APPENDIX D: DATA VALIDATION REPORTS

Attachment D-1: SIM Analyses

LABORATORY DATA CONSULTANTS, INC.
7750 El Camino Real, Suite 2L Carlsbad, CA 92009 Phone: 760/634-0437 Fax: 760/634-0439

LDC #13316
April 13, 2005

Windward Environmental, LLC
200 West Mercer Street, Suite 401
Seattle, WA 98119
ATTN: Ms. Susie McGroddy

SUBJECT: Lower Duwamish Waterway Group Sediment Sample Data Validation

Dear Ms. McGroddy,

Enclosed is our EPA Level II data validation of analytical chemistry results generated in support of the Lower Duwamish Waterway Group project. The analyses were performed by Analytical Resources, Inc. Samples were analyzed for GC/MS Semivolatiles by EPA SW 846 Method 8270C using Selected Ion Monitoring (SIM) for Polynuclear Aromatic Hydrocarbons. Samples are referenced under the following Sample Delivery Group: HV45. See the Sample Analysis Table (Attachment 1) for the number of samples reviewed.

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

Sincerely,

Stella S. Cuenco
Project Manager/Senior Chemist

Job #04-08-06-21

Attachment 1

Level II

LDC #13316 (Windward Environmental, LLC - Seattle WA / Lower Duwamish Waterway Group)

LDC	SDG#	DATE REC'D	DATE DUE	PAHs (8270C -SIM)																															
Matrix:	Tissue/Sediment			T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	W	S	W	S	W	S	W	S	W	S	W	S
A	HV45	03/28/05	04/18/05	0	15																														
Total	B			0	15	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	15

Shaded cells indicate Level IV validation (all other cells are Level II validation). These sample counts do not include MS/MSD, and DUPs

13316ST.wpd

CHEMICAL DATA QUALITY REVIEW FOR BENTHIC SEDIMENT SAMPLES

Lower Duwamish Waterway Group
LDC# 13316

This report details the findings of an EPA Level II data validation review of Analytical chemistry results generated in support of the Lower Duwamish Waterway Group project. The analyses were performed by Analytical Resources, Inc. Samples were analyzed for GC/MS Semivolatiles by EPA SW 846 Method 8270C using Selected Ion Monitoring (SIM) for Polynuclear Aromatic Hydrocarbons. Samples are referenced under the following Sample Delivery Group: HV45. See the Sample Analysis Table (Attachment 1) for the number of samples reviewed and the Sample Validation Table (Attachment 2) for the sample identifications and analyses.

The QC guidelines used for data qualification are those specified in the National Functional Guidelines for Organic Data Review (October 1999). Specific QC criteria used follows the Final Benthic Invertebrate Sampling of the Lower Duwamish Waterway Quality Assurance Project Plan (July 30, 2004). Where specific guidance is not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The following items were evaluated during the review:

- Holding Times
- Sample Preservation
- Cooler Temperatures
- Instrument Calibration*
- Blanks
- Matrix Spike/Matrix Spike Duplicates
- Internal Standards
- Laboratory Control Samples
- Target Compound Identifications*
- Compound Quantitation and CRQLs*
- System Performance
- Field Duplicates

*Data were not reviewed for Level II.

Level II **LDC #13316 (Windward Environmental, LLC - Seattle WA / Lower Duwamish Waterway Group)**

LDC	SDG#	DATE REC'D	DATE DUE	PAHs (8270C -SIM)																													
Matrix: Tissue/Sediment				T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	W	S	W	S	W	S	W	S	W	S
A	HV45	03/28/05	04/18/05	0	15																												
Total	B			0	15	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	15

Shaded cells indicate Level IV validation (all other cells are Level II validation). These sample counts do not include MS/MSD, and DUPs

Attachment 2

SDG#: HV45 VALIDATION SAMPLE TABLE **LDC#:** 13316A

Project Name: Lower Duwamish Waterway Group Parameters/Analytical Method Project #04-08-06-24

Client ID #	Lab ID #	Matrix	Date Collected	SVOA (8270C -SIM)											
LDW-C2-S2	HV45A	sediment	08/26/04	X											
LDW-C3-S1	HV45B	sediment	08/29/04	X											
LDW-C3-S2	HV45C	sediment	08/29/04	X											
LDW-C4-S	HV45D	sediment	08/27/04	X											
LDW-C5-S	HV45E	sediment	08/27/04	X											
LDW-C6-S	HV45F	sediment	08/25/04	X											
LDW-C9-S	HV45G	sediment	08/15/04	X											
LDW-B1a-S	HV45H	sediment	08/12/04	X											
LDW-2Ba-S	HV45I	sediment	08/13/04	X											
LDW-B3a-S	HV45J	sediment	08/26/04	X											
LDW-B10b-S	HV45K	sediment	08/19/04	X											
LDW-B1b-S	HV45L	sediment	09/27/04	X											
LDW-B8b-S	HV45M	sediment	08/19/04	X											
LDW-B9b-S	HV45N	sediment	08/11/04	X											
LDW-B10a-S	HV45O	sediment	08/26/04	X											
LDW-B1a-SMS	HV45HMS	sediment	08/12/04	X											
LDW-B1a-SMSD	HV45HMSD	sediment	08/12/04	X											

Note: X = Validation was performed.

13316VALA.wpd

Only issues which require comment or action are discussed in this report. Data deficiencies are arranged by method. Potential effects of data anomalies have been described where possible.

The following are definitions of the data qualifiers:

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

J Indicates an estimated value.

R Quality control indicates the data is not usable.

N Presumptive evidence of presence of the constituent.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

LDC #: 13238A3
SDG #: DPW615217

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Reviewer: 9
2nd Reviewer: M

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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 RRF Conc (CC)	Recalculated RRF Conc (CC)	Reported %D	Recalculated %D
1	~~PB5C-084 5:1~~	3/17/05	PCB-77 (^{13}C-PCB-77)	0.91	52.1	52.3		4.5
	PB5C-084 5:1		PCB-105 (^{13}C-PCB-105)	0.83	54.0	54.0		7.8
			PCB-156 (^{13}C-PCB-156)	0.98	100	101		0.8
			PCB-180 (^{13}C-PCB-180)	0.79	52.9	53.2		6.4
2	PB5C-074 5:1	12/12/05	PCB-77 (^{13}C-PCB-77)	0.91	47.4	47.3		5.38
			PCB-105 (^{13}C-PCB-105)	0.83	50.4	50.3		0.6
			PCB-156 (^{13}C-PCB-156)	0.98	97.3	97.7		2.3
			PCB-180 (^{13}C-PCB-180)	0.79	51.5	51.8		3.60
3			PCB-77 (^{13}C-PCB-77)					
			PCB-105 (^{13}C-PCB-105)					
			PCB-156 (^{13}C-PCB-156)					
			PCB-180 (^{13}C-PCB-180)					

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

LDC #: 13238A3
SDG #: OPW615217

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Reviewer: 9
2nd reviewer: [illegible]

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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?

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

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)

RRF = Relative response factor of the calibration standard.

V_o = Volume or weight of sample pruged in milliliters (ml) or grams (g).

Df = Dilution factor.

%S = Percent solids, applicable to soils and solid matrices only.

Example:

Sample I.D. 1 , PCBTT:

Conc. = $\frac{(1.99e+07)(2000)(1)}{(9.82e+07)(0.91)(1.44)(\quad)}$

= 309.29 ng/kg.

