-201				
	PCB-189				
	PCB-195				
	PCB-206				
	PCB-209				

Appendix Table J-1. LDC Third Party Level IV Data Validation QA/QC Review Summary - Sediment Data

Sample	Compound	% of PCB data affected	Flag	Reason Code (Code)	Explanation
SWHB-18	PCB-126	0.5%	J, UJ	Continuing calibration (%D) (CH)	High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
	PCB-128				
	PCB-156				
	PCB-169				
	PCB-189				
	PCB-195				
	PCB-194				
SWHB-18	PCB-206	0.4%	J, UJ	Duplicate sample analysis (RPD) (HD)	Potential analytical imprecision.
	PCB-031				
	PCB-028				
	PCB-1 01				
	PCB-110				
	PCB-153				
	PCB-157				
PCB-158					

J = (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.

U = (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).

UJ = (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.

Appendix Table J-2. LDC Third Party Level IV Data Validation QA/QC Review Summary - Tissue Data

Polynuclear Aromatic Hydrocarbons - Data Qualification Summary- SDG 1504003-002

Sample ID	Compound	% of PAH data affected	Flag	Reason Code (Code)	Explanation	
SWHB-27-P	Benzo(b)fluoranthene	0.2%	J	Initial calibration verification (%D) (HV)	High initial calibration verification (ICV) recovery. Analytical results may be biased high.	
	Benzo(k)fluoranthene					
	Benzo(e)pyrene					
	Benzo(a)pyrene					
SWHB-30-CH	Benzo(b)fluoranthene	1.6%	J, UJ	Continuing calibration (%D) (LC)	Low CCV recovery. Analytical result may be biased low.	
SWHB-06-CH-Small	Benzo(a)anthracene					
SWHB-06-M	Chrysene					
SWHB-22-SP	Benzo(k)fluoranthene					
	Benzo(e)pyrene					
	Benzo(a)pyrene					
	Perylene					
SWHB-27-P	Benzo(a)pyrene	0.2%	J	Continuing calibration (%D) (LC)	Low CCV recovery. Analytical result may be biased low.	
	Benzo(b)fluoranthene					
	Benzo(e)pyrene					
SWHB-27-SBB	1-Methylnaphthalene	0.1%	J	Duplicate sample analysis (RPD) (HD)	Potential analytical imprecision.	
	2,6-Dimethylnaphthalene					
SWHB-26-M	1-Methylnaphthalene	4.9%	U, UJ	Laboratory control samples (%R) (LL)	Low LCS recovery. Analytical result may be biased low.	
SWHB-27-SBB	2,3,5-Trimethylnaphthalene					
SWHB-27-P	2,6-Dimethylnaphthalene					
SWHB-30-CH	2-Methylnaphthalene					
SWHB-06-CH-Small	Acenaphthene					
SWHB-06-M	Acenaphthylene					
SWHB-22-SP	Anthracene					
	Biphenyl					
	Dibenzothiophene					
	Fluorene					
	Naphthalene					
SWHB-22-SP	Phenanthrene	5.8%	U, UJ	Laboratory control samples (RPD) (HD)	Potential analytical imprecision.	
	SWHB-26-M					1-Methylnaphthalene
	SWHB-27-SBB					2,3,5-Trimethylnaphthalene
	SWHB-27-P					2,6-Dimethylnaphthalene
	SWHB-30-CH					2-Methylnaphthalene
	SWHB-06-CH-Small					Acenaphthene
	SWHB-06-M					Acenaphthylene
	SWHB-22-SP					Anthracene
						Biphenyl
						Dibenzothiophene
Fluorene						
Naphthalene						
Phenanthrene						
	Pyrene					

Appendix Table J-2. LDC Third Party Level IV Data Validation QA/QC Review Summary - Tissue Data

Chlorinated Pesticides - Data Qualification Summary - SDG 1504003-002

Sample	Compound	% of pesticide data affected	Flag	Reason Code (Code)	Explanation
SWHB-26-M	4,4'-DDT	0.8%	UJ	Initial calibration (r ²) (BC)	The initial calibration (ICAL) curve did not meet method-specified criteria.
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-27-SBB					
SWHB-27-P					
SWHB-30-CH					
SWHB-22-SP	2,4'-DDD	0.9%	J, UJ	Continuing calibration (%D) (CH)	High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-30-CH					
SWHB-22-SP	Toxaphene	0.5%	J, UJ	Continuing calibration (%D) (LC)	Low CCV recovery. Analytical result may be biased low.
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-30-CH	2,4'-DDD	0.3%	J, UJ	Duplicate sample analysis (RPD) (HD)	Potential analytical imprecision.
SWHB-22-SP					
SWHB-27-SBB					
	2,4'-DDE				
	alpha-Chlordane				

Metals- Data Qualification Summary- SDG 1504003-002

Sample	Compound	% of metals data affected	Flag	Reason Code (Code)	Explanation
SWHB-26-SBB	Mercury	1.7%	J	Matrix spike/Matrix spike duplication (%R) (HM)	High MS recovery. Analytical results may be biased high.
SWHB-26-CH	Selenium	11.9%	J	Internal standards (%R) (*XII)	
SWHB-26-SP-Large					
SWHB-26-BP					
SWHB-27-SP					
SWHB-01-SBB					
SWHB-01-CH					
SWHB-26-SBB					

Polychlorinated Biphenyls as Congeners - Data Qualification Summary - SDG 1504003-002

Sample	Compound	% of PCB data affected	Flag	Reason Code (Code)	Explanation
SWHB-26-M	PCB-169	0.2%	UJ	Initial calibration (r ²) (BC)	The initial calibration (ICAL) curve did not meet method-specified criteria.
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-27-SBB					
SWHB-27-P					
SWHB-30-CH					
SWHB-22-SP	PCB-126	2.1%	J, UJ	Initial calibration (%D) (HV)	High initial calibration verification (ICV) recovery. Analytical results may be biased high.
SWHB-26-M					
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-27-SBB					
SWHB-27-P					
SWHB-30-CH					
SWHB-22-SP					
	PCB-189				
	PCB-194				
	PCB-206				

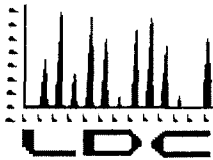
Appendix Table J-2. LDC Third Party Level IV Data Validation QA/QC Review Summary - Tissue Data

Sample	Compound	% of PCB data affected	Flag	Reason Code (Code)	Explanation
SWHB-30-CH	PCB-126	0.8%	J, UJ	Continuing calibration (%D) (CH)	High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
SWHB-06-CH-Small	PCB-128				
SWHB-06-M	PCB-177				
SWHB-22-SP	PCB-156				
	PCB-169				
	PCB-170				
SWHB-27-SBB	PCB-153	0.03%	J	MS/MSD duplicate (%R) (LM)	Low MS recovery. Analytical results may be biased low.
SWHB-27-SBB	PCB-138	0.03%	J	MS/MSD duplicate (%R) (HM)	High MS recovery. Analytical results may be biased high.
SWHB-27-SBB	PCB-099	1.0%	J	MS/MSD duplicate (RPD) (HD)	Potential analytical imprecision.
	PCB-101				
	PCB-118				
	PCB-128				
	PCB-138				
	PCB-153				
SWHB-27-SBB	PCB-180	0.1%	J	Duplicate sample analysis (RPD) (HD)	Potential analytical imprecision.
	PCB-028				
	PCB-044				
	PCB-070				
SWHB-26-M	PCB-018	0.5%	J, UJ	Laboratory control samples (%R) (LL)	Low LCS recovery. Analytical result may be biased low.
SWHB-27-SBB	PCB-028				
SWHB-27-P					
SWHB-30-CH					
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-22-SP					
SWHB-26-M		PCB-018	0.7%	J, UJ	Laboratory control samples (RPD) (HD)
SWHB-27-SBB	PCB-028				
SWHB-27-P	PCB-044				
SWHB-30-CH					
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-22-SP					
SWHB-26-M	PCB-018	0.2%	UJ	CRM (%R) (LP)	Low CRM recovery. Analytical result may be biased low.
SWHB-27-SBB					
SWHB-27-P					
SWHB-30-CH					
SWHB-06-CH-Small					
SWHB-06-M					
SWHB-22-SP					

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U = (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).

UJ = (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.



LABORATORY DATA CONSULTANTS, INC.

2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760-827-1100 Fax: 760-827-1099

AMEC
9210 Sky Park Court, Suite 200
San Diego, CA 92123
ATTN: Mr. Rolf Schottle

June 8, 2016

SUBJECT: City of San Diego, SWBH Study, Data Validation

Dear Mr. Schottle,

Enclosed are the final validation reports for the fractions listed below. These SDGs were received on April 18, 2016. Attachment 1 is a summary of the samples that were reviewed for each analysis.

LDC Project #36197:

<u>SDG #</u>	<u>Fraction</u>
1504003-001	Polynuclear Aromatic Hydrocarbons, Polybrominated Diphenyl
1504003-002	Ethers, Pyrethroids, Chlorinated Pesticides, Metals, Wet Chemistry, Polychlorinated Biphenyls as Congeners

The data validation was performed under Level IV guidelines. The analyses were validated using the following documents, as applicable to each method:

- Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California, August 2013
- USEPA, Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, June 2008
- USEPA Contract Laboratory National Functional Guidelines for Inorganic Superfund Data Review, January 2010
- EPA SW 846, Third Edition, Test Methods for Evaluating Solid Waste, update 1, July 1992; update IIA, August 1993; update II, September 1994; update IIB, January 1995; update III, December 1996; update IIIA, April 1998; IIIB, November 2004; Update IV, February 2007, Update V, July 2014

Please feel free to contact us if you have any questions.

Sincerely,

Pei Geng
Project Manager/Senior Chemist

Level IV Client Select

LDC #36197 (AMEC FW-San Diego, CA / City of San Diego SWBH Study)

LDC	SDG#	DATE REC'D	(3) DATE DUE	PAHs (8270D)		Pest. (8270D)		PCB Cong. (8270D)		PDEs (8270D -NCI)		Pyrethroids (8270D)		Metals (6020)		Se (6020)	Hg (245.7)	NH ₃ -N (4500-NH3 D)		% Solids (2540B)		% Lipids (Grav.)																		
				T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S			
Matrix: Tissue/Sediment																																								
A	1504003-001	04/18/16	05/09/16	0	2	0	2	0	2	0	2	0	2	0	2	-	-	0	2	0	2	-	-																	
B	1504003-002	04/18/16	05/09/16	7	0	7	0	7	0	7	0	-	-	-	-	7	0	7	0	-	-	7	0	7	0															
Total																																								

Shaded cells indicate Level IV validation (all other cells are Level III review). These sample counts do not include DL, RE, MS, MSD, or DUP's.

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study
LDC Report Date: May 9, 2016
Parameters: Polynuclear Aromatic Hydrocarbons
Validation Level: Level IV
Laboratory: Physis Environmental Laboratories, Inc.
Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-07	31524	Sediment	04/09/14
SWHB-18	31535	Sediment	04/08/14
SWHB-18MS	31535MS	Sediment	04/08/14
SWHB-18MSD	31535MSD	Sediment	04/08/14
SWHB-18DUP	31535DUP	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polynuclear Aromatic Hydrocarbons (PAHs) by Environmental Protection Agency (EPA) SW 846 Method 8270D

All sample results were subjected to Level IV evaluation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exception:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-07	All TCL compounds	398	365	J (all detects) UJ (all non-detects)	P
SWHB-18	All TCL compounds	399	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For compounds where average relative response factors (RRFs) were utilized, percent relative standard deviations (%RSD) were less than or equal to 20.0%.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
05/26/15	Benzo(a)anthracene Chrysene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(a)pyrene Perylene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene	0.98699728 0.98595152 0.97632544 0.96600546 0.98481106 0.97008131 0.98831313 0.96116178 0.97922814	All samples in SDG 1504003-001	J (all detects) UJ (all non-detects)	A

Average relative response factors (RRF) for all compounds were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
05/28/16	Benzo(b)fluoranthene Perylene Indeno(1,2,3-cd)pyrene	42 38 44	All samples in SDG 1504003-001	J (all detects) UJ (all non-detects)	A

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
05/29/15	Benzo(b)fluoranthene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene	24 52 71	SWHB-18	J (all detects) J (all detects) J (all detects)	A

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogate Spikes

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits with the following exceptions:

Sample	Surrogate	%R (Limits)	Affected Compound	Flag	A or P
SWHB-07	d10-Acenaphthene d10-Phenanthrene	46 (50-150) 48 (50-150)	Acenaphthene Phenanthrene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SWHB-18MS/MSD (SWHB-18)	Benzo(b)fluoranthene Dibenz(a,h)anthracene	- -	174 (50-150) 158 (50-150)	J (all detects) J (all detects)	A

Relative percent differences (RPD) were within QC limits.

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-18DUP (SWHB-18)	1-Methylphenanthrene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(e)pyrene Dibenz(a,h)anthracene Perylene	40 (≤25) 60 (≤25) 32 (≤25) 32 (≤25) 32 (≤25) 40 (≤25)	J (all detects) UJ (all non-detects)	A

IX. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31515-BS1/BS2 (All samples in SDG 1504003-001)	Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene	54 (70-130) - -	49 (70-130) 63 (70-130) 66 (70-130)	J (all detects) UJ (all non-detects)	P

Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits with the following exceptions:

CRM ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
31579-CRM	1-Methylnaphthalene 2-Methylnaphthalene Naphthalene	39 (60-140) 41 (60-140) 36 (60-140)	All samples in SDG 1504003-001	J (all detects) UJ (all non-detects)	P

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Internal Standards

All internal standard areas and retention times were within QC limits.

XII. Compound Quantitation

All compound quantitations were within validation.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Compound reported below the RL and above the MDL	J (all detects)	A

XIII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIV. System Performance

The system performance was acceptable.

XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , ICV and continuing calibration %D, surrogate %R, MS/MSD %R, DUP RPD, LCS/LCSD %R, CRM %R, and results reported below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
 Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG
 1504003-001**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-07 SWHB-18	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-07 SWHB-18	Benzo(a)anthracene Chrysene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(a)pyrene Perylene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene	J (all detects) UJ (all non-detects)	A	Initial calibration (r^2) (BC)
SWHB-07 SWHB-18	Benzo(b)fluoranthene Perylene Indeno(1,2,3-cd)pyrene	J (all detects) UJ (all non-detects)	A	Initial calibration verification (%D) (LV)
SWHB-18	Benzo(b)fluoranthene Indeno(1,2,3-cd)pyrene Dibenz(a,h)anthracene	J (all detects) J (all detects) J (all detects)	A	Continuing calibration (%D) (CH)
SWHB-07	Acenaphthene Phenanthrene	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Surrogate spikes (%R) (LS)
SWHB-18	Benzo(b)fluoranthene Dibenz(a,h)anthracene	J (all detects) J (all detects)	A	Matrix spike/Matrix spike duplicate (%R) (HM)
SWHB-18	1-Methylphenanthrene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(e)pyrene Dibenz(a,h)anthracene Perylene	J (all detects) UJ (all non-detects)	A	Duplicate sample analysis (RPD) (HD)
SWHB-07 SWHB-18	Naphthalene 1-Methylnaphthalene 2-Methylnaphthalene	J (all detects) UJ (all non-detects)	P	Laboratory control samples (%R) (LL)
SWHB-07 SWHB-18	1-Methylnaphthalene 2-Methylnaphthalene Naphthalene	J (all detects) UJ (all non-detects)	P	Certified material reference (%R) (LP)
SWHB-07 SWHB-18	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification
Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -
SDG 1504003-001**

No Sample Data Qualified in this SDG

LDC #: 36197A2b

VALIDATION COMPLETENESS WORKSHEET

Date: 5/3/16

SDG #: 1504003-001

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: GC/MS Polynuclear Aromatic Hydrocarbons (EPA SW 846 Method 8270D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / MW	
II.	GC/MS Instrument performance check	A	
III.	Initial calibration/ICV	MW/MW	RSOS = 20%. r^2 ICV = 30%
IV.	Continuing calibration	MW	CCV = 20%
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	MW	
VIII.	Matrix spike/Matrix spike duplicates	1 DUP / MW	
IX.	Laboratory control samples	1 CRM / MW	LCs/D. CRM
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinsate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-07	31524	Sediment	04/09/14
2	SWHB-08/18	34526 31535	Sediment	04/09/14
3	↓ MS	↓ MS	↓	↓
4	↓ MSD	↓ MSD	↓	↓
5	↓ DUP	↓ DUP	↓	↓
6				
7				
8				

Notes:

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?			/	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% and relative response factors (RRF) within method criteria?	/			
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990?		/		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	/			
Were all percent differences (%D) < 30% or percent recoveries (%R) 70-130%?		/		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria?		/		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?		/		
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?		/		
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
X. Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds of the associated calibration standard?	/			
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	/			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?	/			
XIV. System performance				
System performance was found to be acceptable.	/			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			

VALIDATION FINDINGS WORKSHEET

METHOD: GC/MS SVOA

A. Phenol	T. 4-Chloroaniline	MM. 4-Chlorophenyl-phenyl ether	FFF. Di-n-octylphthalate	YYY. 2,3,5-Trimethylnaphthalene
B. Bis (2-chloroethyl) ether	U. Hexachlorobutadiene	NN. Fluorene	GGG. Benzo(b)fluoranthene	ZZZ. Perylene
C. 2-Chlorophenol	V. 4-Chloro-3-methylphenol	OO. 4-Nitroaniline	HHH. Benzo(k)fluoranthene	AAAA. Dibenzothiophene
D. 1,3-Dichlorobenzene	W. 2-Methylnaphthalene	PP. 4,6-Dinitro-2-methylphenol	III. Benzo(a)pyrene	BBBB. Benzo(a)fluoranthene
E. 1,4-Dichlorobenzene	X. Hexachlorocyclopentadiene	QQ. N-Nitrosodiphenylamine	JJJ. Indeno(1,2,3-cd)pyrene	CCCC. Benzo(b)fluorene
F. 1,2-Dichlorobenzene	Y. 2,4,6-Trichlorophenol	RR. 4-Bromophenyl-phenylether	KKK. Dibenz(a,h)anthracene	DDDD. cis/trans-Decalin
G. 2-Methylphenol	Z. 2,4,5-Trichlorophenol	SS. Hexachlorobenzene	LLL. Benzo(g,h,i)perylene	EEEE. Biphenyl
H. 2,2'-Oxybis(1-chloropropane)	AA. 2-Chloronaphthalene	TT. Pentachlorophenol	MMM. Bis(2-Chloroisopropyl)ether	FFFF. Retene
I. 4-Methylphenol	BB. 2-Nitroaniline	UU. Phenanthrene	NNN. Aniline	GGGG. C30-Hopane
J. N-Nitroso-di-n-propylamine	CC. Dimethylphthalate	VV. Anthracene	OOO. N-Nitrosodimethylamine	HHHH. 1-Methylphenanthrene
K. Hexachloroethane	DD. Acenaphthylene	WW. Carbazole	PPP. Benzoic Acid	IIII. 1,4-Dioxane
L. Nitrobenzene	EE. 2,6-Dinitrotoluene	XX. Di-n-butylphthalate	QQQ. Benzyl alcohol	JJJJ. Acetophenone
M. Isophorone	FF. 3-Nitroaniline	YY. Fluoranthene	RRR. Pyridine	KKKK. Atrazine
N. 2-Nitrophenol	GG. Acenaphthene	ZZ. Pyrene	SSS. Benzidine	LLLL. Benzaldehyde
O. 2,4-Dimethylphenol	HH. 2,4-Dinitrophenol	AAA. Butylbenzylphthalate	TTT. 1-Methylnaphthalene	MMMM. Caprolactam
P. Bis(2-chloroethoxy)methane	II. 4-Nitrophenol	BBB. 3,3'-Dichlorobenzidine	UUU. Benzo(b)thiophene	NNNN.
Q. 2,4-Dichlorophenol	JJ. Dibenzofuran	CCC. Benzo(a)anthracene	VVV. Benzonaphthothiophene	OOOO.
R. 1,2,4-Trichlorobenzene	KK. 2,4-Dinitrotoluene	DDD. Chrysene	WWW. Benzo(e)pyrene	PPPP.
S. Naphthalene	LL. Diethylphthalate	EEE. Bis(2-ethylhexyl)phthalate	XXX. 2,6-Dimethylnaphthalene	QQQQ.

VALIDATION FINDINGS WORKSHEET Technical Holding Times

All circled dates have exceeded the technical holding times.
Y N N/A Were all cooler temperatures within validation criteria?

METHOD : GC/MS BNA (EPA SW 846 Method 8270D)							
Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifier
1	Sed	Y	4-9-14	5-12-15		398	Y/N/A (H)
2	↓	↓	4-8-14	↓		399	↓
3	↓	↓	↓	↓		↓	↓
4	↓	↓	↓	↓		↓	↓
5	↓	↓	↓	↓		↓	↓
(det STD)							

TECHNICAL HOLDING TIME CRITERIA

Water: Extracted within 7 days, analyzed within 40 days.
Soil: Extracted within 14 days, analyzed within 40 days.

VALIDATION FINDINGS WORKSHEET
Initial Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?
- Y N N/A Were all percent relative standard deviations (%RSD) ≤ 20 and relative response factors (RRF) within method criteria?
- Y N N/A Was a curve fit used for evaluation?
- Y N N/A Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990 ?

#	Date	Standard ID	Compound	Finding %RSD (Limit: $\leq 15.0\%$)	Finding RRF (Limit: ≥ 0.05)	Associated Samples	Qualifications
	<u>5/26/15</u>	<u>10A2</u>	<u>ccc</u>	<u>$r^2 = 0.98699728$</u>		<u>M (dets + ND)</u>	<u>↓ M ↓ A (BC)</u>
			<u>ddd</u>	<u>0.98595152</u>			↓
			<u>eee</u>	<u>0.97632544</u>			
			<u>hhh</u>	<u>0.96600526</u>			
			<u>www</u>	<u>0.98481106</u>			
			<u>lll</u>	<u>0.97008131</u>			
			<u>zzz</u>	<u>0.98831313</u>			
			<u>nn</u>	<u>0.96116178</u>			
			<u>kkk</u>	<u>0.97922814</u>			

VALIDATION FINDINGS WORKSHEET
Initial Calibration Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y/N/N/A Was an initial calibration verification standard analyzed after each ICAL for each instrument?

Y(N)/N/A Were all %D within the validation criteria of ≤30 %D?

#	Date	Standard ID	Compound	Finding %D (Limit: ≤30.0%)	Associated Samples	Qualifications
	<u>5/28/16</u>	<u>ICV</u>	FFF	<u>42</u>	<u>M (lots + ND)</u>	<u>N/A (ICV)</u>
			<u>ZZZ</u>	<u>38</u>		
			<u>VVV</u>	<u>44</u>		

VALIDATION FINDINGS WORKSHEET
Continuing Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?
 Y N N/A Were percent differences (%D) ≤ 20 % and relative response factors (RRF) within the method criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: ≤20.0%)	Finding RRF (Limit)	Associated Samples	Qualifications
	<u>5/9/15</u>	<u>CCV</u>	<u>GGP</u>	<u>24</u>		<u># 2, 5 (dets)</u>	<u>N/A (CH)</u>
			<u>WV</u>	<u>52</u>			
			<u>PKK</u>	<u>71</u>			

VALIDATION FINDINGS WORKSHEET
Surrogate Recovery

METHOD: GC/MS BNA (EPA SW 846 Method 8270C)

Please see qualification below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Were percent recoveries (%R) for surrogates within QC limits?
- Y N N/A If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?
- Y N N/A If any %R was less than 10 percent, was a reanalysis performed to confirm %R?