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

LDC #: 3238A3
SDG #: DPW615217

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Reviewer: 9
2nd Reviewer: 6

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration; SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: OPR

Compound	Spike Added (ng/mL)		Spiked Sample Concentration (ng/mL)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
PCB-77	50	NA	47.4	NA	94.9	94.8				
PCB-81			47.2		94.4	94.4				
PCB-105			51.2		102	102				
PCB-114			51.0		102	102				
PCB-118			52.6		105	105				
PCB-123			51.3		103	103				
PCB-126			50.8		102	102				
PCB-156/PCB157	100		97.1		97.1	97.1				
~~PCB-157~~										
PCB-167	50		49.0		97.9	98				
PCB-169	V		47.6		95.3	95.3				
~~PCB-170~~										
~~PCB-180~~										
PCB-189	50		51.3		103	103				

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

Ions Monitored for HRGC/HRMS Analysis of Polychlorinated Biphenyls

Descriptor	Accurate mass[a]	Ion ID	Analyte	Substance
1	289.9224	M	C12 H6 35Cl4	TCB
	291.9194	M+2	C12 H6 35Cl3 37Cl4	TCB
	301.9626	M	13C12 H6 35Cl4	PeCB
	303.9597	M+2	13C12 H6 35Cl3 37Cl	PeCB
	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
	[292.9825]	Lock	C7 F11	PFK
2	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
	337.9207	M+2	13C12 H5 35Cl4 37Cl	PeCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	PeCB
	359.8415	M+2	C12 H4 35Cl5 37Cl	HxCB
	361.8385	M+4	C12 H4 35Cl4 37Cl2	HxCB
	371.8817	M+2	13C12 H4 35Cl5 37Cl	HxCB
	373.8788	M+4	13C12 H4 35Cl4 37Cl2	HxCB
	393.8025	M+2	C12 H3 35Cl6 37Cl	HpCB
	395.7996	M+4	C12 H3 35Cl5 37Cl2	HpCB
	405.8428	M+2	13C12 H3 35Cl6 37Cl	HpCB
	407.8398	M+4	13C12 H3 35Cl5 37Cl2	HpCB
	[354.9892[	Lock	C9F13	PFK
3	509.7229	M+4	13C12 35Cl10 37Cl2	DCB
	511.7199	M+6	13C12 35Cl9 37Cl3	
	513.7170	M+8	13C12 35Cl8 37Cl4	
	[442.9728]	Lock	C10 F17	PFK

S = internal/recovery standard

H = 1.007825
C = 12.000000
^{13}C = 13.003355
F = 18.9984

^{35}Cl = 34.968853
^{37}Cl = 36.965903

GC/MS Semivolatiles by EPA SW 846 Method 8270C using Selected Ion Monitoring (SIM)

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. GC/MS Instrument Performance Check

Instrument performance check data were not reviewed for Level II.

III. Initial Calibration

Initial calibration data were not reviewed for Level II.

IV. Continuing Calibration

Continuing calibration data were not reviewed for Level II.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No semivolatile contaminants were found in the method blanks with the following exceptions:

Method Blank ID	Extraction Date	Compound TIC (RT in minutes)	Concentration	Associated Samples
MB-031705	3/17/05	Diethylphthalate N-Nitrosodiphenylamine	15 ug/Kg 6.7 ug/Kg	All samples in SDG HV45

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>10X for common contaminants, >5X for other contaminants) than the concentrations found in the associated method blanks with the following exceptions:

Associated SDG	Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
HV45	LDW-C2-S2	Diethylphthalate	6.5 ug/Kg	6.5U ug/Kg
HV45	LDW-B9b-S	Diethylphthalate	6.6 ug/Kg	6.6U ug/Kg

Associated SDG	Sample	Compound TIC (RT in minutes)	Reported Concentration	Modified Final Concentration
HV45	LDW-B10a-S	Diethylphthalate	9.6 ug/Kg	9.6U ug/Kg

VI. Surrogate Spikes

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

Associated SDG	Sample	Surrogate	%R (Limits)	Compound	Flag	A or P
HV45	LDW-B9b-S	2-Fluorobiphenyl 2-Fluorophenol 1,2-Dichlorobenzene-d4 Phenol-d5 2-Chlorophenol-d4 Nitrobenzene-d5	37.2 (40-130) 32.5 (40-130) 24.8 (40-130) 33.1 (40-130) 32.5 (40-130) 30.4 (40-130)	All TCL compounds	J (all detects) UJ (all non-detects)	P

VII. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within QC limits.

VIII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Percent recoveries (%R) were within QC limits.

Standard reference material was performed at the required frequencies.

IX. Regional Quality Assurance and Quality Control

Not applicable.

X. Internal Standards

Internal standards data were not reviewed for Level II.

XI. Target Compound Identifications

Raw data were not reviewed for this SDG.

XII. Compound Quantitation and CRQLs

Raw data were not reviewed for this SDG.

XIII. Tentatively Identified Compounds (TICs)

Raw data were not reviewed for this SDG.

XIV. System Performance

Raw data were not reviewed for this SDG.

XV. Overall Assessment

Data flags have been summarized at the end of the report.

XVI. Field Duplicates

No field duplicates were identified in this SDG.

XVII. Field Blanks

No field blanks were identified in this SDG.

LDC #: 13316A2b **VALIDATION COMPLETENESS WORKSHEET** Date: 3/29/05
SDG #: HV45 Level II Page: 1 of 1
Laboratory: Analytical Resources, Inc. Reviewer: A
2nd Reviewer: X

METHOD: GC/MS ~~Polynuclear Aromatic Hydrocarbons~~ SVOAs (EPA SW 846 Method 8270C-SIM)

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.	Technical holding times	A	Sampling dates: ~~8/26~~ 8/11 - 9/27/04
II.	GC/MS Instrument performance check	N	
III.	Initial calibration	N	
IV.	Continuing calibration	N	
V.	Blanks	SW	
VI.	Surrogate spikes	SW	
VII.	Matrix spike/Matrix spike duplicates	A	
VIII.	Laboratory control samples	A	LCS, SRM
IX.	Regional Quality Assurance and Quality Control	N	
X.	Internal standards	N	
XI.	Target compound identification	N	
XII.	Compound quantitation/CRQLs	N	
XIII.	Tentitatively identified compounds (TICs)	N	
XIV.	System performance	N	
XV.	Overall assessment of data	A	
XVI.	Field duplicates	N	
XVII.	Field blanks	N	

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

Validated Samples
All soils

1	LDW-C2-S2	11	LDW-B10b-S	21	MB-031705	31	
2	LDW-C3-S1	12	LDW-B1b-S	22		32	
3	LDW-C3-S2	13	LDW-B8b-S	23		33	
4	LDW-C4-S	14	LDW-B9b-S	24		34	
5	LDW-C5-S	15	LDW-B10a-S	25		35	
6	LDW-C6-S	16	LDW-B1a-SMS	26		36	
7	LDW-C9-S	17	LDW-B1a-SMSD	27		37	
8	LDW-B1a-S	18		28		38	
9	LDW-2Ba-S	19		29		39	
10	LDW-B3a-S	20		30		40	

VALIDATION FINDINGS WORKSHEET

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

A. Phenol**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenol**	III. Benzo(a)pyrene**
B. Bis (2-chloroethyl) ether	Q. 2,4-Dichlorophenol**	FF. 3-Nitroaniline	UU. Phenanthrene	JJJ. Indeno(1,2,3-cd)pyrene
C. 2-Chlorophenol	R. 1,2,4-Trichlorobenzene	GG. Acenaphthene**	VV. Anthracene	KKK. Dibenz(a,h)anthracene
D. 1,3-Dichlorobenzene	S. Naphthalene	HH. 2,4-Dinitrophenol*	WW. Carbazole	LLL. Benzo(g,h,i)perylene
E. 1,4-Dichlorobenzene**	T. 4-Chloroaniline	II. 4-Nitrophenol*	XX. Di-n-butylphthalate	MMM. Bis(2-Chloroisopropyl)ether
F. 1,2-Dichlorobenzene	U. Hexachlorobutadiene**	JJ. Dibenzofuran	YY. Fluoranthene**	NNN. Aniline
G. 2-Methylphenol	V. 4-Chloro-3-methylphenol**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butylbenzylphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachlorocyclopentadiene*	MM. 4-Chlorophenyl-phenyl ether	BBB. 3,3'-Dichlorobenzidine	QQQ. Benzyl alcohol
J. N-Nitroso-di-n-propylamine*	Y. 2,4,6-Trichlorophenol**	NN. Fluorene	CCC. Benzo(a)anthracene	RRR. Pyridine
K. Hexachloroethane	Z. 2,4,5-Trichlorophenol	OO. 4-Nitroaniline	DDD. Chrysene	SSS. Benzidine
L. Nitrobenzene	AA. 2-Chloronaphthalene	PP. 4,6-Dinitro-2-methylphenol	EEE. Bis(2-ethylhexyl)phthalate	TTT.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	UUU.
N. 2-Nitrophenol**	CC. Dimethylphthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	VVV.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	WWW.

LDC #: 13366b
SDG #: HV45

VALIDATION FINDINGS WORKSHEET
Blanks

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

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 Was a method blank analyzed for each matrix?
(Y) N N/A Was a method blank analyzed for each concentration preparation level?
(Y) N N/A Was a method blank associated with every sample?
(Y) N N/A Was the blank contaminated? If yes, please see qualification below.