#	Date	Sample ID	Surrogate	%R (Limits)	Qualifications
		1	d8-S	40.4 (50-150)	
		1	d10-GG	46 (50-150)	→ N/A / P (dots + N/A) ↓
			d10-UU	48 (✓)	
				()	
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Base/Neutral Surrogates:
 (NBZ) = Nitrobenzene-d5
 (FBP) = 2-Fluorobiphenyl
 (TPH) = Terphenyl-d14
 (DCB) = 1,2-Dichlorobenzene-d4

Acid Surrogates:
 (PHL) = Phenol-d5
 (2FP) = 2-Fluorophenol
 (TBP) = 2,4,6-Tribromophenol
 (2CP) = 2-Chlorophenol-d4

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>3/4</u>	GGG <u>KKK</u>	()	<u>174 (5-150)</u>	()	<u>2 (dets)</u>	<u>↓ dets/A (HM)</u>
				()	<u>158 (↓)</u>	()		<u>↓</u>
				()	()	()		
				()	()	()		
		<u>5</u>	<u>HHH</u>	()	()	<u>40 (<25)</u>	<u>2 (dets)</u>	<u>↓ dets/A (HD)</u>
			<u>III</u>	()	()	<u>60 (↓)</u>		<u>↓</u>
			GGG	()	()	<u>32 (↓)</u>		
			<u>WWW</u>	()	()	<u>32 (↓)</u>		
			KKK	()	()	<u>32 (↓)</u>		
			<u>ZZZ</u>	()	()	<u>40 (↓)</u>		<u>↓</u>
				()	()	()		
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VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N/A Was a LCS required?

Y N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>31515-B51</u>	<u>S</u>	<u>54</u> (70-130)	<u>49</u> (70-130)	()	<u>all (dets+ND)</u>	<u>↓ / W / P (LL)</u>
		<u>-B52</u>	<u>TTT</u>	()	<u>63</u> (↓)	()		↓
			<u>W</u>	()	<u>66</u> (↓)	()		↓
				()	()	()		
		<u>31519-CRM</u>	<u>TTT</u>	<u>39</u> (60-140)	()	()	<u>all (dets+ND)</u>	<u>↓ / W / A (LP)</u>
			<u>W</u>	<u>41</u> (↓)	()	()		↓
			<u>S</u>	<u>36</u> (↓)	()	()		↓
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LDC: 26197A26

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
Reviewwe: 9
2nd Reviewer: JVG

Method: PAHs (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/26/2015	Naphthalene	1	0.0250	0.0282062	0.000025
		2	0.0500	0.0548671	0.00005
		3	0.1250	0.1498233	0.0001
		4	0.2500	0.2980483	0.0002
		5	0.5000	0.5702316	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.152617	1.15262
Correlation Coefficient	0.999800	0.99911
Coefficient of Determination (r ²)	0.999601	

LDC: 36197A>6VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation VerificationPage: 1 of 1
Reviewwe: Q
2nd Reviewer: W6

Method: PAHs (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/26/2015	Benzo(g,h,i)perylene	1	0.0250	0.0269938	0.000025
		2	0.0500	0.0655381	0.00005
		3	0.1250	0.1541564	0.0001
		4	0.2500	0.2993241	0.0002
		5	0.5000	0.6249197	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.239282	1.23925
Correlation Coefficient	0.999836	0.99929
Coefficient of Determination (r ²)	0.999671	

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	<u>CCV</u>	<u>5/29/15</u>	Phenol (1st internal standard) <u>S</u>	<u>500</u>	<u>474.129</u>	<u>474.120</u>	<u>5</u>	<u>5</u>
			Naphthalene (2nd internal standard) <u>LL</u>	<u>↓</u>	<u>530.522</u>	<u>530.521</u>	<u>6</u>	<u>6</u>
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET Surrogate Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: $SF/SS * 100$

Where: SF = Surrogate Found
SS = Surrogate Spiked

Sample ID: 1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5 d10-EE	1000	462.96	51	46	5
2-Fluorobiphenyl d10-UV	↓	478.23	78	48	30
Terphenyl-d14 d12-DDD	↓	1006.66	101	101	0
Phenol-d5 d8-S	↓	403.67	40	40	0
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID: _____

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID: _____

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

$\% \text{ Recovery} = 100 * (\text{SSC} - \text{SC}) / \text{SA}$

Where: SSC = Spiked sample concentration
SA = Spike added

SC = Sample concentration

$\text{RPD} = | \text{MSC} - \text{MSC} | * 2 / (\text{MSC} + \text{MSDC})$

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 3/4

Compound	Spike Added (NS/G)		Sample Concentration (NS/G)	Spiked Sample Concentration (NS/G)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc	Reported	Recalc	Reported	Recalculated
44	82.8	87.4	ND	62.8	73.1	76	76	84	84	10	10
22	↓	↓	7.7	100.2	110.3	111	112	117	117	5	5

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 36197A-6

VALIDATION FINDINGS WORKSHEET

Page: 1 of 1

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer: [Signature]

2nd Reviewer: JVB

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SC/SA)

Where: SSC = Spike concentration
SA = Spike added

RPD = |LCSC - LCSDC| * 2 / (LCSC + LCSDC)

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31515-BS1-BS2

Compound	Spike Added (ug/g)		Spike Concentration (ug/g)		LCS		LCSD		LCS/LCSD	
					Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
EE	500	500	428.2	376	86	86	75	75	14	14
ZZ	↓	↓	528	520.9	106	106	104	104	2	2

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 36197A-6

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1

Reviewer: [Signature]

2nd reviewer: NG

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

(Y) (N) (N/A)
(X) (N) (N/A)

Were all reported results recalculated and verified for all level IV samples?

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_s)(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_s = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1, 3:

$$\text{Conc.} = \frac{(26858)(2000)(0.05)(0.203)}{(61344)(1.152)(0.1)} = 1.60 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (ng/g)	Calculated Concentration ()	Qualification
			1.6		

**Laboratory Data Consultants, Inc.
Data Validation Report**

Project/Site Name: City of San Diego SWBH Study
LDC Report Date: May 9, 2016
Parameters: Polybrominated Diphenyl Ethers
Validation Level: Level IV
Laboratory: Physis Environmental Laboratories, Inc.
Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-07	31524	Sediment	04/09/14
SWHB-18	31535	Sediment	04/08/14
SWHB-18MS	31535MS	Sediment	04/08/14
SWHB-18MSD	31535MSD	Sediment	04/08/14
SWHB-18DUP	31535DUP	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polybrominated Diphenyl Ethers (PBDE) by Environmental Protection Agency (EPA) SW 846 Method 8270D using Negative Chemical Ionization (NCI)

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-07	All TCL compounds	398	365	J (all detects) UJ (all non-detects)	P
SWHB-18	All TCL compounds	399	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

Instrument performance check was not required per method.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination (r^2) was greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
06/14/15	PBDE 209	0.97105483	All samples in SDG 1504003-001	J (all detects) UJ (all non-detects)	A

Average relative response factors (RRF) for all compounds were within validation criteria.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
06/04/15	PBDE 190 PBDE 209	36.40 24.02	SWHB-18	UJ (all non-detects) UJ (all non-detects)	A

All of the continuing calibration relative response factors (RRF) were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SWHB-18MS/MSD (SWHB-18)	PBDE 209	-	44 (50-150)	UJ (all non-detects)	A

Relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-18MS/MSD (SWHB-18)	PBDE 209 PBDE 190	48 (≤ 25) 28 (≤ 25)	NA	-

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-18DUP (SWHB-18)	PBDE 047	27 (≤25)	J (all detects)	A

IX. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits with the following exceptions:

CRM ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
31519-CRM1	PBDE 100	145 (60-140)	SWHB-07	J (all detects)	A
31519-CRM1	PBDE 100	145 (60-140)	SWHB-18	NA	-
31519-CRM1	PBDE 209	49 (60-140)	All samples in SDG 1504003-001	J (all detects) UJ (all non-detects)	A

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Internal Standards

All internal standard areas and retention times were within QC limits.

XII. Compound Quantitation

All compound quantitations were within validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Compound reported below the RL and above the MDL	J (all detects)	A

XIII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIV. System Performance

The system performance was acceptable.

XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , continuing calibration %D, MS/MSD %R, DUP RPD, CRM %R, and results reported below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Polybrominated Diphenyl Ethers - Data Qualification Summary - SDG 1504003-001**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-07 SWHB-18	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-07 SWHB-18	PBDE 209	J (all detects) UJ (all non-detects)	A	Initial calibration (r^2) (BC)
SWHB-18	PBDE 190	UJ (all non-detects)	A	Continuing calibration (%D) (LC)
SWHB-18	PBDE 209	UJ (all non-detects)	A	Continuing calibration (%D) (CH)
SWHB-18	PBDE 209	UJ (all non-detects)	A	Matrix spike/Matrix spike duplicate (%R) (LM)
SWHB-18	PBDE 047	J (all detects)	A	Duplicate sample analysis (RPD) (HD)
SWHB-07	PBDE 100	J (all detects)	A	Certified reference material (%R) (HP)
SWHB-07 SWHB-18	PBDE 209	J (all detects) UJ (all non-detects)	A	Certified reference material (%R) (LP)
SWHB-07 SWHB-18	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Polybrominated Diphenyl Ethers - Laboratory Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Polybrominated Diphenyl Ethers - Field Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

LDC #: 36197A2c

VALIDATION COMPLETENESS WORKSHEET

Date: 4/29/16

SDG #: 1504003-001

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: GC/MS Polybrominated Diphenyl Ethers (EPA SW 846 Method 8270D-NCI)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A/W	
II.	GC/MS Instrument performance check	A/N	
III.	Initial calibration/ICV	W/N	r ² key was not reported
IV.	Continuing calibration	W	700 ≤ 20/0
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	A	
VIII.	Matrix spike/Matrix spike duplicates	BUF W	
IX.	Laboratory control samples	1 CRM A/W	LCs/B
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinsate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-07	31524	Sediment	04/09/14
2	SWHB-0918	31520 31535	Sediment	04/09/14
3	↓ NS	↓ NS	↓	↓
4	↓ NSD	↓ NSD	↓	↓
5	↓ DUF	↓ DUF	↓	↓
6				
7				
8				

Notes:

0-7100				

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	⊗		/	
Were all samples analyzed within the 12 hour clock criteria?			/	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) \leq 20% and relative response factors (RRF) within method criteria?			/	
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990 ?		/		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?				
Were all percent differences (%D) $< 30\%$ or percent recoveries (%R) 70-130%?				
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) $\leq 20\%$ and relative response factors (RRF) within method criteria?		/		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?	/			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	/			
X. Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds of the associated calibration standard?	/			
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	/			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?	/			
XIV. System performance				
System performance was found to be acceptable.	/			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			

VALIDATION FINDINGS WORKSHEET
Initial Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- ~~Y~~ ~~N~~ ~~N/A~~ Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?
- ~~Y~~ ~~N~~ ~~N/A~~ Were all percent relative standard deviations (%RSD) ≤ 20 and relative response factors (RRF) within method criteria?
- ~~Y~~ ~~N~~ ~~N/A~~ Was a curve fit used for evaluation?
- ~~Y~~ ~~N~~ ~~N/A~~ Did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?

#	Date	Standard ID	Compound	Finding %RSD (Limit: ≤15.0%)	Finding RRF (Limit: ≥0.05)	Associated Samples	Qualifications
	6/4/15	ICAL	PBDE 209	$\gamma^2 = 0.97105483$		all (det + ND)	Y N N/A (BC)

VALIDATION FINDINGS WORKSHEET
Continuing Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?
- Y N N/A Were percent differences (%D) $\leq 20\%$ and relative response factors (RRF) within the method criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 20.0\%$)	Finding RRF (Limit)	Associated Samples	Qualifications
	<u>6/4/15</u>	<u>CCV</u>	<u>PBDE 190</u>	<u>36.40</u>		<u>2.5 (N/D)</u>	<u>N/A (LC)</u>
		<u>(4.55)</u>	<u>209</u>	<u>24.02</u>			<u>(CH)</u>

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>3/A</u>	<u>#BDR209</u>	()	<u>44 50-150</u>	()	<u>2 (NO)</u>	<u>✓ N/A (LU)</u>
			<u>✓ 209</u>	()	()	<u>48 (≤25)</u>		<u>↓ det3/A (HD)</u>
			<u>✓ 190</u>	()	()	<u>28 (✓)</u>		<u>↓</u>
				()	()	()		
				()	()	()		
		<u>5</u>	<u>#BDR04T</u>	()	()	<u>27 (≤25)</u>	<u>2 (dets)</u>	<u>↓ det3/A (HD)</u>
				()	()	()		
				()	()	()		
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				()	()	()		

LDC #: 3197A PC

VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

Page: 1 of 1
Reviewer: 9
2nd Reviewer: JVB

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Was a LCS required?

Y N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS CRM %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		31519 CRM	PBDE 100	145 (60-140)	()	()	M (#1=dets, 2=ND)	↓ dets/A (HP)
			↓ 209	49 ()	()	()	(dets + ND)	↓ N/A (LR)
				()	()	()		
				()	()	()		
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VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PBDE (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
6/14/2015	PBDE047	1	0.0050	0.0049101	0.000025
		2	0.0125	0.0125432	0.00005
		3	0.0250	0.0256778	0.0001
		4	0.0375	0.0396473	0.0002
		5	0.0500	0.0550303	0.0004
		6	0.1000	0.1145303	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.122488	1.12249
Correlation Coefficient	0.999440	0.99739
Coefficient of Determination (r ²)	0.998880	

Method: PBDE (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
6/14/2015	PBDE099	1	0.0050	0.0062902	0.000025
		2	0.0125	0.0137423	0.00005
		3	0.0250	0.0257916	0.0001
		4	0.0375	0.0386703	0.0002
		5	0.0500	0.057046	0.0004
		6	0.1000	0.1075875	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.081339	1.08134
Correlation Coefficient	0.999562	0.99781
Coefficient of Determination (r ²)	0.999124	

VALIDATION FINDINGS WORKSHEET

Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

$$\% \text{ Difference} = 100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$$

$$\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	<u>CCV</u>	<u>6/4/15</u> <u>04:55</u>	Phenol (1st internal standard) FBDEOAT	<u>100</u>	<u>97.4399</u>	<u>97.4396</u>	<u>3</u>	<u>3</u>
			Naphthalene (2nd internal standard) <u>099</u>	<u>100</u>	<u>93.4847</u>	<u>93.4846</u>	<u>7</u>	<u>7</u>
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Surrogate Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: $SF/SS * 100$

Where: SF = Surrogate Found
 SS = Surrogate Spiked

Sample ID: 1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5 <u>OTBDE</u>	<u>100</u>	<u>83.02</u>	<u>83</u>	<u>83</u>	<u>0</u>
2-Fluorobiphenyl <u>FTBE</u>	<u>✓</u>	<u>74.96</u>	<u>75</u>	<u>75</u>	<u>0</u>
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

VALIDATION FINDINGS WORKSHEET

Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
SA = Spike added

SC = Sample concentration

RPD = |MSC - MSC| * 2 / (MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 3/4

Compound	Spike Added (uS/g)		Sample Concentration (uS/g)	Spiked Sample Concentration (uS/g)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
DBDZOT	16.55	17.49	0.13	16.14	17.22	97	97	98	98	1	1
↓ 99	↓	↓	0.13	17.4	16.87	104	104	96	96	8	8

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = $100 * (SC/SA)$

Where: SSC = Spike concentration
 SA = Spike added

RPD = $|LCSC - LCSDC| * 2 / (LCSC + LCSDC)$

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 0-7100

Compound	Spike Added (NS/A)		Spike Concentration (NS/A)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc	Reported	Recalc	Reported	Recalculated
PBDEAT	100	100	106.4	105.4	106	106	105	105	1	1
↓ 99	↓	↓	114.39	113.75	114	114	114	114	0	0

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

(X) N N/A
(X) N N/A

Were all reported results recalculated and verified for all level IV samples?

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1, RB02047

$$\text{Conc.} = \frac{(2718)(2000)(0.2103)}{(59352)(1.1248)} = 0.1719 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Pyrethroids

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories, Inc.

Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-07	31524	Sediment	04/09/14
SWHB-18	31535	Sediment	04/08/14
SWHB-18MS	31535MS	Sediment	04/08/14
SWHB-18MSD	31535MSD	Sediment	04/08/14
SWHB-18DUP	31535DUP	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Pyrethroids by Environmental Protection Agency (EPA) SW 846 Method 8270D using Negative Chemical Ionization (NCI)

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-07	All TCL compounds	398	365	J (all detects) UJ (all non-detects)	P
SWHB-18	All TCL compounds	399	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

Instrument performance check was not required per method.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination (r^2) was greater than or equal to 0.990.

Average relative response factors (RRF) for all compounds were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
05/23/15	Prallethrin cis-Permethrin	36 50	All samples in SDG 1504003-001	UJ (all non-detects) UJ (all non-detects)	A

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds.

All of the continuing calibration relative response factors (RRF) were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogates

Surrogate spikes were not required by the method.

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SWHB-18MS/MSD (SWHB-18)	Allethrin Bitenthrin Danitolcfenpropahrin Prallethrin	215 (50-150) 220 (50-150) 182 (50-150) 157 (50-150)	162 (50-150) 183 (50-150) - -	NA	-

Relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-18MS/MSD (SWHB-18)	Allethrin	28 (≤ 25)	NA	-

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits.

IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31515-BS1/BS2 (All samples in SDG 150-4003-001)	Bitenthrin	-	133 (70-130)	NA	-

Relative percent differences (RPD) were within QC limits.

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Internal Standards

All internal standard areas and retention times were within QC limits.

XII. Compound Quantitation

All compound quantitations were within validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Compound reported below the RL and above the MDL	J (all detects)	A

XIII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIV. System Performance

The system performance was acceptable.

XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, ICV %D, and results reported below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Pyrethroids - Data Qualification Summary - SDG 1504003-001**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-07 SWHB-18	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-07 SWHB-18	Prallethrin cis-Permethrin	UJ (all non-detects) UJ (all non-detects)	A	Initial calibration verification (%D) (LV)
SWHB-07 SWHB-18	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Pyrethroids - Laboratory Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Pyrethroids - Field Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A, M	
II.	GC/MS Instrument performance check	N	
III.	Initial calibration/ICV	A, M	r^2 , 1 CV \leq 70%
IV.	Continuing calibration	A	CCV \leq 20%
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	N	
VIII.	Matrix spike/Matrix spike duplicates	<i>1/3 up</i> M/A	
IX.	Laboratory control samples	M	\leq CS/b
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	SWHB-07	31524	Sediment	04/09/14
2	SWHB- 09 18	31526-31525	Sediment	04/09/14
3	MS	↓	↓	↓
4	MSD	↓	↓	↓
5	DUP	↓	↓	↓
6				
7				
8				

Notes:

0-7100				

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?			/	
Were all samples analyzed within the 12 hour clock criteria?			/	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) \leq 20% and relative response factors (RRF) within method criteria?			/	
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990 ?		/		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	/			
Were all percent differences (%D) $<$ 30% or percent recoveries (%R) 70-130%?		/		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) \leq 20% and relative response factors (RRF) within method criteria?	/			
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
X. Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds of the associated calibration standard?	/			
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?			/	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?			/	
Were chromatogram peaks verified and accounted for?	/			
XIV. System performance				
System performance was found to be acceptable.	/			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			

VALIDATION FINDINGS WORKSHEET
Initial Calibration Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Was an initial calibration verification standard analyzed after each ICAL for each instrument?
 N N/A Were all %D within the validation criteria of ≤30 %D ?

#	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	Associated Samples	Qualifications
	<u>5/23/15</u>	<u>ICV</u>	<u>prallethrin</u> <u>cis-Permethrin</u>	<u>36</u> <u>50</u>	<u>all (N/A)</u>	<u>N/A (<V)</u> <input checked="" type="checkbox"/>

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		3/4	Allethrin	215 (9-150)	162 (9-150)	()	2 (NO)	↓ N/A (HM)
			Bifenthrin	220 ()	183 ()	()		↓
			Danitol Fenpropathrin	182 ()	()	()		↓
			Prallethrin	157 ()	()	()		↓
			Allethrin	()	()	28 (≤ 25)		(HO)
				()	()	()		
				()	()	()		
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				()	()	()		

LDC #: 36177A2d

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Was a LCS required?
 Y N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>31515-BS1</u> <u>-BS2</u>	<u>Bifenthrin</u>	()	<u>133 (70-130)</u>	()	<u>ul (N/A)</u>	<u>lots/p(H4)</u>
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
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LDC: 36197A2d

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

Method: Pyrethroids (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/22/2015	Allethrin	1	0.025	0.0052169	0.000025
		2	0.050	0.0097019	0.00005
		3	0.100	0.0189906	0.0001
		4	0.250	0.0503372	0.0002
		5	0.500	0.1100291	0.0004
		6	1.000	0.2378036	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	0.232281	0.23200
Correlation Coefficient	0.998956	0.99800
Coefficient of Determination (r ²)	0.997913	

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	<u>ccv</u>	<u>5/24/15</u>	Phenol (1st internal standard) <u>Allethrin</u>	<u>500</u>	<u>599.35</u>	<u>600.109</u>	<u>20</u>	<u>20</u>
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = | MSC - MSC | * 2 / (MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 0/2

Compound	Spike Added (115/9)		Sample Concentration (115/9)	Spiked Sample Concentration (115/9)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
Allethrin	82.75	87.45	ND	177.8	141.51	215	215	162	162	28	28

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

Reviewer: 9

2nd Reviewer: JRG

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

$$\% \text{ Recovery} = 100 * (\text{SC}/\text{SA})$$

Where: SSC = Spike concentration
SA = Spike added

$$\text{RPD} = | \text{LCSC} - \text{LCSDC} | * 2 / (\text{LCSC} + \text{LCSDC})$$

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31515-18/1352

Compound	Spike Added (n/g)		Spike Concentration (n/g)		LCS		LCSD		LCS/LCSD	
					Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc	Reported	Recalc	Reported	Recalculated
Alletarin	500	500	515.45	549.4	103	103	110	110	7	7

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

**Laboratory Data Consultants, Inc.
Data Validation Report**

Project/Site Name: City of San Diego SWBH Study
LDC Report Date: May 9, 2016
Parameters: Chlorinated Pesticides
Validation Level: Level IV
Laboratory: Physis Environmental Laboratories Inc.
Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-07	31524	Sediment	04/09/14
SWHB-18	31535	Sediment	04/08/14
SWHB-18MS	31535MS	Sediment	04/08/14
SWHB-18MSD	31535MSD	Sediment	04/08/14
SWHB-18DUP	31535DUP	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Chlorinated Pesticides by Environmental Protection Agency (EPA) SW 846 Method 8270D

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-07	All TCL compounds	398	365	UJ (all non-detects)	P
SWHB-18	All TCL compounds	399	365	UJ (all non-detects)	P

II. GC Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at the required frequency.

All ion abundance requirements were met.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For compounds where average calibration factors were utilized, percent relative standard deviations (%RSD) were less than or equal to 20.0%.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
06/03/15	4,4'-DDT	0.98565806	All samples in SDG 1504003-001	UJ (all non-detects)	A

Retention time windows were established as required by the method.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds with the following exceptions:

Date	Standard	Compound	%D	Associated Samples	Flag	A or P
05/27/15	ICV	beta-BHC	484	All samples in SDG 1504003-001	UJ (all non-detects)	A

IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Standard	Compound	%D	Associated Samples	Flag	A or P
05/29/15	00CCV	beta-BHC 4,4'-DDD	27 29	SWHB-18	UJ (all non-detects) UJ (all non-detects)	A

Retention times of all compounds in the calibration standards were within the established retention time windows.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SWHB-18MS/MSD (SWHB-18)	Methoxychlor	192 (50-150)	203 (50-150)	NA	-

Relative percent differences (RPD) were within QC limits.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits.