Blank extraction date: 3/17/05 **Blank analysis date:** 3/18/05
Conc. units: µg/Kg Associated Samples: All

Compound	Blank ID	Sample Identification								
MB	031705	1	14	15						
2-L	15	6.5/U	6.6/U	9.6/U						
QQ	6.7									

Blank extraction date: _____ **Blank analysis date:** _____
Conc. units: _____ Associated Samples: _____

Compound	Blank ID	Sample Identification								

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
Common contaminants such as the phthalates and TICs noted above that were detected in samples within ten times the associated method blank concentration were qualified as not detected, "U". Other contaminants within five times the method blank concentration were also qualified as not detected, "U".

LDC #: 13316B26
SDG #: HV45

VALIDATION FINDINGS WORKSHEET
Surrogate Recovery

Page: 1 of 1
Reviewer: QA
2nd Reviewer: A

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

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	DCB	38.0 (40-130)	No qual.
			↓	( )	↓
		8	↓	39.2 ( )	
				( )	
		14	FBP	37.2 ( )	Y/N/P
			2FP	32.5 ( )	
			DCB	24.8 ( )	
			PHL	33.1 ( )	
			2CH	32.5 ( )	
			NBZ	30.4 ( )	↓
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	
				( )	

* QC limits are advisory	QC Limits (Soil)	QC Limits (Water)
S1 (NBZ) = Nitrobenzene-d5	23-120	35-114
S2 (FBP) = 2-Fluorobiphenyl	30-115	43-116
S3 (TPH) = Terphenyl-d14	18-137	33-141
S4 (PHL) = Phenol-d5	24-113	10-94
S5 (2FP)= 2-Fluorophenol	25-121	21-100
S6 (TBP) = 2,4,6-Tribromophenol	19-122	10-123
S7 (2CP) = 2-Chlorophenol-d4	20-130*	33-110*
S8 (DCB) = 1,2-Dichlorobenzene-d4	20-130*	16-110*

SUR.2S

Attachment D-2: Tissue Chemistry

CHEMICAL DATA QUALITY REVIEW FOR BENTHIC TISSUE SAMPLES

Lower Duwamish Waterway Group
LDC# 13238

This report details the findings of an EPA Level II and Level IV data validation review of Analytical chemistry results generated in support of the Lower Duwamish Waterway Group project. The analyses were performed by Axys Analytical Services, Ltd. Samples were analyzed for HRGC/HRMS Polychlorinated Biphenyl Congeners by modified EPA Method 1668A. Samples are referenced under the following Sample Delivery Group: DPWG15217. See the Sample Analysis Table (Attachment 1) for the number of samples reviewed and the Sample Validation Table (Attachment 2) for the sample identifications and analyses. Sample IDs ending in "**" underwent Level IV review.

The QC guidelines used for data qualification are those specified in the EPA Region 10 SOP for the Validation of 1668 Toxic, Dioxin-like PCB Data (Revision 1.0, December 8, 1995). Specific QC criteria used follows the Final Benthic Invertebrate Sampling of the Lower Duwamish Waterway Quality Assurance Project Plan (July 30, 2004). Where specific guidance is not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The following items were evaluated during the review:

- Holding Times
- Sample Preservation
- Cooler Temperatures
- Instrument Calibration*
- Blanks
- Matrix Spike/Matrix Spike Duplicates
- Internal Standards
- Laboratory Control Samples
- Target Compound Identifications*
- Compound Quantitation and CRQLs*
- System Performance
- Field Duplicates

*Data were not reviewed for Level II.

Only issues which require comment or action are discussed in this report. Data deficiencies are arranged by method. Potential effects of data anomalies have been described where possible.

The following are definitions of the data qualifiers:

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

J Indicates an estimated value.

- J1 Blank Contamination: Indicates possible high bias and/or false positives.
- J2 Calibration Range exceeded: Indicates possible low bias.
- J3 Holding times not met: Indicates low bias for most analytes.
- J4 Other QC parameters outside control limits: bias not readily determined.
- J5 Other QC parameters outside control limits. The reported results appear to be biased high. The actual value of target compound in the sample may be lower than the value reported by the laboratory.
- J6 Other QC parameters outside control limits. The reported results appear to be biased low. The actual value of target compound in the sample may be higher than the value reported by the laboratory.

R Quality control indicates the data is not usable.

N Presumptive evidence of presence of the constituent.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

***Overall Data Assessment**

Method blank contamination have warranted the qualification of several compounds as non-detected (U).

*The frequency of matrix spike (MS) and matrix spike duplicate (MSD) was not met as required by the QAPP. MS/MSD analyses are not required for EPA Method 1668A. The laboratory consulted with the client on this discrepancy and was instructed to proceed with the extraction and analysis despite the absence of MS/MSDs.

Field duplicates were not collected for this sampling event.

The quality control criteria reviewed, other than those discussed above, were met and are considered acceptable. Based upon the data validation all other results are considered valid and usable for all purposes.

Attachment 1

LDC #13238 (Windward Environmental, LLC - Seattle WA / Lower Duwamish Waterway Group)

LDC	SDG#	DATE REC'D	DATE DUE	PCBs Cong. (1668A)																															
Matrix:	Tissue			T	S	T	S	T	S	T	S	T	S	T	S	T	S	T	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
A	DPWG15217	03/07/05	03/28/05	14	0																														
A	DPWG15217	03/07/05	03/28/05	2	0																														
Total	B			16	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	16

Shaded cells indicate Level IV validation (all other cells are Level II validation). These sample counts do not include MS/MSD, and DUPs

13238ST.wpd

Attachment 2

SDG#: DPWG15217 VALIDATION SAMPLE TABLE **LDC#:** 13238A

Project Name: Lower Duwamish Waterway Group Parameters/Analytical Method Project #04-08-06-21

Client ID #	Lab ID #	Matrix	Date Collected	PCB Cong. (1668A)											
LDW-B1b-T**	L7510-1	tissue	08/10/04	X											
LDW-B2a-T	L7510-2	tissue	08/14/04	X											
LDW-B3b-T	L7510-3	tissue	08/10/04	X											
LDW-B4b-T	L7510-4	tissue	08/17/04	X											
LDW-B5a-T	L7510-5	tissue	08/22/04	X											
LDW-B8a-T	L7510-6	tissue	08/27/04	X											
LDW-B9b-T**	L7510-7	tissue	08/11/04	X											
LDW-B10a-T	L7510-8	tissue	08/25/04	X											
LDW-C1-T	L7510-9	tissue	08/26/04	X											
LDW-C2-2-T	L7510-10	tissue	08/26/04	X											
LDW-C4-T	L7510-11	tissue	08/27/04	X											
LDW-C6-T	L7510-12	tissue	08/26/04	X											
LDW-C7-T1	L7510-13	tissue	08/26/04	X											
LDW-C8-T	L7510-14	tissue	08/26/04	X											
LDW-C9-T	L7510-15	tissue	08/25/04	X											
LDW-C10-T1	L7510-16	tissue	08/25/04	X											
LDW-C10-T1DUP	L7510-16DUP	tissue	08/25/04	X											

Note: X = Validation was performed.

13238VALA.wpd

HRGC/HRMS Polychlorinated Biphenyl Congeners by modified EPA Method 1668A

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. GC/MS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all congeners. The chromatographic resolution was less than or equal to 40% for congeners PCB-23 and PCB-34 and congeners PCB-182 and PCB-187.

The static resolving power was at least 10,000 (10% valley definition).

Instrument performance check data were not reviewed for Level II.

III. Initial Calibration

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for all compounds.

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and greater than or equal to 10 for each recovery and internal standard compound.

Initial calibration data were not reviewed for Level II.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

All of the routine calibration percent differences (%D) between the initial calibration RRF and the routine calibration RRF were less than or equal to 25.0% for unlabeled compounds and less than or equal to 35.0% for labeled compounds.

Continuing calibration data were not reviewed for Level II.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated biphenyl contaminants were found in the method blanks with the following exceptions:

Associated SDG	Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
DPWG15217	WGK743-101	1/27/05	PCB-1	0.215 ng/Kg	All samples in SDG DPWG15217
			PCB-2	0.245 ng/Kg	
			PCB-3	0.355 ng/Kg	
			PCB-4	0.371 ng/Kg	
			PCB-6	0.180 ng/Kg	
			PCB-8	0.704 ng/Kg	
			PCB-11	1.36 ng/Kg	
			PCB-15	0.294 ng/Kg	
			PCB-16	0.344 ng/Kg	
			PCB-17	0.452 ng/Kg	
			PCB-18	0.812 ng/Kg	
			PCB-19	0.128 ng/Kg	
			PCB-20	0.982 ng/Kg	
			PCB-21	0.450 ng/Kg	
			PCB-22	0.276 ng/Kg	
			PCB-25	0.105 ng/Kg	
			PCB-26	0.251 ng/Kg	
			PCB-27	0.095 ng/Kg	
			PCB-31	0.795 ng/Kg	
			PCB-32	0.264 ng/Kg	
			PCB-37	0.233 ng/Kg	
			PCB-40	0.374 ng/Kg	
			PCB-42	0.166 ng/Kg	
			PCB-43	0.038 ng/Kg	
			PCB-44	0.945 ng/Kg	
			PCB-45	0.170 ng/Kg	
			PCB-48	0.162 ng/Kg	
			PCB-49	0.668 ng/Kg	
			PCB-50	0.166 ng/Kg	
			PCB-52	1.40 ng/Kg	
			PCB-56	0.287 ng/Kg	
			PCB-59	0.097 ng/Kg	
			PCB-60	0.109 ng/Kg	
			PCB-61	1.03 ng/Kg	
			PCB-64	0.309 ng/Kg	
			PCB-66	0.563 ng/Kg	
			PCB-77	0.121 ng/Kg	
			PCB-83	0.698 ng/Kg	
			PCB-84	0.226 ng/Kg	
			PCB-85	0.130 ng/Kg	
			PCB-86	0.695 ng/Kg	
			PCB-88	0.216 ng/Kg	
			PCB-90	1.28 ng/Kg	
			PCB-92	0.154 ng/Kg	
			PCB-93	1.20 ng/Kg	
			PCB-105	0.754 ng/Kg	
			PCB-110	1.29 ng/Kg	
			PCB-118	1.41 ng/Kg	
			PCB-128	0.549 ng/Kg	
			PCB-129	2.43 ng/Kg	
			PCB-132	0.513 ng/Kg	
			PCB-135	0.534 ng/Kg	
			PCB-136	0.167 ng/Kg	
			PCB-141	0.337 ng/Kg	
			PCB-146	0.306 ng/Kg	
			PCB-147	0.997 ng/Kg	
			PCB-153	1.80 ng/Kg	
			PCB-156	0.631 ng/Kg	
			PCB-158	0.250 ng/Kg	
			PCB-164	0.107 ng/Kg	
			PCB-167	0.166 ng/Kg	
			PCB-170	0.676 ng/Kg	
			PCB-174	0.371 ng/Kg	

*Indicates change as the result of report review.

Associated SDG	Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
DPWG15217	WGK743-101	1/27/05	PCB-177	0.271 ng/Kg	All samples in SDG DPWG15217
			PCB-179	0.133 ng/Kg	
			PCB-180	1.07 ng/Kg	
			PCB-183	0.381 ng/Kg	
			PCB-187	0.393 ng/Kg	
			PCB-190	0.144 ng/Kg	
			PCB-194	0.188 ng/Kg	
			PCB-195	0.111 ng/Kg	
			PCB-196	0.117 ng/Kg	
			PCB-198	0.235 ng/Kg	
			PCB-201	0.023 ng/Kg	
			PCB-202	0.086 ng/Kg	
			PCB-203	0.151 ng/Kg	
			PCB-204	0.012 ng/Kg	
			PCB-205	0.051 ng/Kg	
			PCB-206	0.149 ng/Kg	
			PCB-208	0.133 ng/Kg	
			PCB-209	0.154 ng/Kg	
			Total Monochloro Biphenyls	0.460 ng/Kg	
			Total Dichloro Biphenyls	1.36 ng/Kg	
			Total Trichloro Biphenyls	4.69 ng/Kg	
			Total Tetrachloro Biphenyls	5.37 ng/Kg	
			Total Pentachloro Biphenyls	6.24 ng/Kg	
			Total Hexchloro Biphenyls	7.47 ng/Kg	
			Total Heptachloro Biphenyls	2.50 ng/Kg	
			Total Octachloro Biphenyls	0.432 ng/Kg	
			Total Nonachloro Biphenyls	0.133 ng/Kg	

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated method blanks with the following exceptions:

Associated SDG	Sample	Compound	Reported Concentration	Modified Final Concentration
DPWG15217	LDW-B2a-T	PCB-2	1.06 ng/Kg	1.06U ng/Kg
		PCB-3	1.60 ng/Kg	1.60U ng/Kg
DPWG15217	LDW-B8a-T	PCB-2	0.895 ng/Kg	0.895U ng/Kg
		PCB-3	1.75 ng/Kg	1.75U ng/Kg
DPWG15217	LDW-B9b-T**	PCB-2	0.259 ng/Kg	0.259U ng/Kg
		PCB-3	0.756 ng/Kg	0.756U ng/Kg
		PCB-11	4.45 ng/Kg	4.45U ng/Kg
		Total Monochloro Biphenyls	1.56 ng/Kg	1.56U ng/Kg
DPWG15217	LDW-B10a-T	PCB-2	0.526 ng/Kg	0.526U ng/Kg
		PCB-3	0.442 ng/Kg	0.442U ng/Kg
		Total Monochloro Biphenyls	1.35 ng/Kg	1.35U ng/Kg
		PCB-1	0.382 ng/Kg	0.382U ng/Kg

*Indicates change as the result of report review.

Associated SDG	Sample	Compound	Reported Concentration	Modified Final Concentration
DPWG15217	LDW-C1-T	PCB-2 PCB-3	0.311 ng/Kg 0.819 ng/Kg	0.311U ng/Kg 0.819U ng/Kg
DPWG15217	LDW-C2-2-T	PCB-2 PCB-3 Total Monochloro Biphenyls PCB-1 PCB-204	0.293 ng/Kg 0.715 ng/Kg 2.01 ng/Kg 1.00 ng/Kg 0.021 ng/Kg	0.293U ng/Kg 0.715U ng/Kg 2.01U ng/Kg 1.00U ng/Kg 0.021U ng/Kg
DPWG15217	LDW-C4-T	PCB-2 PCB-3 Total Monochloro Biphenyls PCB-1 PCB-204	0.316 ng/Kg 0.711 ng/Kg 2.00 ng/Kg 0.971 ng/Kg 0.049 ng/Kg	0.316U ng/Kg 0.711U ng/Kg 2.00U ng/Kg 0.971U ng/Kg 0.049U ng/Kg
DPWG15217	LDW-C6-T	PCB-2 PCB-3	0.847 ng/Kg 1.23 ng/Kg	0.847U ng/Kg 1.23U ng/Kg
DPWG15217	LDW-C7-T1	PCB-2 PCB-3	0.665 ng/Kg 1.24 ng/Kg	0.665U ng/Kg 1.24U ng/Kg
DPWG15217	LDW-C9-T	PCB-2 PCB-3	0.310 ng/Kg 0.781 ng/Kg	0.310U ng/Kg 0.781U ng/Kg
DPWG15217	LDW-C10-T1	PCB-2	0.853 ng/Kg	0.853 U ng/Kg

No field blanks were identified in this SDG.

*VI. Matrix Spike/Matrix Spike Duplicates

*Matrix spike (MS) and matrix spike duplicate (MSD) analyses were not required by the method.

Duplicate (DUP) sample analyses were reviewed for each matrix as applicable. Results were within QC limits.

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

Standard reference material was performed at the required frequencies.

VIII. Regional Quality Assurance and Quality Control

Not applicable.

*Indicates change as the result of report review.

IX. Internal Standards

All internal standard recoveries were within QC limits.

X. Target Compound Identifications

All target compound identifications were within validation criteria.

Target compound identifications data were not reviewed for Level II.

XI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria.

Compound quantitation and CRQLs data were not reviewed for Level II.

XII. System Performance

The system performance was acceptable.

System performance data were not reviewed for Level II.

XIII. Overall Assessment of Data

The overall assessment of data was acceptable.

XIV. Field Duplicates

No field duplicates were identified in this SDG.

LDC #: 13238A3

VALIDATION COMPLETENESS WORKSHEET

Level II / IV

Date: 3/15/05
Page: 1 of 1
Reviewer: [illegible]
2nd Reviewer: [illegible]

SDG #: DPWG15217
Laboratory: AXYS Analytical Services, Ltd.