IX. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31515-BS1/BS2 (All samples in SDG 1504003-001)	4,4'-DDT Methoxychlor	131 (70-130) 169 (70-130)	133 (70-130) 176 (70-130)	NA	-

Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits.

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Compound Quantitation

All compound quantitations met validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Compound reported below the RL and above the MDL	J (all detects)	A

XII. Target Compound Identification

All target compound identifications met validation criteria.

XIII. System Performance

The system performance was acceptable.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , ICV and continuing calibration %D, and results reported below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Chlorinated Pesticides - Data Qualification Summary - SDG 1504003-001**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-07 SWHB-18	All TCL compounds	UJ (all non-detects)	P	Technical holding time (H)
SWHB-07 SWHB-18	4,4'-DDT	UJ (all non-detects)	A	Initial calibration (r^2) (BC)
SWHB-18	beta-BHC	UJ (all non-detects)	A	Initial calibration verification (%D) (HV)
SWHB-18	beta-BHC 4,4'-DDD	UJ (all non-detects) UJ (all non-detects)	A	Continuing calibration (%D) (CH)
SWHB-07 SWHB-18	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

METHOD: GC/MS Chlorinated Pesticides (EPA SW 846 Method 8270D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / M	
II.	GC/MS Instrument performance check	A	
III.	Initial calibration/ICV	M / M	RSD ≤ 20%. Y ² ICV ≤ 30%
IV.	Continuing calibration	M	CCV ≤ 20%
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	A	
VIII.	Matrix spike/Matrix spike duplicates	1/DUP M/A	
IX.	Laboratory control samples	M	LCB/D. CRM
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	SWHB-07	31524	Sediment	04/09/14
2	SWHB-09 18	34520 31535	Sediment	04/09/14
3	↓ MS	↓ MS	↓	↓
4	↓ USD	↓ USD	↓	↓
5	↓ DUP	↓ DUP	↓	↓
6				
7				
8				

Notes:

0-7100				

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?	/		/	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% and relative response factors (RRF) within method criteria?	/			
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of ≥ 0.990?		/		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	/			
Were all percent differences (%D) < 30% or percent recoveries (%R) 70-130%?		/		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria?		/		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?	/			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<i>dup</i>
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<i>CRM</i>
Was an LCS analyzed per analytical batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
X. Field duplicates				
Were field duplicate pairs identified in this SDG?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field duplicates?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIV. System performance				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

VALIDATION FINDINGS WORKSHEET

METHOD: Pesticide/PCBs (EPA SW 846 Method 8081/8082)

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG. Chlordane
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH. Chlordane (Technical)
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II. oxy-Chlordane
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ. Mirex
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. 2,4'-DDD	KK. 4,4'-DDMU
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. 2,4'-DDE	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. 2,4'-DDT	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN.

Notes: _____

VALIDATION FINDINGS WORKSHEET
Technical Holding Times

All circled dates have exceeded the technical holding times.
Y N N/A Were all cooler temperatures within validation criteria?

METHOD: GC HPLC							
Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifier
1 (ND)	Sed	Y	4-9-14	5-12-15		398	N/A
2 ↓	↓	↓	4-8-14	↓		399	↓
3	↓	↓	↓	↓		↓	↓
4	↓	↓	↓	↓		↓	↓
5	↓	↓	↓	↓		↓	↓

TECHNICAL HOLDING TIME CRITERIA

VOLATILES: Water unpreserved: Aromatic within 7 days, non-aromatic within 14 days of sample collection.
Water preserved: Both within 14 days of sample collection.
Soils: Both within 14 days of sample collection.

EXTRACTABLES:
Water: Extracted within 7 days, analyzed within 40 days.
Soil: Extracted within 14 days, analyzed within 40 days.

VALIDATION FINDINGS WORKSHEET
Initial Calibration

METHOD: GC/MSHPLC

- N N/A Was a 5 point calibration curve performed?
- N N/A Was a linear fit used for evaluation? If yes, the acceptance criteria for each compound is %RSD less than or equal to 20.0%.
- N N/A Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? _____
- N N/A Did the initial calibration meet the acceptance criteria?
- N N/A Was initial calibration performed at the required frequency?

#	Date	Standard ID	Column / Detector	Compound	Finding RSD Limit ≤20%	Associated Samples	Qualifications
	<u>4/3/15</u>	<u>10A2</u>		<u>0</u>	<u>$r^2 = 0.98565806$</u>	<u>ML(ND)</u>	<u>1 N / A (BC)</u>

Comments _____

VALIDATION FINDINGS WORKSHEET Initial Calibration Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y ~~N~~ ~~N/A~~ Was an initial calibration verification standard analyzed after each ICAL for each instrument?

Y ~~N~~ ~~N/A~~ Were all %D within the validation criteria of ≤30 %D ?

#	Date	Standard ID	Compound	Finding %D (Limit: <30.0%)	Associated Samples	Qualifications
	5/27/15	MIXICV	B	484	all (ND)	N/A (HV)

VALIDATION FINDINGS WORKSHEET Continuing Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

(Y/N N/A) Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

(Y/N) N/A Were percent differences (%D) ≤ 20 % and relative response factors (RRF) within the method criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $<20.0\%$)	Finding RRF (Limit)	Associated Samples	Qualifications
	<u>5/29/15</u>	<u>POB100CCV</u>	<u>B</u>	<u>27</u>		<u>295 (ND)</u>	<u>N/A (CH)</u> ↓
			<u>M</u>	<u>29</u>			

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>3/A</u>	<u>P</u>	<u>192 (50-150)</u>	<u>203 (50-150)</u>	<u>()</u>	<u>2 (ND)</u>	<u>plots/A (HM)</u>
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VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Was a LCS required?

N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>31515-BS1</u>	<u>O</u>	<u>131 (70-130)</u>	<u>133 (70-130)</u>	<u>()</u>	<u>ML (ND)</u>	<u>↓ 203 P (HL)</u>
		<u>-BS2</u>	<u>P</u>	<u>169 (↓)</u>	<u>176 (↓)</u>	<u>()</u>		<u>↓</u>
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		
				<u>()</u>	<u>()</u>	<u>()</u>		

LDC#: 3619A3a

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

Method: GC/MS Pesticides (EPA SW 846 Method 8270D)

Calibration Date	System	Compound	Standard	(Y) Response	(X) Concentration
5/27/2015	Q1	4,4'-DDT	s1	0.0415327	0.050
			s2	0.1015429	0.100
			s3	0.3551243	0.250
			s4	1.0200741	0.500
			s5	2.706278	1.000

Regression Output

Reported

Constant	-0.233960	-0.232555
Std Err of Y Est		
R Squared	0.985266	0.985658
Degrees of Freedom		
X Coefficient(s)	2.839133	2.860585
Std Err of Coef.		
Correlation Coefficient	0.992606	
Coefficient of Determination (r ²)	0.985266	0.985658

LDC#: 36197A3a

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: NB

Method: GC/MS Pesticides (EPA SW 846 Method 8270D)

Calibration Date	System	Compound	Standard	(Y) Response	(X) Concentration
5/27/2015	Q1	4,4'-DDMU	s1	0.0849309	0.025
			s2	0.16469	0.050
			s3	0.348635	0.100
			s4	1.0921435	0.250
			s5	2.4835837	0.500
			s6	5.683011	1.000

Regression Output

Reported

Constant	-0.206259	-0.202600
Std Err of Y Est		
R Squared	0.995686	0.995686
Degrees of Freedom		
X Coefficient(s)	5.763401	5.763406
Std Err of Coef.		
Correlation Coefficient	0.997841	
Coefficient of Determination (r ²)	0.995686	0.995686

Method: Pesticides (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	gamma-BHC	1	0.025	0.0067298	0.000025
		2	0.050	0.0145848	0.00005
		3	0.100	0.0262238	0.0001
		4	0.250	0.0648048	0.0002
		5	0.500	0.1473573	0.0004
		6	1.000	0.2798399	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	0.281557	0.28625
Correlation Coefficient	0.999612	0.99880
Coefficient of Determination (r ²)	0.999225	

VALIDATION FINDINGS WORKSHEET

Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

$$\% \text{ Difference} = 100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$$

$$\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	<u>ccv</u>	<u>5/29/15</u>	Phenol (1st internal standard) <u>D</u>	<u>500</u>	<u>460.0172</u>	<u>460.0185</u>	<u>8</u>	<u>8</u>
			Naphthalene (2nd internal standard) <u>0</u>	<u>↓</u>	<u>413.7876</u>	<u>446.85</u>	<u>17</u>	<u>14</u>
			Fluorene (3rd internal standard) <u>44.0711</u>	<u>↓</u>	<u>467.0538</u>	<u>477.93</u>	<u>7</u>	<u>4</u>
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

Y N N/A
 Y N N/A

Were all reported results recalculated and verified for all level IV samples?

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_s)(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_s = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. All, ND:
#3 gamma-BHC

$$\text{Conc.} = \frac{(16378)(1000)(0.1655)}{(13580)(0.28625)} = 69.7 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (<u>ng/g</u>)	Calculated Concentration ()	Qualification
			<u>69.7</u>		

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = |MSC - MSC| * 2/(MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 3/4

Compound	Spike Added (US/g)		Sample Concentration (US/g)	Spiked Sample Concentration (US/g)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc	Reported	Recalc	Reported	Recalculated
HK	82.75	87.45	ND	73.58	77.14	89	89	88	88	1	1
0	↓	↓	↓	120.39	131.09	145	145	150	150	3	3

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SC/SA)

Where: SSC = Spike concentration
 SA = Spike added

RPD = |LCSC - LCSDC| * 2 / (LCSC + LCSDC)

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31515-BS1/BS2

Compound	Spike Added (113/9)		Spike Concentration (113/9)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
O	500	500	653.2	665.47	131	131	133	133	2	2
KK	✓	✓	482.95	463.46	97	97	93	93	4	4

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Surrogate Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
 SS = Surrogate Spiked

Sample ID: 1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5 <u>PCB030</u>	<u>400.0</u>	<u>282.04</u>	<u>71</u>	<u>71</u>	<u>0</u>
2-Fluorobiphenyl <u>112</u>	<u>↓</u>	<u>346.99</u>	<u>87</u>	<u>87</u>	
Terphenyl-d14 <u>198</u>	<u>↓</u>	<u>439.54</u>	<u>110</u>	<u>110</u>	<u>↓</u>
Phenol-d5 <u>TEM X</u>	<u>↓</u>	<u>271.31</u>	<u>68</u>	<u>68</u>	<u>↓</u>
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Metals

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories, Inc.

Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-14	31531	Sediment	04/08/14
SWHB-19	31536	Sediment	04/08/14
SWHB-14MS	31531	Sediment	04/08/14
SWHB-14MSD	31531	Sediment	04/08/14
SWHB-14DUP	31531	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (January 2010). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Copper, Iron, Lead, Mercury, Nickel, Selenium, Silver, Total Phosphorus, and Zinc by Environmental Protection Agency (EPA) SW 846 Method 6020/EPA Method 245.7

All sample results were subjected to Level IV evaluation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met with the following exceptions:

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
All samples in SDG 1504003-001	All analytes except Silver	408	365	J (all detects)	P
All samples in SDG 1504003-001	Silver	434	365	J (all detects)	P
All samples in SDG 1504003-001	Mercury	413	180	J (all detects)	P

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Instrument Calibration

Initial and continuing calibrations were performed as required by the methods.

The initial calibration verification (ICV) and continuing calibration verification (CCV) standards were within QC limits.

IV. ICP Interference Check Sample Analysis

The interference check sample (ICS) was not performed by the laboratory. The laboratory used a reaction chamber with mixed gases as well as internal equations to compensate for any interferences.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. For SWHB-14MS/MSD, no data were qualified for Aluminum and Lead percent recoveries (%R) outside the QC limits since the parent sample results were greater than 4X the spike concentration. Relative percent differences (RPD) were within QC limits.

VIII. Duplicate Sample Analysis

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits.

IX. Serial Dilution

Serial dilution was not performed for this SDG.

X. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the methods. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits with the following exceptions:

CRM ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
CRM-ERA 540	Aluminum Antimony Iron	167 (80-120) 166 (80-120) 136 (80-120)	All samples in SDG 1504003-001	J (all detects) J (all detects) J (all detects)	P
CRM-ERA 540	Aluminum Antimony Iron	190 (80-120) 173 (80-120) 148 (80-120)	All samples in SDG 1504003-001	J (all detects) J (all detects) J (all detects)	P

XI. Field Duplicates

No field duplicates were identified in this SDG.

XII. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits.

XIII. Sample Result Verification

All sample result verifications were acceptable.

All analytes reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Analyte reported below the RL and above the MDL	J (all detects)	A

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected in this SDG.

Due to holding time exceedance, CRM %R, and results reported below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Metals - Data Qualification Summary - SDG 1504003-001**

Sample	Analyte	Flag	A or P	Reason (Code)
SWHB-14 SWHB-19	All TCL compounds	J (all detects)	P	Technical holding time (H)
SWHB-14 SWHB-19	Aluminum Antimony Iron	J (all detects) J (all detects) J (all detects)	P	Certified reference material (HP)
SWHB-14 SWHB-19	Analyte reported below the RL and above the MDL	J (all detects)	A	Sample result verification (DL)

**City of San Diego SWBH Study
Metals - Laboratory Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Metals - Field Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

LDC #: 36197A4a

VALIDATION COMPLETENESS WORKSHEET

Date: 4/27/14

SDG #: 1504003-001

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: SP

2nd Reviewer: _____

METHOD: Metals (EPA SW 846 Method 6020/EPA Method 245.7)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	SW	4/8/14
II.	ICP/MS Tune	A	
III.	Instrument Calibration	A	
IV.	ICP Interference Check Sample (ICS) Analysis	SW	
V.	Laboratory Blanks	SW	
VI.	Field Blanks	N	
VII.	Matrix Spike/Matrix Spike Duplicates	SD	MSD = (3.4) = Al, Fe 74x
VIII.	Duplicate sample analysis	A	DUP
IX.	Serial Dilution	N	Not Performed
X.	Laboratory control samples	SW	LCS10 = CRM
XI.	Field Duplicates	N	
XII.	Internal Standard (ICP-MS)	A	
XIII.	Sample Result Verification	A	
XIV.	Overall Assessment of Data	A	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinsate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-184 SD	31533 SD	Sediment	04/08/14
2	SWHB-19	31536	Sediment	04/08/14
3	#1 MS			
4	#1 MSD			
5	#1 DUP			
6				
7				
8				
9				
10				
11				
12				

Notes: _____

Method:Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.		/		
Cooler temperature criteria was met.	/			
II. ICP/MS Tune				
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	/			
Were %RSD of isotopes in the tuning solution $\leq 5\%$?	/			
III. Calibration				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% (80-120% for mercury) QC limits?	/			
Were all initial calibration correlation coefficients > 0.995 ?	/			
IV. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?		/		
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?			/	
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.			/	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.			/	
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq 20\%$ for waters and $\leq 35\%$ for soil samples? A control limit of $\pm RL$ ($\pm 2X RL$ for soil) was used for samples that were $\leq 5X$ the RL, including when only one of the duplicate sample values were $< 5X$ the RL.			/	
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?		/		

Validation Area	Yes	No	NA	Findings/Comments
VIII. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)				
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?	/			
If the %Rs were outside the criteria, was a reanalysis performed?	/			
IX. ICP Serial Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL (ICP/MS)?			/	
Were all percent differences (%Ds) < 10%?			/	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/	
X. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XI. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XII. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		
Target analytes were detected in the field duplicates.			/	
XIII. Field blanks				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			/	

VALIDATION FINDINGS WORKSHEET
Sample Specific Element Reference

All circled elements are applicable to each sample.

Table with columns: Sample ID, Matrix, and Target Analyte List (TAL). Contains multiple rows for sample '1-2' with matrix 'sed'. The TAL list includes elements like Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Hg, Ni, K, Se, Ag, Na, Ti, V, Zn, Mo, B, Sn, Ti. Some elements (Cr, Cu, Fe, Pb, Ni, K, Se, Ag) are circled in the original image.

Analysis Method

Table listing analysis methods: ICP, ICP-MS, and GFAA, each followed by the same Target Analyte List (TAL) as the main table.

Comments: Mercury by CVAA if performed

VALIDATION FINDINGS WORKSHEET

Technical Holding Times

All circled dates have exceeded the technical holding time.

Y N N/A Were all samples preserved as applicable to each method ?

Y N N/A Were all cooler temperatures within validation criteria?

Method:		6020 SD 200.8		245.7			
Parameters:		All Analytes except Ag		Hg			
Technical holding time:		365 Days		180 Days			
Sample ID	Sampling date	Analysis date	Analysis date	Analysis date	Analysis date	Analysis date	Qualifier
All	04/08/14	05/21/15	408 Days				J/UJ/P (det) (H)
All	04/08/14			05/26/15	413 Days		J/R/P (det) (H)

Method:		6020 SD 200.8					
Parameters:		Ag					
Technical holding time:		365 Days					
Sample ID	Sampling date	Analysis date	Analysis date	Analysis date	Analysis date	Analysis date	Qualifier
All	04/08/14	06/16/15	434 Days				J/UJ/P (det) (H)

VALIDATION FINDINGS WORKSHEET
ICP Interference Check Sample

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y ~~N~~ ~~N/A~~ Were ICP interference check samples performed as required?

Y ~~N~~ ~~N/A~~ Were the AB solution percent recoveries (%R) within the control limits of 80-120% ?

LEVEL IV ONLY:

Y ~~N~~ ~~N/A~~ Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	Date	ICS Identification	Analyte	Finding	Associated Samples	Qualifications
		ICSA/AB	All except Hg	The ICP interference check sample was not performed by the laboratory. The laboratory used a reaction chamber with mixed gases as well as internal equations to compensate for any interferences.	All	Text

Comments: _____

VALIDATION FINDINGS WORKSHEET
Blanks

METHOD: Trace metals (EPA SW 846 Method 6010/7000)

#	Date	Blank ID	Analyte	Finding	Associated Samples	Qualifications
		CCB	Hg	Closing CCB was not performed	All	Text

Comments: _____

VALIDATION FINDINGS WORKSHEET

Matrix Spike/Matrix Spike Duplicates

METHOD: Trace metals (EPA SW 846 Method 6010/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Was a matrix spike analyzed for each matrix in this SDG?

Y N N/A Were matrix spike percent recoveries (%R) within the control limits of 75-125? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.

Y N N/A Were all duplicate sample relative percent differences (RPD) \leq 20% for samples?

LEVEL IV ONLY:

Y N N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
	3/4	Sed	Al			30	1	No Qual.*

Comments: *Lab calculated RPD based on %R, but %R is not meaningful for Al because Al > 4X spike. RPD of ug/g results = ok. No Qual.

3/4: Al, Fe > 4X

↑
(3% RPD)

VALIDATION FINDINGS WORKSHEET Laboratory Control Samples (LCS)

METHOD: Trace Metals (EPA SW 846 Method 6010B/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- N N/A Was a laboratory control sample (LCS) analyzed for each matrix in this SDG?
- N N/A Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?

LEVEL IV ONLY:

- N N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	LCS/LCSD ID	Matrix	Analyte	LCS %R (limits)	LCSD %R (limits)	RPD (limits)	Associated Samples	Qualifications
	CRM-ERA 540	Sed	Al	167	n/a	n/a	All	Jdet/P (det) (HL)
			Sb	166	n/a	n/a		Jdet/P (det) (HL)
			Fe	136	n/a	n/a		Jdet/P (det) (HL)
	CRM-ERA 540	Sed	Al	190	n/a	n/a	All	Jdet/P (det) (HL)
			Sb	173	n/a	n/a		Jdet/P (det) (HL)
			Fe	148	n/a	n/a		Jdet/P (det) (HL)

Comments: _____

LDC #: 36774

VALIDATION FINDINGS WORKSHEET
Initial and Continuing Calibration Calculation Verification

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

METHOD: Trace Metals (See cover)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

$\%R = \frac{\text{Found}}{\text{True}} \times 100$ Where, Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution
 True = concentration (in ug/L) of each analyte in the ICV or CCV source

Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	Recalculated	Reported	Acceptable (Y/N)
					%R	%R	
	ICP (Initial calibration)						
ICV 11:43	ICP/MS (Initial calibration)	Be	0.105 ug/L	0.1 ug/L	105%R	NR	Y
ICV 16:29	CVAA (Initial calibration)	Hg	959 ppt	1000 ppt	96%R	NR	↓
	ICP (Continuing calibration)						
CCV 14:14	ICP/MS (Continuing calibration)	Cd	0.102 ug/L	0.1 ug/L	102%R	NR	Y
CCV 19:07	CVAA (Continuing calibration)	Hg	966 ppt	1000 ppt	97%R	NR	↓
	GFAA (Initial calibration)						
	GFAA (Continuing calibration)						

Comments: _____

VALIDATION FINDINGS WORKSHEET
Level IV Recalculation Worksheet

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$
 Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).
True = Concentration of each analyte in the source.

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$
 Where, S = Original sample concentration
D = Duplicate sample concentration

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

$$\%D = \frac{|I-SDR|}{I} \times 100$$
 Where, I = Initial Sample Result (mg/L)
SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	Recalculated	Reported	Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D	
N	ICP interference check						
LCS 10:00	Laboratory control sample	Cu	2.041 ug/g	2 ug/g	102%R	102%R	Y
MS 16:10	Matrix spike	Be	(SSR-SR) 61.26 ug/g	56.306 ug/g	109%R	109%R	↓
DUP 14:50	Duplicate	As	7.452 ug/g	7.738 ug/g	4%RPD	4%RPD	↓
	ICP serial dilution						

Comments: _____

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Have results been reported and calculated correctly?
Y N N/A Are results within the calibrated range of the instruments and within the linear range of the ICP?
Y N N/A Are all detection limits below the CRDL?

Detected analyte results for (1) Hg were recalculated and verified using the following equation:

$$\text{Concentration} = \frac{(\text{RD})(\text{FV})(\text{Dil})}{(\text{In. Vol.})}$$

Recalculation:

- RD = Raw data concentration
 FV = Final volume (ml)
 In. Vol. = Initial volume (ml) or weight (G)
 Dil = Dilution factor

$$\text{RD} = 0.0994 \text{ } \mu\text{g/g}$$

#	Sample ID	Analyte	Reported Concentration ($\mu\text{g/g}$)	Calculated Concentration ($\mu\text{g/g}$)	Acceptable (Y/N)
	1	Hg	0.0994	0.0994	Y
	2	Ni	13.06	13.06	↓

Note: _____

**Laboratory Data Consultants, Inc.
Data Validation Report**

Project/Site Name: City of San Diego SWBH Study
LDC Report Date: May 9, 2016
Parameters: Wet Chemistry
Validation Level: Level IV
Laboratory: Physis Environmental Laboratories, Inc.
Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-11	31528	Sediment	04/08/14
SWHB-14	31531	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (January 2010). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Ammonia as Nitrogen by Standard Method 4500-NH₃ D
Percent Solids by Standard Method 2540B

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met with the following exceptions:

Sample	Analyte	Total Time From Sample Collection Until Analysis	Required Holding Time From Sample Collection Until Analysis	Flag	A or P
All samples in SDG 1504003-001	Percent solids	400 days	180 days	J (all detects)	P
All samples in SDG 1504003-001	Ammonia as N	401 days	180 days	J (all detects)	P

II. Initial Calibration

All criteria for the initial calibration of each method were met.

III. Continuing Calibration

Continuing calibration frequency and analysis criteria were met for each method when applicable with the following exceptions:

Date	Lab. Reference/ID	Analyte	%R (Limits)	Associated Samples	Flag	A or P
05/15/15	ICV	Ammonia as N	88 (90-110)	All samples in SDG 1504003-001	J (all detects)	A

IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

V. Field Blanks

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

The laboratory has indicated that there were no matrix spike (MS) and matrix spike duplicate (MSD) analyses specified for the samples in this SDG, and therefore matrix spike and matrix spike duplicate analyses were not performed for this SDG.

VII. Duplicate Sample Analysis

The laboratory has indicated that there were no duplicate (DUP) analyses specified for the samples in this SDG, and therefore duplicate analyses were not performed for this SDG.

VIII. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Sample Result Verification

All sample result verifications were acceptable.

All analytes reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Analyte reported below the RL and above the MDL	J (all detects)	A

XI. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected in this SDG.

Due to holding time exceedance, calibration ICV %D, and results reported below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Wet Chemistry - Data Qualification Summary - SDG 1504003-001**

Sample	Analyte	Flag	A or P	Reason (Code)
SWHB-11 SWHB-14	Percent solids Ammonia as N	J (all detects)	P	Technical holding time (H)
SWHB-11 SWHB-14	Ammonia as N	J (all detects)	A	Calibration (ICV,%D) (LC)
SWHB-11 SWHB-14	Analyte reported below the RL and above the MDL	J (all detects)	A	Sample results verification (DL)

**City of San Diego SWBH Study
Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Wet Chemistry - Field Blank Data Qualification Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

LDC #: 36197A6

VALIDATION COMPLETENESS WORKSHEET

Date: 4/21/14

SDG #: 1504003-001

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: JS

2nd Reviewer: [Signature]

METHOD: (Analyte) Ammonia-N (SM4500-NH3 D), Percent Solids (SM2540B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	SW	4/08/14
II	Initial calibration	A	
III.	Calibration verification	SW	
IV	Laboratory Blanks	A	
V	Field blanks	N	
VI.	Matrix Spike/Matrix Spike Duplicates	N	CS
VII.	Duplicate sample analysis	N	
VIII.	Laboratory control samples	A	LCS10
IX.	Field duplicates	N	
X.	Sample result verification	A	
XI	Overall assessment of data	A	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinsate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-11	31528	Sediment	04/08/14
2	SWHB-14	31531	Sediment	04/08/14
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				

Notes: _____

Method: Inorganics (EPA Method See Cover)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.		/		
Cooler temperature criteria was met.	/			
II. Calibration				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial calibration correlation coefficients > 0.995?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?		/		
Were titrant checks performed as required? (Level IV only)			/	
Were balance checks performed as required? (Level IV only)	X		/	JD (w/a)
III. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.			/	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.			/	
Were the MS/MSD or duplicate relative percent differences (RPD) ≤ 20% for waters and ≤ 35% for soil samples? A control limit of ≤ CRDL (≤ 2X CRDL for soil) was used for samples that were ≤ 5X the CRDL, including when only one of the duplicate sample values were ≤ 5X the CRDL.			/	
V. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?	/			
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			/	
Were the performance evaluation (PE) samples within the acceptance limits?			/	

VALIDATION FINDINGS CHECKLIST

Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
Were detection limits < RL?	/			
VIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		
Target analytes were detected in the field duplicates.			/	
X. Field blanks				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			/	

LDC #: 3097AC

VALIDATION FINDINGS WORKSHEET
Level IV Recalculation Worksheet

Page: 1 of 1
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

METHOD: Inorganics, Method See Cover

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

$\%R = \frac{\text{Found}}{\text{True}} \times 100$ Where, Found = concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).
 True = concentration of each analyte in the source.

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

$RPD = \frac{|S-D|}{(S+D)/2} \times 100$ Where, S = Original sample concentration
 D = Duplicate sample concentration

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated	Reported	Acceptable (Y/N)
					%R / RPD	%R / RPD	
LCS	Laboratory control sample	NH ₃ -N	4.19 mg/kg	3.93 mg/kg	107%R	107%R	Y
N	Matrix spike sample		(SSR-SR)				
N	Duplicate sample						

Comments: ICV/CCV = all recalculated from raw data, no %R summaries provided... These are level 4 Recalc not necessary

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study
LDC Report Date: May 9, 2016
Parameters: Polychlorinated Biphenyls as Congeners
Validation Level: Level IV
Laboratory: Physis Environmental Laboratories Inc.
Sample Delivery Group (SDG): 1504003-001

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-07	31524	Sediment	04/09/14
SWHB-18	315535	Sediment	04/08/14
SWHB-18MS	315535MS	Sediment	04/08/14
SWHB-18MSD	315535MSD	Sediment	04/08/14
SWHB-18DUP	315535DUP	Sediment	04/08/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Biphenyls (PCBs) as Congeners by Environmental Protection Agency (EPA) SW 846 Method 8270D

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-07	All TCL compounds	398	365	J (all detects) UJ (all non-detects)	P
SWHB-18	All TCL compounds	399	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at the required frequency.

All ion abundance requirements were met.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination (r^2) was greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
05/27/15	PCB-169	0.98733282	All samples in SDG 1504003-001	UJ (all non-detects)	A

Average relative response factors (RRF) for all compounds were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
05/28/15	PCB-003	50	All samples in SDG 1504003-001	J (all detects) UJ (all non-detects)	A
	PCB-008	49			
	PCB-018	57			
	PCB-031	46			
	PCB-028	51			
	PCB-033	39			
	PCB-052	53			
	PCB-049	48			
	PCB-044	40			
	PCB-037	39			
	PCB-074	43			
	PCB-070	58			
	PCB-066	46			
	PCB-095	55			
	PCB-056/060	47			
	PCB-101	45			
	PCB-099	41			
	PCB-097	37			
	PCB-087	33			
	PCB-081	38			
	PCB-110	43			
	PCB-077	31			
	PCB-151	50			
	PCB-123	44			
	PCB-149	62			
	PCB-118	44			
	PCB-114	46			
	PCB-153	38			
	PCB-168/132	45			
	PCB-105	34			
	PCB-141	37			
	PCB-138	38			
	PCB-158	50			
	PCB-187	42			
	PCB-183	51			
	PCB-128	41			
	PCB-167	36			
	PCB-174	54			
	PCB-177	87			
	PCB-156	62			
	PCB-157	52			
	PCB-199/200	53			
	PCB-180	48			
	PCB-169	33			
	PCB-170	43			
	PCB-201	31			
	PCB-189	36			
	PCB-195	33			
	PCB-206	39			
	PCB-209	65			

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
05/29/15	PCB-126	25	SWHB-18	UJ (all non-detects)	A
	PCB-128	21		UJ (all non-detects)	
	PCB-156	34		UJ (all non-detects)	
	PCB-169	28		UJ (all non-detects)	
	PCB-189	42		UJ (all non-detects)	
	PCB-195	23		UJ (all non-detects)	
	PCB-194	43		UJ (all non-detects)	
	PCB-206	35		UJ (all non-detects)	

All of the continuing calibration relative response factors (RRF) were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Duplicate (DUP) sample analysis was performed on an associated project sample. Relative percent differences (RPD) were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-18DUP (SWHB-18)	PCB-031	72 (≤ 25)	J (all detects)	A
	PCB-028	89 (≤ 25)	UJ (all non-detects)	
	PCB-101	95 (≤ 25)		
	PCB-110	89 (≤ 25)		
	PCB-153	53 (≤ 25)		
	PCB-157	109 (≤ 25)		
	PCB-158	117 (≤ 25)		

VIII. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Internal Standards

All internal standard areas and retention times were within QC limits.

XI. Compound Quantitation

All compound quantitations were within validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-001	Compound reported below the RL and above the MDL	J (all detects)	A

XII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIII. System Performance

The system performance was acceptable.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , ICV and continuing calibration %D, DUP RPD, and results below the RL and above the MDL, data were qualified as estimated in two samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
 Polychlorinated Biphenyls as Congeners - Data Qualification Summary - SDG
 1504003-001**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-07 SWHB-18	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-07 SWHB-18	PCB-169	UJ (all non-detects)	A	Initial calibration (r ²) (BC)
SWHB-07 SWHB-18	PCB-003 PCB-008 PCB-018 PCB-031 PCB-028 PCB-033 PCB-052 PCB-049 PCB-044 PCB-037 PCB-074 PCB-070 PCB-066 PCB-095 PCB-056/060 PCB-101 PCB-099 PCB-097 PCB-087 PCB-081 PCB-110 PCB-077 PCB-151 PCB-123 PCB-149 PCB-118 PCB-114 PCB-153 PCB-168/132 PCB-105 PCB-141 PCB-138 PCB-158 PCB-187 PCB-183 PCB-128 PCB-167 PCB-174 PCB-177 PCB-156 PCB-157 PCB-199/200 PCB-180 PCB-169 PCB-170 PCB-201 PCB-189 PCB-195 PCB-206 PCB-209	J (all detects) UJ (all non-detects)	A	Initial calibration verification (%D) (HV)

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-18	PCB-126 PCB-128 PCB-156 PCB-169 PCB-189 PCB-195 PCB-194 PCB-206	UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects) UJ (all non-detects)	A	Continuing calibration (%D) (CH)
SWHB-18	PCB-031 PCB-028 PCB-101 PCB-110 PCB-153 PCB-157 PCB-158	J (all detects) UJ (all non-detects)	A	Duplicate sample analysis (RPD) (HD)
SWHB-07 SWHB-18	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Polychlorinated Biphenyls as Congeners - Laboratory Blank Data Qualification
Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Polychlorinated Biphenyls as Congeners - Field Blank Data Qualification
Summary - SDG 1504003-001**

No Sample Data Qualified in this SDG

METHOD: GC/MS Polychlorinated Biphenyls as Congeners (EPA SW 846 Method 8270D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / M	
II.	GC/MS Instrument performance check	A	
III.	Initial calibration/ICV	M / M	r^2 ICV $\leq 35/0$
IV.	Continuing calibration	M	CCV $\leq 20/0$
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	N	
VIII.	Matrix spike/Matrix spike duplicates	1 DUP A / M	
IX.	Laboratory control samples	1 CRM A	LCS/D. CRM
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	SWHB-07	31524	Sediment	04/09/14
2	SWHB-09/18	31526 31535	Sediment	04/09/14
3	↓ MS	↓ MS	↓	↓
4	↓ MSD	↓ MSD	↓	↓
5	↓ DUP	↓ DUP	↓	↓
6				
7				
8				

Notes:

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		<input checked="" type="checkbox"/>		
Was cooler temperature criteria met?	<input checked="" type="checkbox"/>			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	<input checked="" type="checkbox"/>			
Were all samples analyzed within the 12 hour clock criteria?			<input checked="" type="checkbox"/>	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>			
Were all percent relative standard deviations (%RSD) \leq 20% and relative response factors (RRF) within method criteria?			<input checked="" type="checkbox"/>	
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990 ?		<input checked="" type="checkbox"/>		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) \leq 30% or percent recoveries (%R) 70-130%?		<input checked="" type="checkbox"/>		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) \leq 20% and relative response factors (RRF) within method criteria?		<input checked="" type="checkbox"/>		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	<input checked="" type="checkbox"/>			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		<input checked="" type="checkbox"/>		
VI. Field blanks				
Were field blanks were identified in this SDG?		<input checked="" type="checkbox"/>		
Were target compounds detected in the field blanks?			<input checked="" type="checkbox"/>	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?			<input checked="" type="checkbox"/>	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			<input checked="" type="checkbox"/>	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			<input checked="" type="checkbox"/>	

VALIDATION FINDINGS CHECKLIST

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Was an LCS analyzed per analytical batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
X. Field duplicates				
Were field duplicate pairs identified in this SDG?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field duplicates?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIV. System performance				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

VALIDATION FINDINGS WORKSHEET Technical Holding Times

All circled dates have exceeded the technical holding times.
 N N/A Were all cooler temperatures within validation criteria?

METHOD : GC/MS BNA (EPA SW 846 Method 8270D)							
Sample ID	Matrix	Preserved	Sampling Date	<u>Extraction date</u>	Analysis date	Total # of Days	Qualifier
1	sed	Y	4-9-14	5-12-15		398	✓N/A/CH
2	↓	↓	4-8-14	↓		399	↓
3	↓	↓	↓	↓		↓	↓
4	↓	↓	↓	↓		↓	↓
5	↓	↓	↓	↓		↓	↓
(dots + ND)							

TECHNICAL HOLDING TIME CRITERIA
Water: Extracted within 7 days, analyzed within 40 days.
Soil: Extracted within 14 days, analyzed within 40 days.

1504003-001

Southern California Coastal Water Research Project - San Diego Bay SWHB

ICV Summary- PCB Congeners

O-7100

	Date Analyzed	Time Analyzed	Result (ng)	True Value (ng)	Percent Drift
PCB003	5/28/2015	2:46	150.0695	100	-50
PCB008	5/28/2015	2:46	148.7926	100	-49
PCB018	5/28/2015	2:46	156.7789	100	-57
PCB031	5/28/2015	2:46	146.0087	100	-46
PCB028	5/28/2015	2:46	151.2177	100	-51
PCB033	5/28/2015	2:46	138.8935	100	-39
PCB052	5/28/2015	2:46	152.6209	100	-53
PCB049	5/28/2015	2:46	147.9337	100	-48
PCB044	5/28/2015	2:46	140.3380	100	-40
PCB037	5/28/2015	2:46	139.1922	100	-39
PCB074	5/28/2015	2:46	143.0172	100	-43
PCB070	5/28/2015	2:46	158.2131	100	-58
PCB066	5/28/2015	2:46	146.2953	100	-46
PCB095	5/28/2015	2:46	155.2301	100	-55
PCB056(060)	5/28/2015	2:46	146.8170	100	-47
PCB101	5/28/2015	2:46	144.9960	100	-45
PCB099	5/28/2015	2:46	140.9026	100	-41
PCB119	5/28/2015	2:46	126.6638	100	-27
PCB097	5/28/2015	2:46	137.1601	100	-37
PCB087	5/28/2015	2:46	133.2028	100	-33
PCB081	5/28/2015	2:46	138.4812	100	-38
PCB110	5/28/2015	2:46	142.8046	100	-43
PCB077	5/28/2015	2:46	131.4960	100	-31
PCB151	5/28/2015	2:46	149.6005	100	-50
PCB123	5/28/2015	2:46	143.8889	100	-44
PCB149	5/28/2015	2:46	162.1329	100	-62
PCB118	5/28/2015	2:46	144.3602	100	-44
PCB114	5/28/2015	2:46	146.0619	100	-46
PCB153	5/28/2015	2:46	137.9393	100	-38
PCB168+132	5/28/2015	2:46	290.6100	200	-45
PCB105	5/28/2015	2:46	134.3373	100	-34
PCB141	5/28/2015	2:46	137.1614	100	-37
PCB138	5/28/2015	2:46	138.2780	100	-38
PCB158	5/28/2015	2:46	149.5370	100	-50
PCB126	5/28/2015	2:46	129.2916	100	-29
PCB187	5/28/2015	2:46	141.7368	100	-42
PCB183	5/28/2015	2:46	151.0057	100	-51
PCB128	5/28/2015	2:46	141.3365	100	-41
PCB167	5/28/2015	2:46	135.7332	100	-36
PCB174	5/28/2015	2:46	153.5098	100	-54

1/UL/A

1/UL/A

1/UL/A

PCB177	5/28/2015	2:46	187.4373	100	-87
PCB156	5/28/2015	2:46	161.9775	100	-62
PCB157	5/28/2015	2:46	151.8754	100	-52
PCB199(200)	5/28/2015	2:46	152.6292	100	-53
PCB180	5/28/2015	2:46	148.3838	100	-48
PCB169	5/28/2015	2:46	132.5443	100	-33
PCB170	5/28/2015	2:46	142.6102	100	-43
PCB201	5/28/2015	2:46	131.4340	100	-31
PCB189	5/28/2015	2:46	135.9974	100	-36
PCB195	5/28/2015	2:46	132.5293	100	-33
PCB194	5/28/2015	2:46	130.3517	100	-30
PCB206	5/28/2015	2:46	139.1991	100	-39
PCB209	5/28/2015	2:46	164.6459	100	-65

1/11/A
↓
1/11/A
↓

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>5</u>	<u>PCB031</u>	()	()	<u>72 (<75)</u>	<u>2 (dets)</u>	<u>↓/N/A (HD)</u>
			<u>028</u>	()	()	<u>89</u>	↓	
			<u>101</u>	()	()	<u>95</u>	(ND)	
			<u>110</u>	()	()	<u>89</u>	↓	
			<u>153</u>	()	()	<u>53</u>	(dets)	
			<u>157</u>	()	()	<u>109</u>	(ND)	
			<u>158</u>	()	()	<u>117</u>	(dets)	
				()	()	()		
				()	()	()		
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VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PCB congeners (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	PCB031	1	0.0100	0.0081442	0.00010
		2	0.0250	0.0192735	0.00063
		3	0.0500	0.0422875	0.00250
		4	0.0750	0.0633359	0.00563
		5	0.1000	0.0922189	0.01000
		6	0.2000	0.1926116	0.04000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	0.937503	0.93750
Correlation Coefficient	0.998870	0.99485
Coefficient of Determination (r ²)	0.997742	

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PCB congeners (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	PCB189	1	0.0100	0.0189185	0.00010
		2	0.0250	0.0407605	0.00063
		3	0.0500	0.0848498	0.00250
		4	0.0750	0.1175805	0.00563
		5	0.1000	0.15908	0.01000
		6	0.2000	0.3488099	0.04000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.698202	1.69821
Correlation Coefficient	0.999112	0.99576
Coefficient of Determination (r ²)	0.998225	

VALIDATION FINDINGS WORKSHEET
Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
 RRF = (A_x)(C_{is})/(A_{is})(C_x)

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	<u>CEV</u>	<u>5/29/15</u>	Phenol (1st internal standard) <u>PCB 031</u>	<u>100</u>	<u>106.1240</u>	<u>106.1239</u>	<u>6</u>	<u>6</u>
			Naphthalene (2nd internal standard) <u>189</u>	<u>↓</u>	<u>142.3173</u>	<u>142.3166</u>	<u>42</u>	<u>42</u>
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = |MSC - MSC| * 2/(MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 3/4

Compound	Spike Added (NS/A)		Sample Concentration (NS/A)	Spiked Sample Concentration (NS/A)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc	Reported	Recalc	Reported	Recalculated
PCB031	16.55	17.49	0.15	16.65	17.2	99	100	97	97	2	2
✓ 189	↓	↓	ND	22.58	24.14	136	136	138	138	1	1

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = $100 * (SC/SA)$

Where: SSC = Spike concentration
 SA = Spike added

RPD = $| LCSC - LCSDC | * 2 / (LCSC + LCSDC)$

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31515-BS1 / BS2

Compound	Spike Added (n=9)		Spike Concentration (n=9)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc	Reported	Recalc	Reported	Recalculated
PCB031	100	100	114.16	98.87	114	114	99	99	14	14
↓ 189	↓	↓	12.74	125.25	122	122	125	125	2	2

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 3619TAB1

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

Page: 1 of 1
 Reviewer: Q
 2nd reviewer: JLG

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

Y N N/A
 Y N N/A

Were all reported results recalculated and verified for all level IV samples?
 Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_i)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1, PCB03!

$$\text{Conc.} = \frac{(1813)(1000)(0.2103)}{(234165)(0.998)} = 0.33 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (ng/g)	Calculated Concentration ()	Qualification
			0.33		

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Polynuclear Aromatic Hydrocarbons

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories, Inc.

Sample Delivery Group (SDG): 1504003-002

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-26-M	31963	Tissue	04/22/14
SWHB-27-SBB	31966	Tissue	04/23/14
SWHB-27-P	31969	Tissue	04/23/14
SWHB-30-CH	31973	Tissue	04/23/14
SWHB-06-CH-Small	31989	Tissue	04/22/14
SWHB-06-M	31992	Tissue	04/22/14
SWHB-22-SP	32017	Tissue	04/21/14
SWHB-27-SBBMS	31966MS	Tissue	04/23/14
SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
SWHB-27-SBBDUP	31966DUP	Tissue	04/23/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polynuclear Aromatic Hydrocarbons (PAHs) by Environmental Protection Agency (EPA) SW 846 Method 8270D

All sample results were subjected to Level IV evaluation, which is comprised of the QC summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exception:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-26-M SWHB-06-CH-Small SWHB-06-M	All TCL compounds	402	365	J (all detects) UJ (all non-detects)	P
SWHB-27-SBB SWHB-27-P SWHB-30-CH	All TCL compounds	401	365	J (all detects) UJ (all non-detects)	P
SWHB-22-SP	All TCL compounds	403	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at 12 hour intervals.

All ion abundance requirements were met.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For compounds where average relative response factors (RRFs) were utilized, percent relative standard deviations (%RSD) were less than or equal to 20.0%.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990.

Average relative response factors (RRF) for all compounds were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
06/24/15	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(a)pyrene	45.6 42.5 45.2 55.8	SWHB-27-P	J (all detects) J (all detects) J (all detects) J (all detects)	A

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
06/13/15	Benzo(a)anthracene	34	SWHB-30-CH	J (all detects)	A
	Chrysene	34	SWHB-06-CH-Small	UJ (all non-detects)	
	Benzo(b)fluoranthene	54	SWHB-06-M		
	Benzo(k)fluoranthene	53	SWHB-22-SP		
	Benzo(e)pyrene	59			
	Benzo(a)pyrene	56			
	Perylene	58			
06/25/15	Benzo(a)pyrene	21.4	SWHB-27-P	J (all detects)	A
	Benzo(b)fluoranthene	20.9		J (all detects)	
	Benzo(e)pyrene	21.5		J (all detects)	

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogate Spikes

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-27-SBBDUP (SWHB-27-SBB)	1-Methylnaphthalene 2,6-Dimethylnaphthalene	80 (≤25) 34 (≤25)	J (all detects) J (all detects)	A

IX. Laboratory Control Samples

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31945-BS1/BS2 (All samples in SDG 1504003-002)	1-Methylnaphthalene	53 (70-130)	51 (70-130)	J (all detects) UJ (all non-detects)	P
	2,3,5-Trimethylnaphthalene	-	59 (70-130)		
	2,6-Dimethylnaphthalene	-	55 (70-130)		
	2-Methylnaphthalene	-	52 (70-130)		
	Acenaphthene	-	56 (70-130)		
	Acenaphthylene	-	56 (70-130)		
	Anthracene	-	66 (70-130)		
	Biphenyl	-	55 (70-130)		
	Dibenzothiophene	-	61 (70-130)		
	Fluorene	-	59 (70-130)		
	Naphthalene	-	50 (70-130)		
	Phenanthrene	-	66 (70-130)		

Relative percent differences (RPD) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
31945-BS1/BS2 (All samples in SDG 1504003-002)	1-Methylphenanthrene	33 (≤ 25)	J (all detects) UJ (all non-detects)	P
	2,3,5-Trimethylnaphthalene	45 (≤ 25)		
	2,6-Dimethylnaphthalene	49 (≤ 25)		
	2-Methylnaphthalene	51 (≤ 25)		
	Acenaphthene	51 (≤ 25)		
	Acenaphthylene	48 (≤ 25)		
	Anthracene	38 (≤ 25)		
	Biphenyl	49 (≤ 25)		
	Dibenzothiophene	46 (≤ 25)		
	Fluoranthene	30 (≤ 25)		
	Fluorene	47 (≤ 25)		
	Naphthalene	56 (≤ 25)		
	Phenanthrene	39 (≤ 25)		
	Pyrene	29 (≤ 25)		

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Internal Standards

All internal standard areas and retention times were within QC limits.