METHOD: HRGC/HRMS Polychlorinated Biphenyl Congeners (EPA Method 1668A)-Mod

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.	Technical holding times	A	Sampling dates: 8/10 - 27/04
II.	GC/MS Instrument performance check	A	~~TEMP~~ Not reviewed for Level II
III.	Initial calibration	A	%RSD ≤ 20.
IV.	Routine calibration	A	%D ≤ 25/35 (native/label)
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates/DUP	N/A	Not Reqd
VII.	Laboratory control samples	A	OPR. ~~CRL~~
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	Not reviewed for Level II
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	A	
XII.	System performance	A	
XIII.	Overall assessment of data	~~K~~ A	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples: ** Level IV
All Tissues.

1 1	LDW-B1b-T **	11 4/1	LDW-C4-T	21	WG14743-101	31	
2 2/3	LDW-B2a-T	12 4	LDW-C6-T	22		32	
3 2/3	LDW-B3b-T	13 4/2	LDW-C7-T1	23		33	
4 2/3	LDW-B4b-T	14 4/2	LDW-C8-T	24		34	
5 2/3	LDW-B5a-T	15 4/2	LDW-C9-T	25		35	
6 2/3	LDW-B8a-T	16 5/5	LDW-C10-T1	26		36	
7 1	LDW-B9b-T **	17 4/5	LDW-C10-T1DUP	27		37	
8 2	LDW-B10a-T	18		28		38	
9 4	LDW-C1-T	19		29		39	
10 4/1	LDW-C2-2-T	20		30		40	

PCB-16.wpd

LDC #: 132383
SDG #: DPW615217

VALIDATION FINDINGS CHECKLIST

Page: 1 of 2
Reviewer: 9
2nd Reviewer: 8

Method: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS instrument performance check				
Was PFK ~~exact mass 380.9760~~ verified?	✓			
Were the retention time windows established for all homologues?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ 25% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard ≥ 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning of each 12 hour period?	✓			
Were all percent differences (%D) ≤ 40% for unlabeled and labeled standards?	✓			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
Was a method blank associated with every sample in this SDG?	✓			
Was a method blank performed for each matrix and concentration?	✓			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	✓			
VI. 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
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			✓	
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 QC limits?	✓			

LDC #: 132388A3
SDG #: DPWG152I7

VALIDATION FINDINGS CHECKLIST

Reviewer: CT
2nd Reviewer: JL

Validation Area	Yes	No	NA	Findings/Comments
VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		✓		
Were the performance evaluation (PE) samples within the acceptance limits?			✓	
IX. Internal standards				
Were internal standard recoveries within the 25-150% criteria?	✓			
Was the minimum S/N ratio of all internal standard peaks > 10?	✓			
X. Target compound identification				
For polychlorinated biphenyl congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	✓			
For polychlorinated biphenyl congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	✓			
For other polychlorinated biphenyl congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	✓			
Did compound spectra contain all characteristic ions listed in the table attached?	✓			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	✓			
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	✓			
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
XII. System performance				
System performance was found to be acceptable.	✓			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	✓			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		✓		
Target compounds were detected in the field duplicates.			✓	
XV. Field blanks				
Field blanks were identified in this SDG.		✓		
Target compounds were detected in the field blanks.			✓	

LDC #: 13238A3
SDG #: DPWGF1521T

VALIDATION FINDINGS WORKSHEET
Blanks

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

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y N N/A Was method blank contamination less < CRQL for all target compounds?

Blank extraction date: ~~N/A~~ 1/5/05 **Blank analysis date:** 3/3/05 **Associated samples:** All

Conc. units: ~~N/A~~ pg/L

Compound	Blank ID	Sample Identification								
WPK-T43-101		2	6	7	8	9	10	11	12	13
PCB 2	0.249	1.06/U	0.895/U	0.259/U	0.526/U	0.311/U	0.293/U	0.316/U	0.847/U	0.665/U
PCB 3	0.355	1.60/U	1.75/U	0.756/U	0.442/U	0.819/U	0.715/U	0.711/U	1.23/U	1.24/U
PCB 11	1.36			4.45/U						
Total Monochloro Biphenyls	0.460			1.56/U	1.25/U		2.01/U	2.00/U		
PCB 1	0.215				0.382/U		1.00/U	0.971/U		
PCB 204	0.012				~~0.054/U~~		0.021/U	0.049/U		
	↑									
	See attachment									

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All contaminants within five times the method blank concentration were qualified as not detected, "U".

C:\WPDOCS\WRK\PCB\BLANKS.166

LDC #: 132383
SDG #: DPWG152117

VALIDATION FINDINGS WORKSHEET
Blanks

Reviewer: 9
2nd Reviewer: DC

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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

Y N N/A Were all samples associated with a method blank?
Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?
Y N N/A Was method blank contamination less < CRQL for all target compounds?

Blank extraction date: ~~12/6/04~~ 12/7/04 **Blank analysis date:** 2/12/05 **Associated samples:** All

Conc. units: ng/Kg

Compound	Blank ID	Sample Identification								
~~WG#~~ 243-10		~~MF15~~	~~MF16~~							
PCB2	~~0.245~~	0.310/U	0.853/U							
PCB 3	~~0.395~~	0.781/U								
PCB 11	1.36									
Total Monochloro Biphenyls	0.460									
PCB1	0.215									
PCB 204	0.012									
	↑									
See attachment										

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All contaminants within five times the method blank concentration were qualified as not detected, "U".

C:\WPDOCS\WRK\PCB\BLANKS.166

Form 1A
PCB CONGENER ANALYSIS REPORT

CLIENT ID:
LAB BLANK

Lab Name: AXYS ANALYTICAL SERVICES

Contract No.:	4033	Sample Collection:	N/A
Matrix:	CORN OIL	Project Number:	N/A
Sample Receipt Date:	N/A	Lab Sample ID:	WG14743-101
Extraction Date:	27-Jan-2005	Sample Size:	5.00 g
Analysis Date:	12-Feb-2005 Time: 2:26:42	Initial Calibration Date:	04-Feb-2005
Extract Volume (µL):	20	Instrument ID:	HR GC/MS
Injection Volume (µL):	1.0	GC Column ID:	SPB-OCTYL
Dilution Factor:	N/A	Sample Data Filename:	PB5C_074 S:7
Concentration Units :	ng/kg	Blank Data Filename:	PB5C_074 S:7
		Cal. Ver. Data Filename:	PB5C_074 S:1

COMPOUND	IUPAC NO.	5X	CO-ELUTIONS	LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
PCB								
2 - MoCB	1	1.075		JB	0.215	0.0348	3.45	1.001
3 - MoCB	2	1.225		JB	0.245	0.0439	3.42	0.988
4 - MoCB	3	1.775		KJB	0.355	0.0488	3.71	1.000
2,2' - DiCB	4	1.855		KJB	0.371	0.166	1.05	1.001
2,3 - DiCB	5			U		0.150		
2,3' - DiCB	6	0.9		KJB	0.180	0.141	1.61	1.176
2,4 - DiCB	7			U		0.139		
2,4' - DiCB	8	3.52		KJB	0.704	0.133	1.26	1.207
2,5 - DiCB	9			U		0.138		
2,6 - DiCB	10			U		0.137		
3,3' - DiCB	11	6.8		JB	1.36	0.160	1.61	0.969
3,4 - DiCB	12		12 + 13	C U		0.159		
3,4' - DiCB	13		12 + 13	C12				
3,5 - DiCB	14			U		0.162		
4,4' - DiCB	15	1.47		KJB	0.294	0.188	1.14	1.000
2,2',3 - TriCB	16	1.72		JB	0.344	0.0391	1.07	1.166
2,2',4 - TriCB	17	2.26		JB	0.452	0.0355	1.05	1.140
2,2',5 - TriCB	18	4.06	18 + 30	C JB	0.812	0.0289	1.14	1.113
2,2',6 - TriCB	19	0.64		JB	0.128	0.0320	1.04	1.002
2,3,3' - TriCB	20	4.91	20 + 28	C JB	0.982	0.0359	1.02	0.848

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AXYS METHOD MLA-010 Rev 05
Form 1A
Page 2 of 6
Lab Sample ID: WG14743-101