XII. Compound Quantitation

All compound quantitations were within validation.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-002	Compound reported below the RL and above the MDL	J (all detects)	A

XIII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIV. System Performance

The system performance was acceptable.

XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, ICV and continuing calibration %D, DUP RPD, LCS/LCSD %R and RPD, and results reported below the RL and above the MDL, data were qualified as estimated in seven samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
 Polynuclear Aromatic Hydrocarbons - Data Qualification Summary - SDG
 1504003-002**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-26-M SWHB-06-CH-Small SWHB-06-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-22-SP	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-27-P	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(a)pyrene	J (all detects) J (all detects) J (all detects) J (all detects)	A	Initial calibration verification (%D) (HV)
SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	Benzo(a)anthracene Chrysene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(e)pyrene Benzo(a)pyrene Perylene	J (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (LC)
SWHB-27-P	Benzo(a)pyrene Benzo(b)fluoranthene Benzo(e)pyrene	J (all detects) J (all detects) J (all detects)	A	Continuing calibration (%D) (LC)
SWHB-27-SBB	1-Methylnaphthalene 2,6-Dimethylnaphthalene	J (all detects) J (all detects)	A	Duplicate sample analysis (RPD) (HD)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	1-Methylnaphthalene 2,3,5-Trimethylnaphthalene 2,6-Dimethylnaphthalene 2-Methylnaphthalene Acenaphthene Acenaphthylene Anthracene Biphenyl Dibenzothiophene Fluorene Naphthalene Phenanthrene	J (all detects) UJ (all non-detects)	P	Laboratory control samples (%R) (LL)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	1-Methylphenanthrene 2,3,5-Trimethylnaphthalene 2,6-Dimethylnaphthalene 2-Methylnaphthalene Acenaphthene Acenaphthylene Anthracene Biphenyl Dibenzothiophene Fluoranthene Fluorene Naphthalene Phenanthrene Pyrene	J (all detects) UJ (all non-detects)	P	Laboratory control samples (RPD) (HD)

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Polynuclear Aromatic Hydrocarbons - Laboratory Blank Data Qualification
Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Polynuclear Aromatic Hydrocarbons - Field Blank Data Qualification Summary -
SDG 1504003-002**

No Sample Data Qualified in this SDG

LDC #: 36197B2b

VALIDATION COMPLETENESS WORKSHEET

Date: 5/1/14

SDG #: 1504003-002

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: GC/MS Polynuclear Aromatic Hydrocarbons (EPA SW 846 Method 8270D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A/W	
II.	GC/MS Instrument performance check	A	
III.	Initial calibration/ICV	A/A	RSDS ≤ 20% χ^2 ICV ≤ 30%
IV.	Continuing calibration	W	CCV ≤ 20%
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	A	
VIII.	Matrix spike/Matrix spike duplicates /Dup	A/W	
IX.	Laboratory control samples	W	100% ✓
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable
N = Not provided/applicable
SW = See worksheet

ND = No compounds detected
R = Rinsate
FB = Field blank

D = Duplicate
TB = Trip blank
EB = Equipment blank

SB=Source blank
OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-26-M	31963	Tissue	04/22/14
2	SWHB-27-SBB	31966	Tissue	04/23/14
3	SWHB-27-P	31969	Tissue	04/23/14
4	SWHB-30-CH ✓	31973	Tissue	04/23/14
5	SWHB-06-CH-Small	31989	Tissue	04/22/14
6	SWHB-06-M	31992	Tissue	04/22/14
7	SWHB-22-SP	32017	Tissue	04/21/14
8	SWHB-27-SBBMS	31966MS	Tissue	04/23/14
9	SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
10	↓ Dup	↓ Dup	✓	✓
11				
12				
13				

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?			/	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% and relative response factors (RRF) within method criteria?	/			
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990?	/			
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	/			
Were all percent differences (%D) ≤ 30% or percent recoveries (%R) 70-130%?	/			
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria?		/		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?	/			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			dup
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
X. Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds of the associated calibration standard?	/			
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	/			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?	/			
XIV. System performance				
System performance was found to be acceptable.	/			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			

VALIDATION FINDINGS WORKSHEET

METHOD: GC/MS SVOA

A. Phenol	T. 4-Chloroaniline	MM. 4-Chlorophenyl-phenyl ether	FFF. Di-n-octylphthalate	YYY. 2,3,5-Trimethylnaphthalene
B. Bis (2-chloroethyl) ether	U. Hexachlorobutadiene	NN. Fluorene	GGG. Benzo(b)fluoranthene	ZZZ. Perylene
C. 2-Chlorophenol	V. 4-Chloro-3-methylphenol	OO. 4-Nitroaniline	HHH. Benzo(k)fluoranthene	AAAA. Dibenzothiophene
D. 1,3-Dichlorobenzene	W. 2-Methylnaphthalene	PP. 4,6-Dinitro-2-methylphenol	III. Benzo(a)pyrene	BBBB. Benzo(a)fluoranthene
E. 1,4-Dichlorobenzene	X. Hexachlorocyclopentadiene	QQ. N-Nitrosodiphenylamine	JJJ. Indeno(1,2,3-cd)pyrene	CCCC. Benzo(b)fluorene
F. 1,2-Dichlorobenzene	Y. 2,4,6-Trichlorophenol	RR. 4-Bromophenyl-phenylether	KKK. Dibenz(a,h)anthracene	DDDD. cis/trans-Decalin
G. 2-Methylphenol	Z. 2,4,5-Trichlorophenol	SS. Hexachlorobenzene	LLL. Benzo(g,h,i)perylene	EEEE. Biphenyl
H. 2,2'-Oxybis(1-chloropropane)	AA. 2-Chloronaphthalene	TT. Pentachlorophenol	MMM. Bis(2-Chloroisopropyl)ether	FFFF. Retene
I. 4-Methylphenol	BB. 2-Nitroaniline	UU. Phenanthrene	NNN. Aniline	GGGG. C30-Hopane
J. N-Nitroso-di-n-propylamine	CC. Dimethylphthalate	VV. Anthracene	OOO. N-Nitrosodimethylamine	HHHH. 1-Methylphenanthrene
K. Hexachloroethane	DD. Acenaphthylene	WW. Carbazole	PPP. Benzoic Acid	IIII. 1,4-Dioxane
L. Nitrobenzene	EE. 2,6-Dinitrotoluene	XX. Di-n-butylphthalate	QQQ. Benzyl alcohol	JJJJ. Acetophenone
M. Isophorone	FF. 3-Nitroaniline	YY. Fluoranthene	RRR. Pyridine	KKKK. Atrazine
N. 2-Nitrophenol	GG. Acenaphthene	ZZ. Pyrene	SSS. Benzidine	LLLL. Benzaldehyde
O. 2,4-Dimethylphenol	HH. 2,4-Dinitrophenol	AAA. Butylbenzylphthalate	TTT. 1-Methylnaphthalene	MMMM. Caprolactam
P. Bis(2-chloroethoxy)methane	II. 4-Nitrophenol	BBB. 3,3'-Dichlorobenzidine	UUU. Benzo(b)thiophene	NNNN.
Q. 2,4-Dichlorophenol	JJ. Dibenzofuran	CCC. Benzo(a)anthracene	VVV. Benzonaphthothiophene	OOOO.
R. 1,2,4-Trichlorobenzene	KK. 2,4-Dinitrotoluene	DDD. Chrysene	WWW. Benzo(e)pyrene	PPPP.
S. Naphthalene	LL. Diethylphthalate	EEE. Bis(2-ethylhexyl)phthalate	XXX. 2,6-Dimethylnaphthalene	QQQQ.

LDC #: 26197B-10

VALIDATION FINDINGS WORKSHEET
Initial Calibration Verification

Page: 1 of 1
Reviewer: JVGQ
2nd Reviewer: JK

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- ~~N~~ N/A Was an initial calibration verification standard analyzed after each ICAL for each instrument?
 ~~N~~ N/A Were all %D within the validation criteria of ≤ 30 %D?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 30.0\%$)	Associated Samples	Qualifications
	<u>6/4/15</u>	<u>ICV</u>	GGG	<u>45.6</u>	<u>3 (dots)</u>	<u>✓ (HVA) (HV)</u>
			<u>HHH</u>	<u>42.5</u>		↓
			<u>WWW</u>	<u>45.2</u>		
			<u>III</u>	<u>55.8</u>		

VALIDATION FINDINGS WORKSHEET
Continuing Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

Y (N N/A) Were percent differences (%D) $\leq 20\%$ and relative response factors (RRF) within the method criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 20.0\%$)	Finding RRF (Limit)	Associated Samples	Qualifications
	6/13/15	CCV	CCC	34		4-7 (dets+ND)	↓ N/A (LC) ↓
			DDD	34			
			GGG	54			
			HHH	53			
			WWW	59			
			LLL	56			
			ZZZ	58			
	6/5/15	CCV	III	21.4		3 (dets)	↓ N/A (LC) ↓
			GGG	20.9			
			WWW	21.5			

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		10	TTT	()	()	80 (<75)	2 (dets)	dets/A (HD)
			XXX	()	()	34 (✓)		✓
				()	()	()		
				()	()	()		
				()	()	()		
				()	()	()		
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VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Was a LCS required?
Y N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>31945-BS1</u>	<u>TTT</u>	<u>53 (70-130)</u>	<u>51 (70-130)</u>	()	<u>ML (det/ND)</u>	<u>J/W/PILL</u>
		<u>BS2</u>	<u>YYY</u>	()	<u>59</u> ()	()		
			<u>XXX</u>	()	<u>55</u> ()	()		
			<u>W</u>	()	<u>52</u> ()	()		
			<u>GF</u>	()	<u>56</u> ()	()		
			<u>DD</u>	()	<u>56</u> ()	()		
			<u>VV</u>	()	<u>66</u> ()	()		
			<u>EEEE</u>	()	<u>55</u> ()	()		
			<u>AAAA</u>	()	<u>61</u> ()	()		
			<u>NN</u>	()	<u>59</u> ()	()		
			<u>S</u>	()	<u>50</u> ()	()		
			<u>UU</u>	()	<u>66</u> ()	()		
			<u>HHHH</u>	()	()	<u>33 (≤25)</u>		<u>(HD)</u>
			<u>YYY</u>	()	()	<u>45</u> ()		
			<u>XXX</u>	()	()	<u>49</u> ()		
			<u>W</u>	()	()	<u>51</u> ()		
			<u>GF</u>	()	()	<u>51</u> ()		
			<u>DD</u>	()	()	<u>48</u> ()		
			<u>VV</u>	()	()	<u>38</u> ()		
			<u>EEEE</u>	()	()	<u>49</u> ()		
			<u>AAAA</u>	()	()	<u>46</u>		
			<u>YY</u>	()	()	<u>30</u> ()		
			<u>NN</u>	()	()	<u>47</u> ()		
			<u>S</u>	()	()	<u>56</u> ()		
			<u>UU</u>	()	()	<u>39</u> ()		
			<u>22</u>			<u>29</u>		

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PAHs (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
6/11/2015	Naphthalene	1	0.0125	0.0133099	0.0001562
		2	0.0250	0.0259891	0.0006250
		3	0.0500	0.052762	0.0025000
		4	0.1250	0.1246596	0.0156250
		5	0.2500	0.2651601	0.0625000
		6	0.5000	0.5106450	0.2500000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.027890	1.02255
Correlation Coefficient	0.999865	0.99930
Coefficient of Determination (r ²)	0.999730	

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PAHs (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
6/11/2015	Benzo(g,h,i)perylene	1	0.0125	0.0207516	0.0001562
		2	0.0250	0.0357094	0.0006250
		3	0.0500	0.0685033	0.0025000
		4	0.1250	0.167498	0.0156250
		5	0.2500	0.3311191	0.0625000
		6	0.5000	0.6637250	0.2500000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.328149	1.32858
Correlation Coefficient	0.999973	0.99987
Coefficient of Determination (r ²)	0.999947	

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PAHs (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
6/24/2015	Benzo(a)pyrene	1	0.0125	0.00669168	0.0001562
		2	0.0250	0.0135001	0.0006250
		3	0.0500	0.028699463	0.0025000
		4	0.1250	0.07367835	0.0156250
		5	0.2500	0.162364147	0.0625000
		6	0.5000	0.3055086	0.2500000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	0.616800	0.61680
Correlation Coefficient	0.999619	0.99859
Coefficient of Determination (r ²)	0.999239	

VALIDATION FINDINGS WORKSHEET
Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
 RRF = (A_x)(C_{is})/(A_{is})(C_x)

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	CCV	6/13/15	Phenol (1st internal standard) S	1000	1097.42/15		10	
			Naphthalene (2nd internal standard) LL	500	503.3481		1	
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2	CCV	6/13/15	Phenol (1st internal standard) S	500	546.0908	546.0878	9	9
			Naphthalene (2nd internal standard) LL	V	503.3481	503.347	1	1
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3	CCV	6/25/15	Phenol (1st internal standard) III	500	393.15	393.15	21.4	21.4
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Surrogate Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
 SS = Surrogate Spiked

Sample ID: 1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5 <u>d10-44</u>	<u>1000</u>	<u>985.24</u>	<u>99</u>	<u>99</u>	<u>0</u>
2-Fluorobiphenyl <u>d10-UU</u>	↓	<u>978.11</u>	<u>98</u>	<u>98</u>	↓
Terphenyl-d14 <u>d12-BDD</u>	↓	<u>740.67</u>	<u>74</u>	<u>74</u>	↓
Phenol-d5 <u>d8-S</u>	↓	<u>847.33</u>	<u>85</u>	<u>85</u>	↓
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = |MSC - MSC| * 2 / (MSC + MSC)

MSC = Matrix spike concentration

MSC = Matrix spike duplicate concentration

MS/MSD samples: 8/9

Compound	Spike Added (113/9)		Sample Concentration (113/9)	Spiked Sample Concentration (113/9)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
LLL	200.2	204.1	ND	192.4	202.6	96	96	99	99	3	3
S	✓	✓	10.8	180	175.9	85	85	81	81	5	5

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SC/SA)

Where: SSC = Spike concentration
 SA = Spike added

RPD = | LCSC - LCSDC | * 2 / (LCSC + LCSDC)

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31945-BS1/-BS2

Compound	Spike Added (178/9)		Spike Concentration (178/9)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc	Reported	Recalc	Reported	Recalculated
S	500	500	445.8	248.9	89	89	50	50	56	56
HL	✓	✓	493.3	485	99	99	97	97	2	2

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

- Y N N/A Were all reported results recalculated and verified for all level IV samples?
 Y N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- $\%S$ = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1, 5:

$$\text{Conc.} = \frac{(17593)(2000)(1.6033)}{(287063)(1.0255)} = 19.49 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (ng/g)	Calculated Concentration ()	Qualification
		<u>S</u>	<u>19.5</u>		

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Polybrominated Diphenyl Ethers

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories, Inc.

Sample Delivery Group (SDG): 1504003-002

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-26-M	31963	Tissue	04/22/14
SWHB-27-SBB	31966	Tissue	04/23/14
SWHB-27-P	31969	Tissue	04/23/14
SWHB-30-CH	31973	Tissue	04/23/14
SWHB-06-CH-Small	31989	Tissue	04/22/14
SWHB-06-M	31992	Tissue	04/22/14
SWHB-22-SP	32017	Tissue	04/21/14
SWHB-27-SBBMS	31966MS	Tissue	04/23/14
SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
SWHB-27-SBBDUP	31966DUP	Tissue	04/23/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polybrominated Diphenyl Ethers (PBDE) by Environmental Protection Agency (EPA) SW 846 Method 8270D using Negative Chemical Ionization (NCI)

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-26-M SWHB-06-CH-Small SWHB-06-M	All TCL compounds	402	365	J (all detects) UJ (all non-detects)	P
SWHB-27-SBB SWHB-27-P SWHB-30-CH	All TCL compounds	401	365	J (all detects) UJ (all non-detects)	P
SWHB-22-SP	All TCL compounds	403	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

Instrument performance check was not required per method.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination (r^2) was greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
06/17/15	PBDE 209	0.98570797	All samples in SDG 1504003-002	UJ (all non-detects)	A

Average relative response factors (RRF) for all compounds were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
06/18/15	PBDE 100 PBDE 099 PBDE 085 PBDE 154 PBDE 153 PBDE 138 PBDE 183 PBDE 190 PBDE 209	22 22 28 29 37 38 39 49 55	SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	J (all detects) UJ (all non-detects)	A

All of the continuing calibration relative response factors (RRF) were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-27-SBBDUP (SWHB-27-SBB)	PBDE 047 PBDE 100 PBDE 153	30 (≤25) 54 (≤25) 33 (≤25)	J (all detects) J (all detects) J (all detects)	A

IX. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31945-BS1/BS2 (All samples in SDG 1504003-002)	PBDE 017	59 (70-130)	62 (70-130)	UJ (all non-detects)	P

Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits.

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Internal Standards

All internal standard areas and retention times were within QC limits.

XII. Compound Quantitation

All compound quantitations were within validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-002	Compound reported below the RL and above the MDL	J (all detects)	A

XIII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIV. System Performance

The system performance was acceptable.

XV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , continuing calibration %D, DUP RPD, LCS/LCSD %R, and results reported below the RL and above the MDL, data were qualified as estimated in seven samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Polybrominated Diphenyl Ethers - Data Qualification Summary - SDG 1504003-002**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-26-M SWHB-06-CH-Small SWHB-06-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-22-SP	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PBDE 209	UJ (all non-detects)	A	Initial calibration (r^2) (BC)
SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PBDE 100 PBDE 099 PBDE 085 PBDE 154 PBDE 153 PBDE 138 PBDE 183 PBDE 190 PBDE 209	J (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (LC)
SWHB-27-SBB	PBDE 047 PBDE 100 PBDE 153	J (all detects) J (all detects) J (all detects)	A	Duplicate sample analysis (RPD) (HD)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PBDE 017	UJ (all non-detects)	P	Laboratory control samples (%R) (LL)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Polybrominated Diphenyl Ethers - Laboratory Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Polybrominated Diphenyl Ethers - Field Blank Data Qualification Summary - SDG
1504003-002**

No Sample Data Qualified in this SDG

METHOD: GC/MS Polybrominated Diphenyl Ethers (EPA SW 846 Method 8270D-NCI)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / MW	
II.	GC/MS Instrument performance check	N	
III.	Initial calibration/ICV	MW A	r^2 ICV $\leq 30\%$
IV.	Continuing calibration	MW	CCV $\leq 20\%$
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	A	
VIII.	Matrix spike/Matrix spike duplicates / DUP	A / MW	
IX.	Laboratory control samples / CRM	MW/A	LOQ/D. CRM
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinsate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-26-M	31963	Tissue	04/22/14
2	SWHB-27-SBB	31966	Tissue	04/23/14
3	SWHB-27-P	31969	Tissue	04/23/14
4	SWHB-30-CH	31973	Tissue	04/23/14
5	SWHB-06-CH-Small	31989	Tissue	04/22/14
6	SWHB-06-M	31992	Tissue	04/22/14
7	SWHB-22-SP	32017	Tissue	04/21/14
8	SWHB-27-SBBMS	31966MS	Tissue	04/23/14
9	SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
10	↓ Dup	↓ Dup	↓	↓
11				
12				
13	0-7118			

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		<input checked="" type="checkbox"/>		
Was cooler temperature criteria met?	<input checked="" type="checkbox"/>			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?			<input checked="" type="checkbox"/>	
Were all samples analyzed within the 12 hour clock criteria?			<input checked="" type="checkbox"/>	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	<input checked="" type="checkbox"/>			
Were all percent relative standard deviations (%RSD) ≤ 20% and relative response factors (RRF) within method criteria?			<input checked="" type="checkbox"/>	
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990?		<input checked="" type="checkbox"/>		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) < 30% or percent recoveries (%R) 70-130%?	<input checked="" type="checkbox"/>			
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	<input checked="" type="checkbox"/>			
Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria?		<input checked="" type="checkbox"/>		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	<input checked="" type="checkbox"/>			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		<input checked="" type="checkbox"/>		
VI. Field blanks				
Were field blanks were identified in this SDG?		<input checked="" type="checkbox"/>		
Were target compounds detected in the field blanks?			<input checked="" type="checkbox"/>	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?	<input checked="" type="checkbox"/>			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			<input checked="" type="checkbox"/>	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			<input checked="" type="checkbox"/>	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			04P
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	/			
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			ERM
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
X. Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds of the associated calibration standard?	/			
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	/			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?	/			
XIV. System performance				
System performance was found to be acceptable.	/			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			

VALIDATION FINDINGS WORKSHEET
Technical Holding Times

All circled dates have exceeded the technical holding times.

N/A Were all cooler temperatures within validation criteria?

METHOD : GC/MS BNA (EPA SW 846 Method 8270D)							
Sample ID	Matrix	Preserved	Sampling Date	<u>Extraction date</u>	Analysis date	Total # of Days	Qualifier
1	Tissues	Y	4-22-14	5-29-15		402	1/11/15 (H)
2			4-23-14			401	
3			↓			↓	
4			↓			↓	
5			4-22-14			402	
6			↓			↓	
7			4-21-14			403	
8			4-23-14			401	
9			↓			↓	
10	↓	↓	↓	↓		↓	↓
(10/13 + 10/10)	↓						

TECHNICAL HOLDING TIME CRITERIA

Water: Extracted within 7 days, analyzed within 40 days.
Soil: Extracted within 14 days, analyzed within 40 days.

VALIDATION FINDINGS WORKSHEET
Initial Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270^c)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?
- Y N N/A Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?
- Y N N/A Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? _____
- Y N N/A Did the initial calibration meet the acceptance criteria?
- Y N N/A Were all %RSDs and RRFs within the validation criteria of $\leq 30/15$ %RSD and ≥ 0.05 RRF? 20/70 ?

#	Date	Standard ID	Compound	Finding %RSD (Limit: $\leq 30.0/15\%$)	Finding RRF (Limit: ≥ 0.05)	Associated Samples	Qualifications
	6/17/15	1CAL	PBDE 209	$r^2 = 0.98570797$		N/A (ND)	Y/N/A (BC)

VALIDATION FINDINGS WORKSHEET
Continuing Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

Y N/A Were percent differences (%D) $\leq 20\%$ and relative response factors (RRF) within the method criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: $\leq 20.0\%$)	Finding RRF (Limit)	Associated Samples	Qualifications
	6/18/15	CCV	PBDE 100	22		3-7 (detst+NB)	N/A ($\leq C$)
			099	22			
			085	28			
			154	29			
			159	37			
			138	38			
			183	39			
			190	49			
			209	55			

**VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates**

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		10	PBDE047	()	()	30 (≤75)	2 (dets)	↓ dets / A (HD)
			100	()	()	54 (↓)		↓
			153	()	()	33 (↓)		
				()	()	()		
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LDC #: 369TB2C

**VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)**

Page: 1 of 1
Reviewer: Q
2nd Reviewer: NB

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Was a LCS required?