CLIENT ID: LAB BLANK
Project Number: N/A
Sample Data Filename: PB5C_074 S:7

COMPOUND	IUPAC NO.		CO-ELUTIONS	LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
		5X						
2,3,4 - TriCB	21	2.25	21 + 33	C JB	0.450	0.0354	0.95	0.857
2,3,4' - TriCB	22	1.38		JB	0.276	0.0395	1.04	0.871
2,3,5 - TriCB	23			U		0.0367		
2,3,6 - TriCB	24			U		0.0252		
2,3',4 - TriCB	25	0.525		JB	0.105	0.0324	1.00	0.825
2,3',5 - TriCB	26	1.255	26 + 29	C JB	0.251	0.0371	0.96	1.302
2,3',6 - TriCB	27	0.475		JB	0.095	0.0246	1.10	1.151
2,4,4' - TriCB	28		20 + 28	C20				
2,4,5 - TriCB	29		26 + 29	C26				
2,4,6 - TriCB	30		18 + 30	C18				
2,4',5 - TriCB	31	3.975		JB	0.795	0.0362	0.99	0.837
2,4',6 - TriCB	32	1.32		KJB	0.264	0.0338	1.21	1.198
2',3,4 - TriCB	33		21 + 33	C21				
2',3,5 - TriCB	34			U		0.0369		
3,3',4 - TriCB	35			U		0.0436		
3,3',5 - TriCB	36			U		0.0361		
3,4,4' - TriCB	37	1.165		KJB	0.233	0.0451	1.26	1.001
3,4,5 - TriCB	38			U		0.0374		
3,4',5 - TriCB	39			U		0.0373		
2,2',3,3' - TeCB	40	1.87	40 + 41 + 71	C KJB	0.374	0.0261	0.55	1.338
2,2',3,4 - TeCB	41		40 + 41 + 71	C40				
2,2',3,4' - TeCB	42	0.83		JB	0.166	0.0277	0.72	1.313
2,2',3,5 - TeCB	43	0.19		KJB	0.038	0.0308	0.29	1.247
2,2',3,5' - TeCB	44	4.725	44 + 47 + 65	C JB	0.945	0.0243	0.78	1.287
2,2',3,6 - TeCB	45	0.85	45 + 51	C JB	0.170	0.0256	0.80	1.147
2,2',3,6' - TeCB	46			U		0.0302		
2,2',4,4' - TeCB	47		44 + 47 + 65	C44				
2,2',4,5 - TeCB	48	0.81		KJB	0.162	0.0260	0.61	1.275
2,2',4,5' - TeCB	49	3.34	49 + 69	C JB	0.668	0.0231	0.83	1.260
2,2',4,6 - TeCB	50	0.83	50 + 53	C KJB	0.166	0.0247	0.98	1.112
2,2',4,6' - TeCB	51		45 + 51	C45				
2,2',5,5' - TeCB	52	7.0		JB	1.40	0.0251	0.81	1.235
2,2',5,6' - TeCB	53		50 + 53	C50				
2,2',6,6' - TeCB	54			U		0.0157		
2,3,3',4 - TeCB	55			U		0.0650		
2,3,3',4' - TeCB	56	1.435		KJB	0.287	0.0664	1.16	0.904
2,3,3',5 - TeCB	57			U		0.0634		
2,3,3',5' - TeCB	58			U		0.0622		
2,3,3',6 - TeCB	59	0.485	59 + 62 + 75	C KJB	0.097	0.0200	0.48	1.303
2,3,4,4' - TeCB	60	0.545		KJB	0.109	0.0640	1.52	0.911
2,3,4,5 - TeCB	61	5.15	61 + 70 + 74 + 76	C JB	1.03	0.0597	0.66	0.875
2,3,4,6 - TeCB	62		59 + 62 + 75	C59				
2,3,4',5 - TeCB	63			U		0.0611		

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AXYS METHOD MLA-010 Rev 05
Form 1A
Page 3 of 6
Lab Sample ID: WG14743-101

CLIENT ID: LAB BLANK
Project Number: N/A
Sample Data Filename: PB5C_074 S:7

COMPOUND	IUPAC NO.		CO-ELUTIONS	LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
		5X						
PCB 2,3,4',6 - TeCB	64	1.545		JB	0.309	0.0194	0.76	1.350
2,3,5,6 - TeCB	65		44 + 47 + 65	C44				
2,3',4,4' - TeCB	66	2.815		JB	0.563	0.0617	0.71	0.884
2,3',4,5 - TeCB	67			U		0.0578		
2,3',4,5' - TeCB	68			U		0.0615		
2,3',4,6 - TeCB	69		49 + 69	C49				
2,3',4',5 - TeCB	70		61 + 70 + 74 + 76	C61				
2,3',4',6 - TeCB	71		40 + 41 + 71	C40				
2,3',5,5' - TeCB	72			U		0.0646		
2,3',5',6 - TeCB	73			U		0.0192		
2,4,4',5 - TeCB	74		61 + 70 + 74 + 76	C61				
2,4,4',6 - TeCB	75		59 + 62 + 75	C59				
2',3,4,5 - TeCB	76		61 + 70 + 74 + 76	C61				
3,3',4,4' - TeCB	77	0.605		JB	0.121	0.0727	0.85	1.001
3,3',4,5 - TeCB	78			U		0.0683		
3,3',4,5' - TeCB	79			U		0.0548		
3,3',5,5' - TeCB	80			U		0.0616		
3,4,4',5 - TeCB	81			U		0.0671		
2,2',3,3',4 - PeCB	82			U		0.0816		
2,2',3,3',5 - PeCB	83	3.49	83 + 99	C JB	0.698	0.0726	1.74	0.886
2,2',3,3',6 - PeCB	84	1.13		KJB	0.226	0.0805	2.37	1.163
2,2',3,4,4' - PeCB	85	0.65	85 + 116 + 117	C KJB	0.130	0.0625	2.31	0.920
2,2',3,4,5 - PeCB	86	3.475	86 + 87 + 97 + 108 + 119 + 125	C KJB	0.695	0.0618	1.28	0.901
2,2',3,4,5' - PeCB	87		86 + 87 + 97 + 108 + 119 + 125	C86				
2,2',3,4,6 - PeCB	88	1.08	88 + 91	C JB	0.216	0.0694	1.32	1.155
2,2',3,4,6' - PeCB	89			U		0.0749		
2,2',3,4',5 - PeCB	90	6.4	90 + 101 + 113	C JB	1.28	0.0637	1.39	0.869
2,2',3,4',6 - PeCB	91		88 + 91	C88				
2,2',3,5,5' - PeCB	92	0.77		JB	0.154	0.0743	1.49	0.853
2,2',3,5,6 - PeCB	93	6.0	93 + 95 + 98 + 100 + 102	C JB	1.20	0.0686	1.42	1.120
2,2',3,5,6' - PeCB	94			U		0.0742		
2,2',3,5',6 - PeCB	95		93 + 95 + 98 + 100 + 102	C93				
2,2',3,6,6' - PeCB	96			U		0.142		
2,2',3',4,5 - PeCB	97		86 + 87 + 97 + 108 + 119 + 125	C86				
2,2',3',4,6 - PeCB	98		93 + 95 + 98 + 100 + 102	C93				
2,2',4,4',5 - PeCB	99		83 + 99	C83				
2,2',4,4',6 - PeCB	100		93 + 95 + 98 + 100 + 102	C93				
2,2',4,5,5' - PeCB	101		90 + 101 + 113	C90				
2,2',4,5,6' - PeCB	102		93 + 95 + 98 + 100 + 102	C93				
2,2',4,5',6 - PeCB	103			U		0.0652		
2,2',4,6,6' - PeCB	104			U		0.112		
2,3,3',4,4' - PeCB	105	3.77		KJB	0.754	0.0004	2.60	1.001
2,3,3',4,5 - PeCB	106			U		0.0831		

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AXYS METHOD MLA-010 Rev 05
Form 1A
Page 4 of 6
Lab Sample ID: WG14743-101