Y N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>31945-BS1</u> <u>BS2</u>	<u>PBDEsIT</u>	<u>59 (70-130)</u>	<u>62 (70-130)</u>	()	<u>NI (ND)</u>	<u>✓ NI / P (LL)</u>
				()	()	()		
				()	()	()		
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				()	()	()		

Method: PBDE (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
6/17/2015	PBDE047	1	0.0050	0.0047302	0.000025
		2	0.0125	0.0121440	0.00005
		3	0.0250	0.0274772	0.0001
		4	0.0375	0.0411628	0.0002
		5	0.0500	0.0539130	0.0004
		6	0.1000	0.1139320	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.121144	1.13813
Correlation Coefficient	0.999642	0.99953
Coefficient of Determination (r ²)	0.999284	

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	CCV	6/18/15	Phenol (1st internal standard) <u>FBDE047</u>	<u>100</u>	<u>87.4297</u>	<u>87.429</u>	<u>13</u>	<u>13</u>
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Surrogate Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: SF/SS * 100

Where: SF = Surrogate Found
 SS = Surrogate Spiked

Sample ID: 1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5 DTBDE	50.0	54.83	110	110	0
2-Fluorobiphenyl FTBDE	↓	44.53	89	89	0
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = |MSC - MSC| * 2/(MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 8/9

Compound	Spike Added (115/9)		Sample Concentration (115/9)	Spiked Sample Concentration (115/9)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
PBDE047	40.04	40.82	2.12	46.88	57.1	111	112	122	123	9	9

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SC/SA)

Where: SSC = Spike concentration
 SA = Spike added

RPD = | LCSC - LCSDC | * 2 / (LCSC + LCSDC)

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31945-B51/-B52

Compound	Spike Added (125/9)		Spike Concentration (125/9)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc	Reported	Recalc	Reported	Recalculated
PBDE 047	100	100	98.38	89.98	98	98	90	90	9	9

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

N N/A
 N N/A

Were all reported results recalculated and verified for all level IV samples?

Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1, PBDE 047

$$\text{Conc.} = \frac{(6827) \times (2000) \times (1.6033)}{(782/90) \times (1.13815)}$$

$$= 1.95 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (ng/g)	Calculated Concentration ()	Qualification
		<u>PBDE 047</u>	<u>1.95</u>		

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Chlorinated Pesticides

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories Inc.

Sample Delivery Group (SDG): 1504003-002

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-26-M	31963	Tissue	04/22/14
SWHB-27-SBB	31966	Tissue	04/23/14
SWHB-27-P	31969	Tissue	04/23/14
SWHB-30-CH	31973	Tissue	04/23/14
SWHB-06-CH-Small	31989	Tissue	04/22/14
SWHB-06-M	31992	Tissue	04/22/14
SWHB-22-SP	32017	Tissue	04/21/14
SWHB-27-SBBMS	31966MS	Tissue	04/23/14
SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
SWHB-27-SBBDUP	31966DUP	Tissue	04/23/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Chlorinated Pesticides by Environmental Protection Agency (EPA) SW 846 Method 8270D

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-26-M SWHB-06-CH-Small SWHB-06-M	All TCL compounds	402	365	J (all detects) UJ (all non-detects)	P
SWHB-27-SBB SWHB-27-P SWHB-30-CH	All TCL compounds	401	365	J (all detects) UJ (all non-detects)	P
SWHB-22-SP	All TCL compounds	403	365	J (all detects) UJ (all non-detects)	P

II. GC Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at the required frequency.

All ion abundance requirements were met.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

For compounds where average calibration factors were utilized, percent relative standard deviations (%RSD) were less than or equal to 20.0%.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
05/27/15	4,4'-DDT	0.98565806	All samples in SDG 1504003-002	UJ (all non-detects)	A

Retention time windows were established as required by the method.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds.

IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Standard	Compound	%D	Associated Samples	Flag	A or P
06/13/15	CCV	2,4'-DDD 4,4'-DDD Toxaphene	21 28 23	SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	J (all detects) UJ (all non-detects)	A

Retention times of all compounds in the calibration standards were within the established retention time windows.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Surrogates

Surrogates were added to all samples as required by the method. All surrogate recoveries (%R) were within QC limits.

VIII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-27-SBBDUP (SWHB-27-SBB)	2,4'-DDD 2,4'-DDE alpha-Chlordane	26 (≤25) 65 (≤25) 164 (≤25)	J (all detects) UJ (all non-detects)	A

IX. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31945-BS1/BS2 (All samples in SDG 1504003-002)	4,4'-DDT	136 (70-130)	-	NA	-

Relative percent differences (RPD) were within QC limits.

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits.

X. Field Duplicates

No field duplicates were identified in this SDG.

XI. Compound Quantitation

All compound quantitations met validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-002	Compound reported below the RL and above the MDL	J (all detects)	A

XII. Target Compound Identification

All target compound identifications met validation criteria.

XIII. System Performance

The system performance was acceptable.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , continuing calibration %D, DUP RPD, and results reported below the RL and above the MDL, data were qualified as estimated in seven samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Chlorinated Pesticides - Data Qualification Summary - SDG 1504003-002**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-26-M SWHB-06-CH-Small SWHB-06-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-22-SP	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-26-M SWHB-06-CH-Small SWHB-06-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-22-SP	4,4'-DDT	UJ (all non-detects)	A	Initial calibration (r^2) (BC)
SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	2,4'-DDD 4,4'-DDD	J (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (CH)
SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	Toxaphene	J (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (LC)
SWHB-27-SBB	2,4'-DDD 2,4'-DDE alpha-Chlordane	J (all detects) UJ (all non-detects)	A	Duplicate sample analysis (RPD) (HD)
SWHB-26-M SWHB-06-CH-Small SWHB-06-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-22-SP	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Chlorinated Pesticides - Laboratory Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Chlorinated Pesticides - Field Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

METHOD: GC/MS Chlorinated Pesticides (EPA SW 846 Method 8270D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A/W	
II.	GC/MS Instrument performance check	A	
III.	Initial calibration/ICV	W/A	RSB ≤ 20% . r ² 1CV ≤ 30%
IV.	Continuing calibration	W	CCV ≤ 20%
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	A	
VIII.	Matrix spike/Matrix spike duplicates 1/DUP	A/W	
IX.	Laboratory control samples 1/CRM	W/A	LCB/D. CRM
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	SWHB-26-M	31963	Tissue	04/22/14
2	SWHB-27-SBB	31966	Tissue	04/23/14
3	SWHB-27-P	31969	Tissue	04/23/14
4	SWHB-30-CH	31973	Tissue	04/23/14
5	SWHB-06-CH-Small	31989	Tissue	04/22/14
6	SWHB-06-M	31992	Tissue	04/22/14
7	SWHB-22-SP	32017	Tissue	04/21/14
8	SWHB-27-SBBMS	31966MS	Tissue	04/23/14
9	SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
10	↓ DUP	31966 DUP	↓	↓
11				
12				
13	0-T118 / 31945-B1			

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?	/			
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% and relative response factors (RRF) within method criteria?	/			
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990?		/		
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	/			
Were all percent differences (%D) < 30% or percent recoveries (%R) 70-130%?	/			
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria?		/		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?	/			
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<u>DUP</u>
Was a MS/MSD analyzed every 20 samples of each matrix?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<u>CFM</u>
Was an LCS analyzed per analytical batch?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
X. Field duplicates				
Were field duplicate pairs identified in this SDG?	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Were target compounds detected in the field duplicates?	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were retention times within + 30 seconds of the associated calibration standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Were chromatogram peaks verified and accounted for?	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XIV. System performance				
System performance was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	

VALIDATION FINDINGS WORKSHEET

METHOD: Pesticide/PCBs (EPA SW 846 Method 8081/8082)

A. alpha-BHC	I. Dieldrin	Q. Endrin ketone	Y. Aroclor-1242	GG. Chlordane
B. beta-BHC	J. 4,4'-DDE	R. Endrin aldehyde	Z. Aroclor-1248	HH. Chlordane (Technical)
C. delta-BHC	K. Endrin	S. alpha-Chlordane	AA. Aroclor-1254	II. oxy-Chlordane
D. gamma-BHC	L. Endosulfan II	T. gamma-Chlordane	BB. Aroclor-1260	JJ. Mirex
E. Heptachlor	M. 4,4'-DDD	U. Toxaphene	CC. 2,4'-DDD	KK. 4,4'-DDMU
F. Aldrin	N. Endosulfan sulfate	V. Aroclor-1016	DD. 2,4'-DDE	LL.
G. Heptachlor epoxide	O. 4,4'-DDT	W. Aroclor-1221	EE. 2,4'-DDT	MM.
H. Endosulfan I	P. Methoxychlor	X. Aroclor-1232	FF. Hexachlorobenzene	NN.

Notes: _____

VALIDATION FINDINGS WORKSHEET
Technical Holding Times

All circled dates have exceeded the technical holding times.
 Y N N/A Were all cooler temperatures within validation criteria?

METHOD : GC/MS BNA (EPA SW 846 Method 8270D)							
Sample ID	Matrix	Preserved	Sampling Date	Extraction date	Analysis date	Total # of Days	Qualifier
1	Tissue	Y	4-22-14	5-29-15		402	N/A (H)
2			4-23-14			401	
3			↓			↓	
4			↓			↓	
5			4-22-14			402	
6			↓			↓	
7			4-21-14			403	
8			4-23-14			401	
9			↓			↓	
10	↓	↓	↓	↓		↓	↓
(lots + NO)							

TECHNICAL HOLDING TIME CRITERIA

Water: Extracted within 7 days, analyzed within 40 days.
 Soil: Extracted within 14 days, analyzed within 40 days.

VALIDATION FINDINGS WORKSHEET
Initial Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Did the laboratory conduct an acceptable 5 point calibration prior to sample analysis?
- Y N N/A Were percent relative standard deviations (%RSD) and relative response factors (RRF) within method criteria for all CCC's and SPCC's?
- Y N N/A Was a curve fit used for evaluation? If yes, what was the acceptance criteria used for evaluation? _____
- Y N N/A Did the initial calibration meet the acceptance criteria?
- Y N N/A Were all %RSDs and RRFs within the validation criteria of $\leq 30/15$ %RSD and ≥ 0.05 RRF ?

#	Date	Standard ID	Compound	Finding %RSD (Limit: $\leq 30.0/15\%$)	Finding RRF (Limit: > 0.05)	Associated Samples	Qualifications
	5/27/15	ICAL	0	$r^2 = 0.98565806$		ML (ND)	Y/N/A (BC)

VALIDATION FINDINGS WORKSHEET
Continuing Calibration

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?

Y (N) N/A Were percent differences (%D) \leq 20 % and relative response factors (RRF) within the method criteria?

#	Date	Standard ID	Compound	Finding %D (Limit: \leq 20.0%)	Finding RRF (Limit)	Associated Samples	Qualifications
	6/13/15	QCV	CC	21		4-7 (dot 3 + 10)	Y/N/A (CH) ↓ ↓
			M	28			
			U	23			(L.C.)

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

Y N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

Y N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>2/9 10</u>	<u>CC</u>	()	()	<u>26 (≤25)</u>	<u>2 (dots + ND)</u>	<u>N/A (HO)</u>
			<u>DD</u>	()	()	<u>65</u>	↓	↓
			<u>S</u>	()	()	<u>16A</u>		
				()	()	()		
				()	()	()		
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VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Was a LCS required?
- Y N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		31945 BS		()	()	()		
		31945 BS		()	()	()		
		<u>31945 BS</u>	<u>0</u>	<u>136 (70-130)</u>	()	()	<u>ML (N/D)</u>	<u>lots of (HL)</u>
		<u><BS></u>		()	()	()		
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Method: Pesticides (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	Oxychlorane	1	0.025	0.00645	0.000025
		2	0.050	0.0110995	0.00005
		3	0.100	0.0196845	0.0001
		4	0.250	0.0491241	0.0002
		5	0.500	0.1043256	0.0004
		6	1.000	0.2097125	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	0.208838	0.21239
Correlation Coefficient	0.999882	0.99962
Coefficient of Determination (r ²)	0.999764	

Method: Pesticides (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	4,4'-DDE	1	0.025	0.0655146	0.000025
		2	0.050	0.1322760	0.00005
		3	0.100	0.2668935	0.0001
		4	0.250	0.7433727	0.0002
		5	0.500	1.5492299	0.0004
		6	1.000	3.2876129	0.0008

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	3.230938	3.24249
Correlation Coefficient	0.999423	0.99804
Coefficient of Determination (r ²)	0.998846	

LDC#: 36197B3A

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
 Reviewer: 9
 2nd Reviewer: JL

Method: GC/MS Pesticides (EPA SW 846 Method 8270D)

Calibration Date	System	Compound	Standard	(Y) Response	(X) Concentration
5/27/2015	Q1	4,4'-DDMU	s1	0.0849309	0.025
			s2	0.16469	0.050
			s3	0.348635	0.100
			s4	1.0921435	0.250
			s5	2.4835837	0.500
			s6	5.683011	1.000

Regression Output

Reported

Constant	-0.206259	-0.202600
Std Err of Y Est		
R Squared	0.995686	0.995686
Degrees of Freedom		
X Coefficient(s)	5.763401	5.763406
Std Err of Coef.		
Correlation Coefficient	0.997841	
Coefficient of Determination (r ²)	0.995686	0.995686

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = $100 * (\text{ave. RRF} - \text{RRF}) / \text{ave. RRF}$
 $\text{RRF} = (A_x)(C_{is}) / (A_{is})(C_x)$

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	<u>02V</u>	<u>6/13/15</u>	Phenol (1st internal standard) <u>11</u>	<u>500</u>	<u>397.6829</u>	<u>397.6933</u>	<u>20</u>	<u>20</u>
			Naphthalene (2nd internal standard) <u>J</u>	<u>↓</u>	<u>497.9500</u>	<u>497.9500</u>	<u>0</u>	<u>0</u>
			Fluorene (3rd internal standard) <u>HK</u>	<u>↓</u>	<u>585.0594</u>	<u>584.4332</u>	<u>17</u>	<u>17</u>
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Surrogate Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery: $SF/SS * 100$ Where: SF = Surrogate Found
SS = Surrogate SpikedSample ID: 1

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5 <u>RB030</u>	<u>400</u>	<u>346.87</u>	<u>87</u>	<u>87</u>	<u>0</u>
2-Fluorobiphenyl <u>112</u>	<u>↓</u>	<u>402.12</u>	<u>101</u>	<u>101</u>	<u>↓</u>
Terphenyl-d14 <u>198</u>	<u>↓</u>	<u>348.47</u>	<u>87</u>	<u>87</u>	<u>↓</u>
Phenol-d5 <u>TMX</u>	<u>↓</u>	<u>350.59</u>	<u>88</u>	<u>88</u>	<u>↓</u>
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

Sample ID:

	Surrogate Spiked	Surrogate Found	Percent Recovery Reported	Percent Recovery Recalculated	Percent Difference
Nitrobenzene-d5					
2-Fluorobiphenyl					
Terphenyl-d14					
Phenol-d5					
2-Fluorophenol					
2,4,6-Tribromophenol					
2-Chlorophenol-d4					
1,2-Dichlorobenzene-d4					

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270C)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = |MSC - MSC| * 2 / (MSC + MSDC)

MSC = Matrix spike concentration

MSDC = Matrix spike duplicate concentration

MS/MSD samples: 8/9

Compound	Spike Added (ng/g)		Sample Concentration (ng/g)	Spiked Sample Concentration (ng/g)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc	Reported	Recalc	Reported	Recalculated
J	200.2	204.1	35.51	220.0	233.8	92	92	92	92	0	0
11	↓	↓	161.6 ND	161.6	162.5	81	81	80	80	1	1
KK	↓	↓	30.48	204.53	194.47	87	87	80	80	8	8

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270^DC)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SC/SA)

Where: SSC = Spike concentration
 SA = Spike added

RPD = | LCSC - LCSDC | * 2 / (LCSC + LCSDC)

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31945-B31-B32

Compound	Spike Added (NS/A)		Spike Concentration (NS/A)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
J	500	500	392.42	366.08	78	78	73	73	7	7
II	↓	↓	415.63	403.41	89	89	81	81	9	9
KK	↓	↓	437.07	393.05	87	87	79	79	10	10

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

Y / N / N/A
Y / N / N/A

Were all reported results recalculated and verified for all level IV samples?
 Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1 , J :

$$\text{Conc.} = \frac{(5563)(1000)(1.6033)}{(12499)(3.2419)} = 22.55 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (ng/g)	Calculated Concentration ()	Qualification
		<u> J </u>	<u> 22.55 </u>		

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Metals

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories, Inc.

Sample Delivery Group (SDG): 1504003-002

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-26-CH	31957	Tissue	04/22/14
SWHB-26-SP-Large	31959	Tissue	04/22/14
SWHB-26-BP	31960	Tissue	04/22/14
SWHB-27-SP	31968	Tissue	04/23/14
SWHB-01-SBB	31980	Tissue	04/22/14
SWHB-01-CH	31981	Tissue	04/22/14
SWHB-26-SBB	31956	Tissue	04/22/14
SWHB-26-SBBMS	31956	Tissue	04/22/14
SWHB-26-SBBMSD	31956	Tissue	04/22/14
SWHB-26-SBBDUP	31956	Tissue	04/22/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (January 2010). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Mercury and Selenium by Environmental Protection Agency (EPA) SW 846 Method 6020/EPA Method 245.7

All sample results were subjected to Level IV evaluation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met with the following exceptions:

Sample	Analyte	Total Days From Sample Collection Until Analysis	Required Holding Time (in Days) From Sample Collection Until Analysis	Flag	A or P
SWHB-26-CH SWHB-26-SP-Large SWHB-26-BP SWHB-01-SBB SWHB-01-CH SWHB-26-SBB	Selenium Mercury	414 419	365 180	J (all detects) J (all detects)	P
SWHB-27-SP	Selenium Mercury	413 418	365 180	J (all detects) J (all detects)	P

II. ICPMS Tune

The mass calibration was within 0.1 AMU and the percent relative standard deviation (%RSD) was less than or equal to 5%.

III. Instrument Calibration

Initial and continuing calibrations were performed as required by the methods.

The initial calibration verification (ICV) and continuing calibration verification (CCV) standards were within QC limits.

IV. ICP Interference Check Sample Analysis

The interference check sample (ICS) was not performed by the laboratory. The laboratory used a reaction chamber with mixed gases as well as internal equations to compensate for any interferences.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Analyte	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SWHB-26-SBBMS/MSD (SWHB-26-SBB)	Mercury	121 (80-120)	121 (80-120)	J (all detects)	A

Relative percent differences (RPD) were within QC limits.

VIII. Duplicate Sample Analysis

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits.

IX. Serial Dilution

Serial dilution was not performed for this SDG.

X. Laboratory Control Samples/Standard Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the methods. Percent recoveries (%R) were within QC limits. Relative percent differences (RPD) were within QC limits.

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits.

XI. Field Duplicates

No field duplicates were identified in this SDG.

XII. Internal Standards (ICP-MS)

All internal standard percent recoveries (%R) were within QC limits with the following exceptions:

Sample	Internal Standard	%R (Limits)	Affected Analyte	Flag	A or P
SWHB-26-CH	Scandium-45	160.8 (30-120)	Selenium	J (all detects)	A
SWHB-26-SP-Large	Scandium-45	159.8 (30-120)	Selenium	J (all detects)	A

Sample	Internal Standard	%R (Limits)	Affected Analyte	Flag	A or P
SWHB-26-BP	Scandium-45	151.5 (30-120)	Selenium	J (all detects)	A
SWHB-27-SP	Scandium-45	144.0 (30-120)	Selenium	J (all detects)	A
SWHB-01-SBB	Scandium-45	144.1 (30-120)	Selenium	J (all detects)	A
SWHB-01-CH	Scandium-45	146.0 (30-120)	Selenium	J (all detects)	A
SWHB-26-SBB	Scandium-45	156.0 (30-120)	Selenium	J (all detects)	A

XIII. Sample Result Verification

All sample result verifications were acceptable.

All analytes reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-002	Analyte reported below the RL and above the MDL	J (all detects)	A

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected in this SDG.

Due to holding time exceedance, MS/MSD %R, internal standard %R, and results reported below the RL and above the MDL, data were qualified as estimated in seven samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Metals - Data Qualification Summary - SDG 1504003-002**

Sample	Analyte	Flag	A or P	Reason (Code)
SWHB-26-CH SWHB-26-SP-Large SWHB-26-BP SWHB-01-SBB SWHB-01-CH SWHB-26-SBB SWHB-27-SP	Selenium Mercury	J (all detects) J (all detects)	P	Technical holding time (H)
SWHB-26-SBB	Mercury	J (all detects)	A	Matrix spike/Matrix spike duplicate (%R) (HM)
SWHB-26-CH SWHB-26-SP-Large SWHB-26-BP SWHB-27-SP SWHB-01-SBB SWHB-01-CH SWHB-26-SBB	Selenium	J (all detects)	A	Internal standards (%R) (*XII)
SWHB-26-CH SWHB-26-SP-Large SWHB-26-BP SWHB-27-SP SWHB-01-SBB SWHB-01-CH SWHB-26-SBB	Analyte reported below the RL and above the MDL	J (all detects)	A	Sample result verification (DL)

**City of San Diego SWBH Study
Metals - Laboratory Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Metals - Field Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

LDC #: 36197B4a

VALIDATION COMPLETENESS WORKSHEET

Date: 4/22/14

SDG #: 1504003-002

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: Mercury & Selenium (EPA SW 846 Method 6020/EPA Method 245.7)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	SW	4/22
II.	ICP/MS Tune	A	
III.	Instrument Calibration	A	
IV.	ICP Interference Check Sample (ICS) Analysis	SW	
V.	Laboratory Blanks	SW	
VI.	Field Blanks	N	
VII.	Matrix Spike/Matrix Spike Duplicates	SW	MSD = (8,9)
VIII.	Duplicate sample analysis	A	DUP
IX.	Serial Dilution	N	
X.	Laboratory control samples	A	LCSID's SEM
XI.	Field Duplicates	N	
XII.	Internal Standard (ICP-MS)	SW	
XIII.	Sample Result Verification	A	
XIV.	Overall Assessment of Data	A	

Note: A = Acceptable
 N = Not provided/applicable
 SW = See worksheet

ND = No compounds detected
 R = Rinstate
 FB = Field blank

D = Duplicate
 TB = Trip blank
 EB = Equipment blank

SB=Source blank
 OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-26-CH	31957	Tissue	04/22/14
2	SWHB-26-SP-Large	31959	Tissue	04/22/14
3	SWHB-26-BP	31960	Tissue	04/22/14
4	SWHB-27-SP	31968	Tissue	04/23/14
5	SWHB-01-SBB	31980	Tissue	04/22/14
6	SWHB-01-CH	31981	Tissue	04/22/14
7	SWHB-26-SBB SWHB-01-P	31984	Tissue	04/22/14
8	# 7 MS			
9	# 7 MSD			
10	# 7 DUP			
11				
12				

Notes: _____

Method:Metals (EPA SW 846 Method 6010B/7000/6020)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.		/		
Cooler temperature criteria was met.	/			
II. ICP/MS Tune				
Were all isotopes in the tuning solution mass resolution within 0.1 amu?	/			
Were %RSD of isotopes in the tuning solution $\leq 5\%$?	/			
III. Calibration				
Were all instruments calibrated daily, each set-up time?	/			
Were the proper number of standards used?	/			
Were all initial and continuing calibration verification %Rs within the 90-110% (80-120% for mercury) QC limits?	/			
Were all initial calibration correlation coefficients ≥ 0.995 ?	/			
IV. Blanks				
Was a method blank associated with every sample in this SDG?	/			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		/		
V. ICP Interference Check Sample				
Were ICP interference check samples performed daily?		/		
Were the AB solution percent recoveries (%R) with the 80-120% QC limits?			/	
VI. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.	/			
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq 20\%$ for waters and $\leq 35\%$ for soil samples? A control limit of +/- RL (+/-2X RL for soil) was used for samples that were $\leq 5X$ the RL, including when only one of the duplicate sample values were $\leq 5X$ the RL.	/			
VII. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			
Was an LCS analyzed per extraction batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% QC limits for water samples and laboratory established QC limits for soils?	/			

Validation Area	Yes	No	NA	Findings/Comments
VIII. Internal Standards (EPA SW 846 Method 6020/EPA 200.8)				
Were all the percent recoveries (%R) within the 30-120% (6020)/60-125% (200.8) of the intensity of the internal standard in the associated initial calibration?		/		
If the %Rs were outside the criteria, was a reanalysis performed?		/		
IX. ICP Serial Dilution				
Was an ICP serial dilution analyzed if analyte concentrations were > 50X the MDL (ICP)/>100X the MDL(ICP/MS)?			/	
Were all percent differences (%Ds) < 10%?			/	
Was there evidence of negative interference? If yes, professional judgement will be used to qualify the data.			/	
X. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XI. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
XII. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		
Target analytes were detected in the field duplicates.			/	
XIII. Field blanks				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			/	

VALIDATION FINDINGS WORKSHEET
Technical Holding Times

All circled dates have exceeded the technical holding time.
 Y/N N/A Were all samples preserved as applicable to each method?
 Y/N N/A Were all cooler temperatures within validation criteria?

Method:		200.8		245.7			
Parameters:		Se		Hg			
Technical holding time:		180 Days		28 Days			
Sample ID	Sampling date	Analysis date	Analysis date	Analysis date	Analysis date	Analysis date	Qualifier
1-3, 5-10	04/22/14	06/10/15	414 Days				J/R/P (det) (H)
4	04/23/14	06/10/15	413 Days				J/R/P (det) (H)
1-3, 5-10	04/22/14			06/15/15	419 Days		J/R/P (det) (H)
4	04/23/14			06/15/15	418 Days		J/R/P (det) (H)

VALIDATION FINDINGS WORKSHEET
ICP Interference Check Sample

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y ~~N~~ ~~N/A~~ Were ICP interference check samples performed as required?

Y ~~N~~ ~~N/A~~ Were the AB solution percent recoveries (%R) within the control limits of 80-120% ?

LEVEL IV ONLY:

Y ~~N~~ ~~N/A~~ Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	Date	ICS Identification	Analyte	Finding	Associated Samples	Qualifications
		ICSA/AB	Se	The ICP interference check sample was not performed by the laboratory. The laboratory used a reaction chamber with mixed gases as well as internal equations to compensate for any interferences.	All	Text

Comments: _____

VALIDATION FINDINGS WORKSHEET

Blanks

METHOD: Trace metals (EPA SW 846 Method 6010/7000)

#	Date	Blank ID	Analyte	Finding	Associated Samples	Qualifications
		CCB	Hg	Closing CCB was not performed	All	Text

Comments: _____

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: Trace metals (EPA SW 846 Method 6010/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Was a matrix spike analyzed for each matrix in this SDG?
- Y N N/A Were matrix spike percent recoveries (%R) within the control limits of 80-120? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.
- Y N N/A Were all duplicate sample relative percent differences (RPD) \leq 20% for samples?
- LEVEL IV ONLY:**
- Y N N/A Were recalculated results acceptable? See Level IV Recalculation Worksheet for recalculations.

#	MS/MSD ID	Matrix	Analyte	MS %Recovery	MSD %Recovery	RPD (Limits)	Associated Samples	Qualifications
	8/9	Tissue	Hg	121	121		7	Jdet/A (det) (HM)

Comments: _____

VALIDATION FINDINGS WORKSHEET
Internal Standards (ICP-MS)

METHOD: Metals (EPA SW 846 Method 6020C)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

Y N N/A Were all internal standard percent recoveries within 30-120% of the intensity of the internal standard in the initial calibration standard ?

Y N N/A If the response to either of the above questions is no, were the samples reanalyzed as required ?

#	Date	Internal Standard	Associated Metals	%R (Limits)	Associated Samples	Qualifications
		Sc (45) [1]	Se	160.8 (30-120)	1	J/UJ/A (det)
		Sc (45) [1]	Se	159.8 (30-120)	2	J/UJ/A (det)
		Sc (45) [1]	Se	151.5 (30-120)	3	J/UJ/A (det)
		Sc (45) [1]	Se	144.0 (30-120)	4	J/UJ/A (det)
		Sc (45) [1]	Se	144.1 (30-120)	5	J/UJ/A (det)
		Sc (45) [1]	Se	146.0 (30-120)	6	J/UJ/A (det)
		Sc (45) [1]	Se	156.0 (30-120)	7	J/UJ/A (det)

LDC #: 36A1B4a

VALIDATION FINDINGS WORKSHEET
Initial and Continuing Calibration Calculation Verification

Page: 1 of 1
 Reviewer: SD
 2nd Reviewer: A

METHOD: Trace Metals (See cover)

An initial and continuing calibration verification percent recovery (%R) was recalculated for each type of analysis using the following formula:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where, Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution
 True = concentration (in ug/L) of each analyte in the ICV or CCV source

Standard ID	Type of Analysis	Element	Found (ug/L)	True (ug/L)	Recalculated	Reported	Acceptable (Y/N)
					%R	%R	
	ICP (Initial calibration)						
<u>ICV</u>	ICP/MS (Initial calibration)	<u>Se</u>	<u>106 ppm</u>	<u>100 ppm</u>	<u>106 %R</u>	<u>NR</u>	<u>Y</u>
<u>ICV</u> <u>17:51</u>	CVAA (Initial calibration)	<u>Hg</u>	<u>1080 ppt</u>	<u>1000 ppt</u>	<u>108 %R</u>	<u>NR</u>	<u>Y</u>
	ICP (Continuing calibration)						
<u>CCV</u>	ICP/MS (Continuing calibration)	<u>Se</u>	<u>103 ppm</u>	<u>100 ppm</u>	<u>103 %R</u>	<u>NR</u>	<u>Y</u>
<u>CCV</u> <u>18:54</u>	CVAA (Continuing calibration)	<u>Hg</u>	<u>1110 ppt</u>	<u>1000 ppt</u>	<u>111 %R</u>	<u>NR</u>	<u>Y</u>
	GFAA (Initial calibration)						
	GFAA (Continuing calibration)						

Comments: _____

LDC #: 30197B4a

VALIDATION FINDINGS WORKSHEET
Level IV Recalculation Worksheet

Page: 1 of 1
Reviewer: JS
2nd Reviewer: [Signature]

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Percent recoveries (%R) for an ICP interference check sample, a laboratory control sample and a matrix spike sample were recalculated using the following formula:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$
 Where, Found = Concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).
 True = Concentration of each analyte in the source.

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$
 Where, S = Original sample concentration
 D = Duplicate sample concentration

An ICP serial dilution percent difference (%D) was recalculated using the following formula:

$$\%D = \frac{|I-SDR|}{I} \times 100$$
 Where, I = Initial Sample Result (mg/L)
 SDR = Serial Dilution Result (mg/L) (Instrument Reading x 5)

Sample ID	Type of Analysis	Element	Found / S / I (units)	True / D / SDR (units)	Recalculated	Reported	Acceptable (Y/N)
					%R / RPD / %D	%R / RPD / %D	
N	ICP interference check						
LCS 18:13	Laboratory control sample	Hg	1130 ug/g	1000 ug/g	113%R	113%R	Y
MS 15:05	Matrix spike	Se	(SSR-SR) 69.71 ug/g	60.79 ug/g	106%R	106%R	↓
DUP 12:55	Duplicate	Se	1.668 ug/g	1.613 ug/g	3%RPD	3%RPD	↓
N	ICP serial dilution						

Comments: _____

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

METHOD: Trace Metals (EPA SW 846 Method 6010/6020/7000)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Have results been reported and calculated correctly?
- Y N N/A Are results within the calibrated range of the instruments and within the linear range of the ICP?
- Y N N/A Are all detection limits below the CRDL?

Detected analyte results for (1) Se were recalculated and verified using the following equation:

Concentration = $\frac{(RD)(FV)(Dil)}{(In. Vol.)}$ Recalculation:

- RD = Raw data concentration
- FV = Final volume (ml)
- In. Vol. = Initial volume (ml) or weight (G)
- Dil = Dilution factor

RD = 1.891 ug/g

#	Sample ID	Analyte	Reported Concentration (ug/g)	Calculated Concentration (ug/g)	Acceptable (Y/N)
	1	Se	1.891	1.891	Y
	2	Hg	0.1387	0.1387	↓
	3	Se	1.59	1.59	
	4	Hg	0.1524	0.1524	
	5	Se	2.01	2.01	
	6	Hg	0.1846	0.1846	
	7	Se	1.613	1.613	

Note: _____

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Wet Chemistry

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories, Inc.

Sample Delivery Group (SDG): 1504003-002

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-26-P	31962	Tissue	04/22/14
SWHB-26-M	31963	Tissue	04/22/14
SWHB-27-SBB	31966	Tissue	04/23/14
SWHB-27-CH	31967	Tissue	04/23/14
SWHB-27-P	31969	Tissue	04/23/14
SWHB-30-SBB	31972	Tissue	04/23/14
SWHB-30-BP	31974	Tissue	04/23/14
SWHB-27-SBBDUP	31966	Tissue	04/23/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (January 2010). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following methods:

Percent Lipitds by Gravimetric
Percent Solids by Standard Method 2540B

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition.

All technical holding time requirements were met with the following exceptions:

Sample	Analyte	Total Time From Sample Collection Until Analysis	Required Holding Time From Sample Collection Until Analysis	Flag	A or P
SWHB-26-P SWHB-26-M	Percent solids	402 days	180 days	J (all detects)	P
SWHB-27-SBB SWHB-27-CH SWHB-27-P SWHB-30-SBB SWHB-30-BP	Percent solids	401 days	180 days	J (all detects)	P
SWHB-26-P SWHB-26-M	Percent lipids	408 days	180 days	J (all detects)	P
SWHB-27-SBB SWHB-27-CH SWHB-27-P SWHB-30-SBB SWHB-30-BP	Percent lipids	407 days	180 days	J (all detects)	P

II. Initial Calibration

All criteria for the initial calibration of each method were met.

III. Continuing Calibration

Continuing calibration frequency and analysis criteria were met for each method when applicable.

IV. Laboratory Blanks

Laboratory blanks were analyzed as required by the methods. No contaminants were found in the laboratory blanks.

V. Field Blanks

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicates (MSD) analyses were not required by the method.

VII. Duplicate Sample Analysis

Duplicate (DUP) sample analysis was performed on an associated project sample. Results were within QC limits.

VIII. Standard Reference Material

Standard reference materials (SRM) were analyzed as required by the method. The results were within QC limits.

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Sample Result Verification

All sample result verifications were acceptable.

All analytes reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-002	Analyte reported below the RL and above the MDL	J (all detects)	A

XI. Overall Assessment of Data

The analysis was conducted within all specifications of the methods. No results were rejected in this SDG.

Due to holding time exceedance and results reported below the RL and above the MDL, data were qualified as estimated in seven samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Wet Chemistry - Data Qualification Summary - SDG 1504003-002**

Sample	Analyte	Flag	A or P	Reason (Code)
SWHB-26-P SWHB-26-M SWHB-27-SBB SWHB-27-CH SWHB-27-P SWHB-30-SBB SWHB-30-BP	Percent solids	J (all detects)	P	Technical holding time (H)
SWHB-26-P SWHB-26-M SWHB-27-SBB SWHB-27-CH SWHB-27-P SWHB-30-SBB SWHB-30-BP	Percent lipids	J (all detects)	P	Technical holding time (H)
SWHB-26-P SWHB-26-M SWHB-27-SBB SWHB-27-CH SWHB-27-P SWHB-30-SBB SWHB-30-BP	Analyte reported below the RL and above the MDL	J (all detects)	A	Sample results verification (DL)

**City of San Diego SWBH Study
Wet Chemistry - Laboratory Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Wet Chemistry - Field Blank Data Qualification Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

LDC #: 36197B6

VALIDATION COMPLETENESS WORKSHEET

Date: 4/22/14

SDG #: 1504003-002

Level IV

Page: 1 of 1

Laboratory: Physis Environmental Laboratories, Inc.

Reviewer: [Signature]

2nd Reviewer: [Signature]

METHOD: (Analyte) Percent Lipids (Gravimetric), Percent Solids (SM2540B)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	SW	4/22-23/14
II	Initial calibration	A	
III.	Calibration verification	A	
IV	Laboratory Blanks	A	
V	Field blanks	N	
VI.	Matrix Spike/Matrix Spike Duplicates	N	Not Req.
VII.	Duplicate sample analysis	A	DUP
VIII.	Laboratory control samples	A	SRM
IX.	Field duplicates	N	
X.	Sample result verification	A	
XI	Overall assessment of data	A	

Note: A = Acceptable
N = Not provided/applicable
SW = See worksheet

ND = No compounds detected
R = Rinsate
FB = Field blank

D = Duplicate
TB = Trip blank
EB = Equipment blank

SB=Source blank
OTHER:

	Client ID	Lab ID	Matrix	Date
1	SWHB-26-P	31962	Tissue	04/22/14
2	SWHB-26-M	31963	Tissue	04/22/14
3	SWHB-27-SBB	31966	Tissue	04/23/14
4	SWHB-27-CH	31967	Tissue	04/23/14
5	SWHB-27-P	31969	Tissue	04/23/14
6	SWHB-30-SBB	31972	Tissue	04/23/14
7	SWHB-30-BP	31974	Tissue	04/23/14
8	#3 DUP			
9				
10				
11				
12				
13				
14				

Notes:

Method: Inorganics (EPA Method 309.1)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.		<input checked="" type="checkbox"/>		
Cooler temperature criteria was met.	<input checked="" type="checkbox"/>			
II. Calibration				
Were all instruments calibrated daily, each set-up time?			<input checked="" type="checkbox"/>	
Were the proper number of standards used?			<input checked="" type="checkbox"/>	
Were all initial calibration correlation coefficients > 0.995 ?			<input checked="" type="checkbox"/>	
Were all initial and continuing calibration verification %Rs within the 90-110% QC limits?			<input checked="" type="checkbox"/>	
Were titrant checks performed as required? (Level IV only)	<input checked="" type="checkbox"/>			
Were balance checks performed as required? (Level IV only)			<input checked="" type="checkbox"/>	
III. Blanks				
Was a method blank associated with every sample in this SDG?	<input checked="" type="checkbox"/>			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		<input checked="" type="checkbox"/>		
IV. Matrix spike/Matrix spike duplicates and Duplicates				
Were a matrix spike (MS) and duplicate (DUP) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD or MS/DUP. Soil / Water.	<input checked="" type="checkbox"/>			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the 75-125 QC limits? If the sample concentration exceeded the spike concentration by a factor of 4 or more, no action was taken.			<input checked="" type="checkbox"/>	
Were the MS/MSD or duplicate relative percent differences (RPD) $\leq 20\%$ for waters and $\leq 35\%$ for soil samples? A control limit of $\leq \text{CRDL}$ ($\leq 2\text{X CRDL}$ for soil) was used for samples that were $\leq 5\text{X}$ the CRDL, including when only one of the duplicate sample values were $< 5\text{X}$ the CRDL.	<input checked="" type="checkbox"/>			
V. Laboratory control samples				
Was an LCS analyzed for this SDG?	<input checked="" type="checkbox"/>			
Was an LCS analyzed per extraction batch?	<input checked="" type="checkbox"/>			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the 80-120% (85-115% for Method 300.0) QC limits?	<input checked="" type="checkbox"/>			
VI. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			<input checked="" type="checkbox"/>	
Were the performance evaluation (PE) samples within the acceptance limits?			<input checked="" type="checkbox"/>	

LDC #: 3619784

VALIDATION FINDINGS CHECKLIST

Page: 2 of 2
 Reviewer: [Signature]
 2nd Reviewer: [Signature]

Validation Area	Yes	No	NA	Findings/Comments
VII. Sample Result Verification				
Were RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
Were detection limits < RL?	/			
VIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			
IX. Field duplicates				
Field duplicate pairs were identified in this SDG.		/		
Target analytes were detected in the field duplicates.			/	
X. Field blanks				
Field blanks were identified in this SDG.		/		
Target analytes were detected in the field blanks.			/	

VALIDATION FINDINGS WORKSHEET
Sample Specific Analysis Reference

All circled methods are applicable to each sample.

Sample ID	Parameter
(-)	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄ <u>%solids</u> <u>%Lipids</u>
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
DC:8	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄ <u>%solids</u> <u>%Lipids</u>
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
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	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
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	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄
	pH TDS Cl F NO ₃ NO ₂ SO ₄ O-PO ₄ Alk CN NH ₃ TKN TOC Cr6+ ClO ₄

Comments: _____

VALIDATION FINDINGS WORKSHEET
Technical Holding Times

All circled dates have exceeded the technical holding time.

Y N N/A Were all samples preserved as applicable to each method?

Y N N/A Were all cooler temperatures within validation criteria?

Method:		SM2540B		Gravimetric			
Parameters:		Percent Solids		Percent Lipids			
Technical holding time:		<u>130</u> 7 Days		<u>130</u> 365 Days			
Sample ID	Sampling date	Analysis date	Analysis date	Analysis date	Analysis date	Analysis date	Qualifier
1-2	04/22/14	05/29/15	402 Days				J/R/P (det) (H)
3-8	04/23/14	05/29/15	401 Days				J/R/P (det) (H)
1-2	04/22/14			06/04/15	408 Days		J/UJ/P (det) (H)
3-8	04/23/14			06/04/15	407 Days		J/UJ/P (det) (H)

LDC #: 369786

VALIDATION FINDINGS WORKSHEET
Level IV Recalculation Worksheet

Page: 1 of 1
Reviewer: SD
2nd Reviewer: _____

METHOD: Inorganics, Method See Coer

Percent recoveries (%R) for a laboratory control sample and a matrix spike sample were recalculated using the following formula:

$\%R = \frac{\text{Found}}{\text{True}} \times 100$ Where, Found = concentration of each analyte measured in the analysis of the sample. For the matrix spike calculation, Found = SSR (spiked sample result) - SR (sample result).
True = concentration of each analyte in the source.

A sample and duplicate relative percent difference (RPD) was recalculated using the following formula:

$RPD = \frac{|S-D|}{(S+D)/2} \times 100$ Where, S = Original sample concentration
D = Duplicate sample concentration

Sample ID	Type of Analysis	Element	Found / S (units)	True / D (units)	Recalculated	Reported	Acceptable (Y/N)
					%R / RPD	%R / RPD	
SRM	Laboratory control sample	%Lipids	11.72%	10.4%	113%R	113%R	Y
N	Matrix spike sample		(SSR-SR)				
DUP	Duplicate sample	%Solids	23.0%	22.9%	0%RPD	0%RPD	Y

Comments: _____

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

METHOD: Inorganics, Method See Cover

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

- Y N N/A Have results been reported and calculated correctly?
- Y N N/A Are results within the calibrated range of the instruments?
- Y N N/A Are all detection limits below the CRQL?

Compound (analyte) results for (6) % Lipids reported with a positive detect were recalculated and verified using the following equation:

Concentration = $\frac{W - W_v}{(g)(\% \text{ Solids})}$ Recalculation: $\frac{(2.3580g) - (2.2886g)}{(5.429g)(0.245)} = 5.22\%$

$g = 5.429$
 $W = 2.3580g$
 $W_v = 2.2886g$ % Solids = 24.5

#	Sample ID	Analyte	Reported Concentration (%)	Calculated Concentration (%)	Acceptable (Y/N)
	1	% Solids	22.9	22.9	Y
	2	% Lipids	6.11	6.10	Y*
	3	% Solids	22.9	22.9	Y
	4	% Lipids	3.13	3.10	Y*
	5	% Solids	22	22	Y
	6	% Lipids	5.22	5.22	Y
	7	% Solids	21.8	21.8	Y

Note: _____

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: City of San Diego SWBH Study

LDC Report Date: May 9, 2016

Parameters: Polychlorinated Biphenyls as Congeners

Validation Level: Level IV

Laboratory: Physis Environmental Laboratories Inc.

Sample Delivery Group (SDG): 1504003-002

Sample Identification	Laboratory Sample Identification	Matrix	Collection Date
SWHB-26-M	31963	Tissue	04/22/14
SWHB-27-SBB	31966	Tissue	04/23/14
SWHB-27-P	31969	Tissue	04/23/14
SWHB-30-CH	31973	Tissue	04/23/14
SWHB-06-CH-Small	31989	Tissue	04/22/14
SWHB-06-M	31992	Tissue	04/22/14
SWHB-22-SP	32017	Tissue	04/21/14
SWHB-27-SBBMS	31966MS	Tissue	04/23/14
SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
SWHB-27-SBBDUP	31966DUP	Tissue	04/23/14

Introduction

This Data Validation Report (DVR) presents data validation findings and results for the associated samples listed on the cover page. Data validation was performed in accordance with Final Quality Assurance Project Plan, Regional Harbor Monitoring Program, San Diego, California (August 2013) and a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines (CLPNFG) for Superfund Organic Methods Data Review (June 2008). Where specific guidance was not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The analyses were performed by the following method:

Polychlorinated Biphenyls (PCBs) as Congeners by Environmental Protection Agency (EPA) SW 846 Method 8270D

All sample results were subjected to Level IV data validation, which is comprised of the quality control (QC) summary forms as well as the raw data, to confirm sample quantitation and identification.

The following are definitions of the data qualifiers utilized during data validation:

- J (Estimated): The compound or analyte was analyzed for and positively identified by the laboratory; however the reported concentration is estimated due to non-conformances discovered during data validation.
- U (Non-detected): The compound or analyte was analyzed for and positively identified by the laboratory; however the compound or analyte should be considered non-detected at the reported concentration due to the presence of contaminants detected in the associated blank(s).
- UJ (Non-detected estimated): The compound or analyte was reported as not detected by the laboratory; however the reported quantitation/detection limit is estimated due to non-conformances discovered during data validation.
- R (Rejected): The sample results were rejected due to gross non-conformances discovered during data validation. Data qualified as rejected is not usable.
- NA (Not Applicable): The non-conformance discovered during data validation demonstrates a high bias, while the affected compound or analyte in the associated sample(s) was reported as not detected by the laboratory and did not warrant the qualification of the data.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Qualification Code Reference

- BC The initial calibration (ICAL) curve did not meet method-specified criteria.
- CH High continuing calibration verification (CCV) recovery. Analytical results may be biased high.
- DL The analyte concentration was between the method detection limit (MDL) and the reporting limit (RL).
- FB The analyte was detected in the sample and its associated field blank and the concentration detected in the sample is less than five times the concentration detected in the blanks.
- H Holding time.
- HD Potential analytical imprecision.
- HL High LCS recovery. Analytical results may be biased high.
- HM High MS recovery. Analytical results may be biased high.
- HP High certified reference material (CRM) recovery. Analytical results may be biased high.
- HV High initial calibration verification (ICV) recovery. Analytical results may be biased high.
- LC Low CCV recovery. Analytical result may be biased low.
- LL Low LCS recovery. Analytical result may be biased low.
- LM Low MS recovery. Analytical result may be biased low.
- LP Low CRM recovery. Analytical result may be biased low.
- LS Low Surrogate recovery. Analytical results may be biased low.
- LV Low ICV recovery. Analytical result may be biased low.
- NC Calibration verification standard concentrations were outside the calibration range.
- NQ There is lack of QC for this analyte.
- RB The analyte was detected in the sample and its associated equipment blank and the concentration detected in the sample is less than five times the concentration detected in the blank.
- TD The dissolved metals concentration is significantly higher than the total metal concentration.
- *# Unusual problems found with the data. The number following the asterisk (*) will indicate the section in the validation report where a description of the problem can be found.