CLIENT ID: LAB BLANK
Project Number: N/A
Sample Data Filename: PB5C_074 S:7

	COMPOUND	IUPAC NO.	CO-ELUTIONS	LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
		5X						
PCB	2,3,3',4',5 - PeCB	107	107 + 124	C U		0.0842		
	2,3,3',4,5' - PeCB	108	86 + 87 + 97 + 108 + 119 + 125	C86				
	2,3,3',4,6 - PeCB	109		U		0.0820		
	2,3,3',4',6 - PeCB	110 6.45	110 + 115	C JB	1.29	0.0546	1.49	0.925
	2,3,3',5,5' - PeCB	111		U		0.0548		
	2,3,3',5,6 - PeCB	112		U		0.0560		
	2,3,3',5',6 - PeCB	113	90 + 101 + 113	C90				
	2,3,4,4',5 - PeCB	114		U		0.0866		
	2,3,4,4',6 - PeCB	115	110 + 115	C110				
	2,3,4,5,6 - PeCB	116	85 + 116 + 117	C85				
	2,3,4',5,6 - PeCB	117	85 + 116 + 117	C85				
	2,3',4,4',5 - PeCB	118 7.05		JB	1.41	0.0822	1.49	1.001
	2,3',4,4',6 - PeCB	119	86 + 87 + 97 + 108 + 119 + 125	C86				
	2,3',4,5,5' - PeCB	120		U		0.0554		
	2,3',4,5',6 - PeCB	121		U		0.0545		
	2',3,3',4,5 - PeCB	122		U		0.0913		
	2',3,4,4',5 - PeCB	123		U		0.0835		
	2',3,4,5,5' - PeCB	124	107 + 124	C107				
	2',3,4,5,6' - PeCB	125	86 + 87 + 97 + 108 + 119 + 125	C86				
	3,3',4,4',5 - PeCB	126		U		0.102		
	3,3',4,5,5' - PeCB	127		U		0.0905		
	2,2',3,3',4,4' - HxCB	128 2.745	128 + 166	C JB	0.549	0.0889	1.38	0.958
	2,2',3,3',4,5 - HxCB	129 12.15	129 + 138 + 160 + 163	C JB	2.43	0.0843	1.10	0.928
	2,2',3,3',4,5' - HxCB	130		U		0.107		
	2,2',3,3',4,6 - HxCB	131		U		0.0975		
	2,2',3,3',4,6' - HxCB	132 2.565		KJB	0.513	0.0959	1.50	1.175
	2,2',3,3',5,5' - HxCB	133		U		0.0926		
	2,2',3,3',5,6 - HxCB	134	134 + 143	C U		0.0937		
	2,2',3,3',5,6' - HxCB	135 2.67	135 + 151 + 154	C JB	0.534	0.106	1.16	1.103
	2,2',3,3',6,6' - HxCB	136 0.835		JB	0.167	0.0798	1.09	1.024
	2,2',3,4,4',5 - HxCB	137		U		0.0971		
	2,2',3,4,4',5' - HxCB	138	129 + 138 + 160 + 163	C129				
	2,2',3,4,4',6 - HxCB	139	139 + 140	C U		0.0877		
	2,2',3,4,4',6' - HxCB	140	139 + 140	C130				
	2,2',3,4,5,5' - HxCB	141 1.685		KJB	0.337	0.0931	0.73	0.903
	2,2',3,4,5,6 - HxCB	142		U		0.0979		
	2,2',3,4,5,6' - HxCB	143	134 + 143	C134				
	2,2',3,4,5',6 - HxCB	144		U		0.110		
	2,2',3,4,6,6' - HxCB	145		U		0.0796		
	2,2',3,4',5,5' - HxCB	146 1.53		KJB	0.306	0.0881	1.66	0.885
	2,2',3,4',5,6 - HxCB	147 4.985	147 + 149	C JB	0.997	0.0866	1.22	1.133
	2,2',3,4',5,6' - HxCB	148		U		0.111		
	2,2',3,4',5',6 - HxCB	149	147 + 149	C147				

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Approved by: JR QA/QC Chemist

27-02-2005
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AXYS METHOD MLA-010 Rev 05
Form 1A
Page 5 of 6
Lab Sample ID: WG14743-101

CLIENT ID: LAB BLANK
Project Number: N/A
Sample Data Filename: PB5C_074 S:7

COMPOUND	IUPAC NO.		CO-ELUTIONS	LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
		5X						
2,2',3,4',6,6' - HxCB	150			U		0.0787		
2,2',3,5,5',6 - HxCB	151		135 + 151 + 154	C135				
2,2',3,5,6,6' - HxCB	152			U		0.0762		
2,2',4,4',5,5' - HxCB	153	9.0	153 + 168	C JB	1.80	0.0766	1.28	0.899
2,2',4,4',5,6' - HxCB	154		135 + 151 + 154	C135				
2,2',4,4',6,6' - HxCB	155			U		0.0579		
2,3,3',4,4',5 - HxCB	156	3.155	156 + 157	C JB	0.631	0.0930	1.29	1.000
2,3,3',4,4',5' - HxCB	157		156 + 157	C156				
2,3,3',4,4',6 - HxCB	158	1.25		JB	0.250	0.0720	1.12	0.936
2,3,3',4,5,5' - HxCB	159			U		0.0751		
2,3,3',4,5,6 - HxCB	160		129 + 138 + 160 + 163	C129				
2,3,3',4,5',6 - HxCB	161			U		0.0690		
2,3,3',4',5,5' - HxCB	162			U		0.0730		
2,3,3',4',5,6 - HxCB	163		129 + 138 + 160 + 163	C129				
2,3,3',4',5',6 - HxCB	164	0.575		JB	0.107	0.0740	1.12	0.921
2,3,3',5,5',6 - HxCB	165			U		0.0771		
2,3,4,4',5,6 - HxCB	166		128 + 166	C128				
2,3',4,4',5,5' - HxCB	167	0.83		KJB	0.166	0.0664	1.47	1.001
3,3',4,4',5,5' - HxCB	169			U		0.0786		
2,2',3,3',4,4',5 - HpCB	170	3.38		JB	0.676	0.169	1.00	0.936
2,2',3,3',4,4',6 - HpCB	171		171 + 173	C U		0.156		
2,2',3,3',4,5,5' - HpCB	172			U		0.159		
2,2',3,3',4,5,6 - HpCB	173		171 + 173	C171				
2,2',3,3',4,5,6' - HpCB	174	1.855		JB	0.371	0.140	1.02	1.133
2,2',3,3',4,5',6 - HpCB	175			U		0.135		
2,2',3,3',4,6,6' - HpCB	176			U		0.101		
2,2',3,3',4',5,6 - HpCB	177	1.355		KJB	0.271	0.151	1.60	1.145
2,2',3,3',5,5',6 - HpCB	178			U		0.138		
2,2',3,3',5,6,6' - HpCB	179	0.665		KJB	0.133	0.0963	2.25	1.010
2,2',3,4,4',5,5' - HpCB	180	5.35	180 + 193	C JB	1.07	0.130	1.13	0.910
2,2',3,4,4',5,6 - HpCB	181			U		0.142		
2,2',3,4,4',5,6' - HpCB	182			U		0.140		
2,2',3,4,4',5',6 - HpCB	183	1.905	183 + 185	C JB	0.381	0.136	1.15	1.127
2,2',3,4,4',6,6' - HpCB	184			U		0.0927		
2,2',3,4,5,5',6 - HpCB	185		183 + 185	C183				
2,2',3,4,5,6,6' - HpCB	186			U		0.101		
2,2',3,4',5,5',6 - HpCB	187	1.965		KJB	0.393	0.126	1.39	1.110
2,2',3,4',5,6,6' - HpCB	188			U		0.0770		
2,3,3',4,4',5,5' - HpCB	189			U		0.108		
2,3,3',4,4',5,6 - HpCB	190	0.72		KJB	0.144	0.129	0.68	0.947
2,3,3',4,4',5',6 - HpCB	191			U		0.125		
2,3,3',4,5,5',6 - HpCB	192			U		0.127		

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27-02-2005
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AXYS ANALYTICAL SERVICES LTD P.O. BOX 2219, 2045 MILLS RD. WEST, SIDNEY, B.C., CANADA V8L 3S8 TEL (250) 655-5800 FAX (250) 655-5811