I. Sample Receipt and Technical Holding Times

All samples were received in good condition and cooler temperatures upon receipt met validation criteria.

All technical holding time requirements were met with the following exceptions:

Sample	Compound	Total Days From Sample Collection Until Extraction	Required Holding Time (in Days) From Sample Collection Until Extraction	Flag	A or P
SWHB-26-M SWHB-06-CH-Small SWHB-06-M	All TCL compounds	402	365	J (all detects) UJ (all non-detects)	P
SWHB-27-SBB SWHB-27-P SWHB-30-CH	All TCL compounds	401	365	J (all detects) UJ (all non-detects)	P
SWHB-22-SP	All TCL compounds	403	365	J (all detects) UJ (all non-detects)	P

II. GC/MS Instrument Performance Check

A decafluorotriphenylphosphine (DFTPP) tune was performed at the required frequency.

All ion abundance requirements were met.

III. Initial Calibration and Initial Calibration Verification

An initial calibration was performed as required by the method.

A curve fit, based on the initial calibration, was established for quantitation. The coefficient of determination (r^2) was greater than or equal to 0.990 with the following exceptions:

Date	Compound	r^2	Associated Samples	Flag	A or P
05/27/15	PCB-169	0.98733282	All samples in SDG 1504003-002	UJ (all non-detects)	A

Average relative response factors (RRF) for all compounds were within validation criteria.

The percent differences (%D) of the initial calibration verification (ICV) standard were less than or equal to 30.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
06/12/15	PCB-126 PCB-128 PCB-156 PCB-180 PCB-169 PCB-170 PCB-189 PCB-194 PCB-206	41 36 53 35 72 31 74 64 60	All samples in SDG 1504003-002	J (all detects) UJ (all non-detects)	A

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

The percent differences (%D) were less than or equal to 20.0% for all compounds with the following exceptions:

Date	Compound	%D	Associated Samples	Flag	A or P
06/13/15	PCB-126 PCB-128 PCB-177 PCB-156 PCB-169 PCB-170	30 22 26 27 24 25	SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	J (all detects) UJ (all non-detects)	A

All of the continuing calibration relative response factors (RRF) were within validation criteria.

V. Laboratory Blanks

Laboratory blanks were analyzed as required by the method. No contaminants were found in the laboratory blanks.

VI. Field Blanks

No field blanks were identified in this SDG.

VII. Matrix Spike/Matrix Spike Duplicates/Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) sample analysis was performed on an associated project sample. Percent recoveries (%R) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	MS (%R) (Limits)	MSD (%R) (Limits)	Flag	A or P
SWHB-27-SBBMS/MSD (SWHB-27-SBB)	PCB-018	166 (50-150)	250 (50-150)	NA	-
SWHB-27-SBBMS/MSD (SWHB-27-SBB)	PCB-153	33 (50-150)	165 (50-150)	J (all detects)	A
SWHB-27-SBBMS/MSD (SWHB-27-SBB)	PCB-138	-	170 (50-150)	J (all detects)	A

Relative percent differences (RPD) were within QC limits with the following exceptions:

Spike ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-27-SBBMS/MSD (SWHB-27-SBB)	PCB-018	40 (≤ 25)	NA	-
SWHB-27-SBBMS/MSD (SWHB-27-SBB)	PCB-099 PCB-101 PCB-118 PCB-128 PCB-138 PCB-153 PCB-180	41 (≤ 25) 28 (≤ 25) 51 (≤ 25) 28 (≤ 25) 87 (≤ 25) 130 (≤ 25) 40 (≤ 25)	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A

Duplicate (DUP) sample analysis was performed on an associated project sample. Relative percent differences (RPD) were within QC limits with the following exceptions:

DUP ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
SWHB-27-SBBDUP (SWHB-27-SBB)	PCB-028 PCB-044 PCB-070	29 (≤ 25) 41 (≤ 25) 26 (≤ 25)	J (all detects) J (all detects) J (all detects)	A

VIII. Laboratory Control Samples/Certified Reference Materials

Laboratory control samples (LCS) and laboratory control samples duplicates (LCSD) were analyzed as required by the method. Percent recoveries (%R) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	LCS %R (Limits)	LCSD %R (Limits)	Flag	A or P
31945-BS1/BS2 (All samples in SDG 1504003-002)	PCB-018 PCB-028	- -	61 (70-130) 60 (70-130)	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P

Relative percent differences (RPD) were within QC limits with the following exceptions:

LCS ID (Associated Samples)	Compound	RPD (Limits)	Flag	A or P
31945-BS1/BS2 (All samples in SDG 1504003-002)	PCB-018 PCB-028 PCB-044	31 (≤ 25) 30 (≤ 25) 28 (≤ 25)	J (all detects) UJ (all non-detects)	P

Certified reference materials (CRM) were analyzed as required by the method. The results were within QC limits with the following exceptions:

CRM ID	Compound	%R (Limits)	Associated Samples	Flag	A or P
31953-CRM1	PCB-018	59 (60-140)	All samples in SDG 1504003-002	UJ (all non-detects)	P

IX. Field Duplicates

No field duplicates were identified in this SDG.

X. Internal Standards

All internal standard areas and retention times were within QC limits.

XI. Compound Quantitation

All compound quantitations were within validation criteria.

All compounds reported below the reporting limit (RL) and above the minimum detection limit (MDL) were qualified as follows:

Sample	Finding	Flag	A or P
All samples in SDG 1504003-002	Compound reported below the RL and above the MDL	J (all detects)	A

XII. Target Compound Identifications

All target compound identifications were within validation criteria.

XIII. System Performance

The system performance was acceptable.

XIV. Overall Assessment of Data

The analysis was conducted within all specifications of the method. No results were rejected in this SDG.

Due to holding time exceedance, initial calibration r^2 , ICV and continuing calibration %D, MS/MSD %R and RPD, DUP RPD, LCS/LCSD %R and RPD, CRM %R, and results below the RL and above the MDL, data were qualified as estimated in seven samples.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Sample results that were found to be estimated (J) are usable for limited purposes only. Based upon the data validation all other results are considered valid and usable for all purposes.

**City of San Diego SWBH Study
Polychlorinated Biphenyls as Congeners - Data Qualification Summary - SDG
1504003-002**

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-26-M SWHB-06-CH-Small SWHB-06-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-22-SP	All TCL compounds	J (all detects) UJ (all non-detects)	P	Technical holding time (H)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PCB-169	UJ (all non-detects)	A	Initial calibration (r ²) (BC)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PCB-126 PCB-128 PCB-156 PCB-180 PCB-169 PCB-170 PCB-189 PCB-194 PCB-206	J (all detects) UJ (all non-detects)	A	Initial calibration verification (%D) (HV)
SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PCB-126 PCB-128 PCB-177 PCB-156 PCB-169 PCB-170	J (all detects) UJ (all non-detects)	A	Continuing calibration (%D) (CH)
SWHB-27-SBB	PCB-153	J (all detects)	A	Matrix spike/Matrix spike duplicate (%R) (LM)
SWHB-27-SBB	PCB-138	J (all detects)	A	Matrix spike/Matrix spike duplicate (%R) (HM)
SWHB-27-SBB	PCB-099 PCB-101 PCB-118 PCB-128 PCB-138 PCB-153 PCB-180	J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects) J (all detects)	A	Matrix spike/Matrix spike duplicate (RPD) (HD)
SWHB-27-SBB	PCB-028 PCB-044 PCB-070	J (all detects) J (all detects) J (all detects)	A	Duplicate sample analysis (RPD) (HD)

Sample	Compound	Flag	A or P	Reason (Code)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PCB-018 PCB-028	J (all detects) UJ (all non-detects) J (all detects) UJ (all non-detects)	P	Laboratory control samples (%R) (LL)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PCB-018 PCB-028 PCB-044	J (all detects) UJ (all non-detects)	P	Laboratory control samples (RPD) (HD)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	PCB-018	UJ (all non-detects)	P	Certified reference materials (%R) (LP)
SWHB-26-M SWHB-27-SBB SWHB-27-P SWHB-30-CH SWHB-06-CH-Small SWHB-06-M SWHB-22-SP	Compound reported below the RL and above the MDL	J (all detects)	A	Compound quantitation (DL)

**City of San Diego SWBH Study
Polychlorinated Biphenyls as Congeners - Laboratory Blank Data Qualification
Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

**City of San Diego SWBH Study
Polychlorinated Biphenyls as Congeners - Field Blank Data Qualification
Summary - SDG 1504003-002**

No Sample Data Qualified in this SDG

METHOD: GC/MS Polychlorinated Biphenyls as Congeners (EPA SW 846 Method 8270D)

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Sample receipt/Technical holding times	A / M	
II.	GC/MS Instrument performance check	A	
III.	Initial calibration/ICV	M / M	γ^2 ICV $\leq 20/0$
IV.	Continuing calibration	M	CCV $\leq 20/0$
V.	Laboratory Blanks	A	
VI.	Field blanks	N	
VII.	Surrogate spikes	N	
VIII.	Matrix spike/Matrix spike duplicates / DUP	M	
IX.	Laboratory control samples / CRM	M	LOS / 0, CRM
X.	Field duplicates	N	
XI.	Internal standards	A	
XII.	Compound quantitation RL/LOQ/LODs	A	
XIII.	Target compound identification	A	
XIV.	System performance	A	
XV.	Overall assessment of data	A	

Note: A = Acceptable ND = No compounds detected D = Duplicate SB=Source blank
 N = Not provided/applicable R = Rinsate TB = Trip blank OTHER:
 SW = See worksheet FB = Field blank EB = Equipment blank

	Client ID	Lab ID	Matrix	Date
1	SWHB-26-M	31963	Tissue	04/22/14
2	SWHB-27-SBB	31966	Tissue	04/23/14
3	SWHB-27-P	31969	Tissue	04/23/14
4	SWHB-30-CH	31973	Tissue	04/23/14
5	SWHB-06-CH-Small	31989	Tissue	04/22/14
6	SWHB-06-M	31992	Tissue	04/22/14
7	SWHB-22-SP	32017	Tissue	04/21/14
8	SWHB-27-SBBMS	31966MS	Tissue	04/23/14
9	SWHB-27-SBBMSD	31966MSD	Tissue	04/23/14
10	↓ DUP	↓ DUP	↓	↓
11				
12				
13	0-7118/31945-B1			

Method: Semivolatiles (EPA SW 846 Method 8270D)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
Were all technical holding times met?		/		
Was cooler temperature criteria met?	/			
II. GC/MS Instrument performance check				
Were the DFTPP performance results reviewed and found to be within the specified criteria?	/			
Were all samples analyzed within the 12 hour clock criteria?			/	
IIIa. Initial calibration				
Did the laboratory perform a 5 point calibration prior to sample analysis?	/			
Were all percent relative standard deviations (%RSD) ≤ 20% and relative response factors (RRF) within method criteria?			/	
Was a curve fit used for evaluation? If yes, did the initial calibration meet the curve fit acceptance criteria of > 0.990?			/	
IIIb. Initial Calibration Verification				
Was an initial calibration verification standard analyzed after each initial calibration for each instrument?	/			
Were all percent differences (%D) < 30% or percent recoveries (%R) 70-130%?		/		
IV. Continuing calibration				
Was a continuing calibration standard analyzed at least once every 12 hours for each instrument?	/			
Were all percent differences (%D) ≤ 20% and relative response factors (RRF) within method criteria?		/		
V. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	/			
Was a laboratory blank analyzed at least once every 12 hours for each matrix and concentration?	/			
Was there contamination in the laboratory blanks? If yes, please see the Blanks validation completeness worksheet.		/		
VI. Field blanks				
Were field blanks were identified in this SDG?		/		
Were target compounds detected in the field blanks?			/	
VII. Surrogate spikes				
Were all surrogate percent recovery (%R) within QC limits?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis?			/	
If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?			/	

Validation Area	Yes	No	NA	Findings/Comments
VIII. Matrix spike/Matrix spike duplicates				
Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.	/			DUP
Was a MS/MSD analyzed every 20 samples of each matrix?	/			
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?		/		
IX. Laboratory control samples				
Was an LCS analyzed for this SDG?	/			SPW
Was an LCS analyzed per analytical batch?	/			
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?		/		
X. Field duplicates				
Were field duplicate pairs identified in this SDG?		/		
Were target compounds detected in the field duplicates?			/	
XI. Internal standards				
Were internal standard area counts within -50% to +100% of the associated calibration standard?	/			
Were retention times within + 30 seconds of the associated calibration standard?	/			
XII. Compound quantitation				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/			
Were compound quantitation and RLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	/			
XIII. Target compound identification				
Were relative retention times (RRT's) within + 0.06 RRT units of the standard?	/			
Did compound spectra meet specified EPA "Functional Guidelines" criteria?	/			
Were chromatogram peaks verified and accounted for?	/			
XIV. System performance				
System performance was found to be acceptable.	/			
XV. Overall assessment of data				
Overall assessment of data was found to be acceptable.	/			

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Were a matrix spike (MS) and matrix spike duplicate (MSD) analyzed for each matrix in this SDG? If no, indicate which matrix does not have an associated MS/MSD. Soil / Water.

N N/A Was a MS/MSD analyzed every 20 samples of each matrix?

N N/A Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		<u>8/9</u>	<u>PCB018</u>	<u>166 (50-150)</u>	<u>250 (50-150)</u>	()	<u>2 (ND)</u>	<u>↓ det/A (HM)</u>
			<u>153</u>	<u>33 (↓)</u>	<u>165 ()</u>	()	<u>(dets)</u>	<u>↓ N/A (KM)</u>
			<u>138</u>	()	<u>170 (↓)</u>	()	↓	<u>↓ det/A (HM)</u>
			153	()	165 (↓)	()	↓	↓
			<u>018</u>	()	()	<u>40 (≤ 25)</u>	<u>(ND)</u>	<u>(HD)</u>
			<u>099</u>	()	()	<u>41 ()</u>	<u>(dets)</u>	
			<u>101</u>	()	()	<u>28 ()</u>	↓	
			<u>118</u>	()	()	<u>51 ()</u>	↓	
			<u>128</u>	()	()	<u>28 ()</u>	↓	
			<u>138</u>	()	()	<u>87 ()</u>	↓	
			<u>153</u>	()	()	<u>130 ()</u>	↓	
			<u>180</u>	()	()	<u>40 (↓)</u>	↓	
			()	()	()	()		
			()	()	()	()		
		<u>10</u>	<u>PCB028</u>	()	()	<u>29 (≤ 25)</u>	<u>2 (dets)</u>	<u>↓ det/A (HD)</u>
			<u>044</u>	()	()	<u>41 (↓)</u>	↓	↓
			<u>070</u>	()	()	<u>26 (↓)</u>		
			()	()	()	()		
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**VALIDATION FINDINGS WORKSHEET
Laboratory Control Samples (LCS)**

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

N N/A Was a LCS required?

N N/A Were the LCS/LCSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?

#	Date	LCS/LCSD ID	Compound	LCS %R (Limits)	LCSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		31945-BS1	PCB018	()	61 (70-130)	()	all (lots + ND)	✓ (L) / P (L)
		-BS2	028	()	60 (↓)	()		↓
			018	()	()	31 (≤ 25)		(HD)
			028	()	()	30 (↓)		↓
			044	()	()	28 (↓)		↓
				()	()	()		
				()	()	()		
		31953-CRM1	PCB018	59 (60-140)	()	()	all (ND)	✓ (L) / A (L)
				()	()	()		
				()	()	()		
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VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Method: PCB congeners (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	PCB052	1	0.0100	0.0084187	0.00010
		2	0.0250	0.0208292	0.00063
		3	0.0500	0.0374208	0.00250
		4	0.0750	0.0559400	0.00563
		5	0.1000	0.0727386	0.01000
		6	0.2000	0.1473598	0.04000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	0.737762	0.73776
Correlation Coefficient	0.999867	0.99932
Coefficient of Determination (r ²)	0.999733	

Method: PCB congeners (EPA SW 846 Method 8270D)

Calibration Date	Analyte	Standard	(X) Concentration	(Y) Area	(X ²) Conc
5/27/2015	PCB189	1	0.0100	0.0189185	0.00010
		2	0.0250	0.0407605	0.00063
		3	0.0500	0.0848498	0.00250
		4	0.0750	0.1175805	0.00563
		5	0.1000	0.15908	0.01000
		6	0.2000	0.3488099	0.04000

Linear through the origin

	<i>calculated</i>	<i>Reported</i>
Constant	0.000000	0.0000
X Coefficient(s)	1.698202	1.69821
Correlation Coefficient	0.999112	0.99576
Coefficient of Determination (r ²)	0.998225	

VALIDATION FINDINGS WORKSHEET Continuing Calibration Results Verification

METHOD: GC/MS BNA (EPA SW 846 Method 8270D)

The percent difference (%D) of the initial calibration average Relative Response Factors (RRFs) and the continuing calibration RRFs were recalculated for the compounds identified below using the following calculation:

% Difference = 100 * (ave. RRF - RRF)/ave. RRF
 RRF = (A_x)(C_{is})/(A_{is})(C_x)

Where: ave. RRF = initial calibration average RRF
 RRF = continuing calibration RRF
 A_x = Area of compound, A_{is} = Area of associated internal standard
 C_x = Concentration of compound, C_{is} = Concentration of internal standard

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Reported	Recalculated	Reported	Recalculated
					RRF (CC)	RRF (CC)	%D	%D
1	CCV	6/13/15	Phenol (1st internal standard) PCB052	100	112.1763	112.1761	12	12
			Naphthalene (2nd internal standard) 489		113.3914	113.3995	13	13
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
2			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					
3			Phenol (1st internal standard)					
			Naphthalene (2nd internal standard)					
			Fluorene (3rd internal standard)					
			Pentachlorophenol (4th internal standard)					
			Bis(2-ethylhexyl)phthalate (5th internal standard)					
			Benzo(a)pyrene (6th internal standard)					

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET
Matrix Spike/Matrix Spike Duplicates Results Verification

METHOD: GC/MS PAH (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the matrix spike and matrix spike duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SSC - SC)/SA

Where: SSC = Spiked sample concentration
 SA = Spike added

SC = Sample concentration

RPD = | MSC - MSC | * 2 / (MSC + MSCD)

MSC = Matrix spike concentration

MSCD = Matrix spike duplicate concentration

MS/MSD samples: 8/9

Compound	Spike Added (NS/G)		Sample Concentration (NS/G)	Spiked Sample Concentration (NS/G)		Matrix Spike		Matrix Spike Duplicate		MS/MSD	
	MS	MSD		MS	MSD	Percent Recovery		Percent Recovery		RPD	
						Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
PCB28 189	40.04		20.22								
PCB052 189	40.04	40.82	19.41	54.18	58.63	85	87	94	96	10	10
	↓	↓	ND	49.44	45.9	123	123	112	112	9	9

Comments: Refer to Matrix Spike/Matrix Spike Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Laboratory Control Sample/Laboratory Control Sample Duplicates Results Verification

METHOD: GC/MS Semivolatiles (EPA SW 846 Method 8270D)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

% Recovery = 100 * (SC/SA)

Where: SSC = Spike concentration
 SA = Spike added

RPD = | LCSC - LCSDC | * 2 / (LCSC + LCSDC)

LCSC = Laboratory control sample concentration LCSDC = Laboratory control sample duplicate concentration

LCS/LCSD samples: 31945-B31-B32

Compound	Spike Added (1234)		Spike Concentration (1234)		LCS		LCSD		LCS/LCSD	
	LCS	LCSD	LCS	LCSD	Percent Recovery		Percent Recovery		RPD	
					Reported	Recalc	Reported	Recalc	Reported	Recalculated
#B052	100	100	91.59	78.24	92	92	78	78	16	16
↓ 189	↓	↓	122.04	121.43	122	122	121	121	1	1

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicates findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

VALIDATION FINDINGS WORKSHEET

Sample Calculation Verification

METHOD: GC/MS SVOA (EPA SW 846 Method 8270D)

Y N N/A Were all reported results recalculated and verified for all level IV samples?
Y N N/A Were all recalculated results for detected target compounds agree within 10.0% of the reported results?

$$\text{Concentration} = \frac{(A_x)(I_s)(V_i)(DF)(2.0)}{(A_{is})(RRF)(V_o)(V_t)(\%S)}$$

- A_x = Area of the characteristic ion (EICP) for the compound to be measured
- A_{is} = Area of the characteristic ion (EICP) for the specific internal standard
- I_s = Amount of internal standard added in nanograms (ng)
- V_o = Volume or weight of sample extract in milliliters (ml) or grams (g).
- V_i = Volume of extract injected in microliters (ul)
- V_t = Volume of the concentrated extract in microliters (ul)
- Df = Dilution Factor.
- %S = Percent solids, applicable to soil and solid matrices only.
- 2.0 = Factor of 2 to account for GPC cleanup

Example:

Sample I.D. 1, PCB052

$$\text{Conc.} = \frac{1131 \times 1000 \times 1.6033}{(60403)(0.7376)} = 4.069 \text{ ng/g}$$

#	Sample ID	Compound	Reported Concentration (ng/g)	Calculated Concentration (ng/g)	Qualification
		PCB052	4.07		

LDC #: 36197

EDD POPULATION COMPLETENESS WORKSHEET

Date: 5/10/16
Page: 1 of 1
2nd Reviewer: [Signature]

The LDC job number listed above was entered by JE.

EDD Process			Comments/Action
I.	EDD Completeness	-	
Ia.	- All methods present?	Y	
Ib.	- All samples present/match report?	Y	extra samples
Ic.	- All reported analytes present?	Y	
Id.	- 10% or 100% verification of EDD?	Y	
II.	EDD Preparation/Entry	-	
IIa.	- Carryover U/J?	Y	
IIb.	- Reason Codes used? If so, note which codes	Y	Client
IIc.	-Additional Information (QC Level, Validator, Date, Validated Y/N, etc.)	N	
III.	Reasonableness Checks	-	
IIIa.	- Do all qualified ND results have ND qualifier (i.e. UJ)?	Y	
IIIb.	- Do all qualified detect results have detect qualifier (i.e. J)?	Y	
IIIc.	- If reason codes used, do all qualified results have reason code field populated?	Y	
IIId.	-Does the detect flag require changing for blank qualifiers? If so, are all U results marked ND?	NA	
IIIe.	- Do blank concentrations in report match EDD, where data was qualified due to blank?	NA	
IIIf.	- Were any results rejected for overall assessment? If so, were results changed to nonreportable?	NA	
IIIg.	- Is the readme complete? If applicable, were edits or discrepancies listed in the readme?	Y	

Notes: _____

The attached zipped file contains three files:

<u>File</u>	<u>Format</u>	<u>Description</u>
1) Readme_SCCWRP_051016.docx	MS Word 2007	A "Readme" file (this document).
	MS Excel 2007	
2) PHYSIS SCCWRP SWHS 1504003-001 EDD.xlsx		<u>SDG</u> <u>LDC#</u> 1504003-001 36197A
3) PHYSIS SCCWRP SWHS tissue 1504003-002 EDD.xlsx		1504003-002 36197B

Although a 100% verification of the EDD was not performed, LDC observed the following discrepancies between hardcopy data packages and the electronic data deliverables:

SDG/File	Analytical Method	Discrepancy	LDC's approach to the discrepancy
All	All	Additional records are included in the EDD for samples not validated by LDC.	LDC made no changes in the EDD.

Please contact Pei Geng at (760) 827-1100 if you have any questions regarding this electronic data submittal.