AXYS METHOD MLA-010 Rev 05
Form 1A
Page 6 of 6
Lab Sample ID: WG14743-101

CLIENT ID: LAB BLANK
Project Number: N/A
Sample Data Filename: PB5C_074 S:7

COMPOUND	IUPAC NO.		CO-ELUTIONS	LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
		5X						
2,3,3',4',5,5',6 - HpCB	193		180 + 193	C180				
2,2',3,3',4,4',5,5' - OcCB	194	0.94		KJB	0.188	0.0067	1.11	0.991
2,2',3,3',4,4',5,6 - OcCB	195	0.555		JB	0.111	0.0075	0.84	0.945
2,2',3,3',4,4',5,6' - OcCB	196	0.585		KJB	0.117	0.0079	1.35	0.916
2,2',3,3',4,4',6,6' - OcCB	197		197 + 200	C U		0.0052		
2,2',3,3',4,5,5',6 - OcCB	198	1.175	198 + 199	C JB	0.235	0.0079	0.84	1.115
2,2',3,3',4,5,5',6' - OcCB	199		198 + 199	C198				
2,2',3,3',4,5,6,6' - OcCB	200		197 + 200	C197				
2,2',3,3',4,5',6,6' - OcCB	201	0.115		KJB	0.023	0.0053	5.00	1.023
2,2',3,3',5,5',6,6' - OcCB	202	0.43		JB	0.086	0.0051	0.79	1.000
2,2',3,4,4',5,5',6 - OcCB	203	0.755		KJB	0.151	0.0072	1.33	0.920
2,2',3,4,4',5,6,6' - OcCB	204	0.06		KJB	0.012	0.0054	45.99	1.040
2,3,3',4,4',5,5',6 - OcCB	205	0.255		KJB	0.051	0.0061	3.40	1.000
2,2',3,3',4,4',5,5',6 - NoCB	206	0.745		KJB	0.149	0.111	0.96	1.001
2,2',3,3',4,4',5,6,6' - NoCB	207			U		0.0915		
2,2',3,3',4,5,5',6,6' - NoCB	208	0.665		JB	0.133	0.0922	0.82	1.001
2,2',3,3',4,4',5,5',6,6' - DeCB	209	0.77		KJB	0.154	0.0076	0.96	1.001

(1) C = co-eluting congener; U = not detected; K = peak detected, but did not meet quantification criteria, result reported represents the estimated maximum possible concentration; E = exceeds calibrated linear range, see dilution data; D = dilution data; Z = compound not requested; J = concentration less than LMCL; B = analyte found in sample and the associated blank; X = results reported separately

These pages are part of a larger report that may contain information necessary for full data evaluation.

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Approved by: _____ QA/QC Chemist

27-02-2005
dd-mm-yyyy

CLIENT ID:
LAB BLANK

Form 1A
HOMOLOGUE TOTAL POLYCHLORINATED BIPHENYLS (PCB) ANALYSIS REPORT

			Sample Collection:	N/A
Lab Name: AXYS ANALYTICAL SERVICES			Project Number:	N/A
Contract No.:	4033		Lab Sample ID:	WG14743-101
Matrix:	CORN OIL		Sample Size:	5.00 g
Sample Receipt Date:	N/A		Initial Calibration Date:	04-Feb-2005
Extraction Date:	27-Jan-2005		Instrument ID:	HR GC/MS
Analysis Date:	12-Feb-2005	Time: 2:26:42	GC Column ID:	SPB-OCTYL
Extract Volume (µL):	20		Blank Data Filename:	PB5C 074 S:7
Injection Volume (µL):	1.0		Cal. Ver. Data Filename:	PB5C_074 S:1
Dilution Factor:	N/A		Sample Datafile(s):	PB5C_074 S:7
Concentration Units :	ng/kg			

PCB HOMOLOGUE GROUP		LAB FLAG[1]	CONC. FOUND	DETECTION LIMIT
	5X			
Total Monochloro Biphenyls	2.3		0.460	0.0488
Total Dichloro Biphenyls	6.8		1.36	0.188
Total Trichloro Biphenyls	23.45		4.69	0.0451
Total Tetrachloro Biphenyls	26.85		5.37	0.0727
Total Pentachloro Biphenyls	31.2		6.24	0.142
Total Hexachloro Biphenyls	37.35		7.47	0.111
Total Heptachloro Biphenyls	12.5		2.50	0.169
Total Octachloro Biphenyls	2.16		0.432	0.0079
Total Nonachloro Biphenyls	0.665		0.133	0.111
Decachloro Biphenyl		U		0.0076
TOTAL PCBs			~~20.6~~	

(1) U = Not detected
(2) All header information pertains to the initial instrumental analysis of the sample extract.
Additional sample datafiles listed refer to secondary analysis of the sample extract.

These pages are part of a larger report that may contain information necessary for full data evaluation.

14743PCBTOTAL_1.xls, S3

Approved by: _____ QA/QC Chemist

02-03-2005
dd-mm-yyyy

0350

LDC #: 13238A3
SDG #: DPNG15217

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Reviewer: 9
2nd Reviewer: K

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

The Relative Response Factor (RRF), average RRF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following calculations:

$RRF = (A_x)(C_{is})/(A_{is})(C_x)$
average RRF = sum of the RRFs/number of standards
%RSD = 100 * (S/X)

A_x = Area of compound, A_{is} = Area of associated internal standard
C_x = Concentration of compound, C_{is} = Concentration of internal standard
S = Standard deviation of the RRFs, X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported Average RRF (Initial)	Recalculated Average RRF (Initial)	Reported RRF (/ std)	Recalculated RRF (/ std)	Reported %RSD	Recalculated %RSD
1	ICAL2	2/2/05	PCB-77 (^{13}C-PCB-77)	0.91	0.91	0.87	0.86	3.54	3.20
			PCB-105 (^{13}C-PCB-105)	0.83	0.83	0.82	0.82	3.00	3.19
			PCB-156 (^{13}C-PCB-156)	0.98	0.98	0.96	0.96	1.52	1.61
			PCB-180 (^{13}C-PCB-180)	0.79	0.79	0.79	0.79	2.01	1.65
2			PCB-77 (^{13}C-PCB-77)						
			PCB-105 (^{13}C-PCB-105)						
			PCB-156 (^{13}C-PCB-156)						
			PCB-180 (^{13}C-PCB-180)						
3			PCB-77 (^{13}C-PCB-77)						
			PCB-105 (^{13}C-PCB-105)						
			PCB-156 (^{13}C-PCB-158)						
			PCB-180 (^{13}C-PCB-180)						

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

LDC #: 132384A3
SDG #: DPW615217

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

Page: 1 of 1
Reviewer: 9r
2nd Reviewer: M

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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 RRF Conc (CC)	Recalculated RRF Conc (CC)	Reported %D	Recalculated %D
1	~~PBSC-084 5:1~~	2/17/05	PCB-77 (^{13}C-PCB-77)	0.91	52.1	52.3		4.5
	PBSC-084 5:1		PCB-105 (^{13}C-PCB-105)	0.83	54.0	54.0		7.8
			PCB-156 (^{13}C-PCB-156)	0.98	100	101		0.8
			PCB-180 (^{13}C-PCB-180)	0.79	52.9	53.2		6.4
2	PBSC-074 5:1	12/2/05	PCB-77 (^{13}C-PCB-77)	0.91	47.4	47.3		5.38
			PCB-105 (^{13}C-PCB-105)	0.83	50.4	50.3		0.6
			PCB-156 (^{13}C-PCB-156)	0.98	97.3	97.7		2.3
			PCB-180 (^{13}C-PCB-180)	0.79	51.5	51.8		3.60
3			PCB-77 (^{13}C-PCB-77)					
			PCB-105 (^{13}C-PCB-105)					
			PCB-156 (^{13}C-PCB-156)					
			PCB-180 (^{13}C-PCB-180)					

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

C:\WPDOCS\WRK\PCB\CONCLC.166

LDC #: 13238A3
SDG #: DPWGLS217

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Page: 1 of 1
Reviewer: 9
2nd Reviewer: 0

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration; SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: OPR

Compound	Spike Added (ng/mL)		Spiked Sample Concentration (ng/mL)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
PCB-77	50	NA	47.4	NA	94.9	94.8				
PCB-81	↓	↓	47.2	↓	94.4	94.4				
PCB-105			51.2		102	102				
PCB-114			51.0		102	102				
PCB-118			52.6		105	105				
PCB-123			51.3		103	103				
PCB-126	↓		50.8		102	102				
PCB-156/PCB157	100		97.1		97.1	97.1				
~~PCB-157~~										
PCB-167	50		49.0		97.9	98				
PCB-169	↓		47.6		95.3	95.3				
~~PCB-170~~										
~~PCB-180~~										
PCB-189	50	↓	51.3	↓	103	103				

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

LCSCLC.16A

Ions Monitored for HRGC/HRMS Analysis of Polychlorinated Biphenyls

Descriptor	Accurate mass[a]	Ion ID	Analyte	Substance
1	289.9224	M	C12 H6 35Cl4	TCB
	291.9194	M+2	C12 H6 35Cl3 37Cl4	TCB
	301.9626	M	13C12 H6 35Cl4	PeCB
	303.9597	M+2	13C12 H6 35Cl3 37Cl	PeCB
	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
	[292.9825]	Lock	C7 F11	PFK
2	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
	337.9207	M+2	13C12 H5 35Cl4 37Cl	PeCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	PeCB
	359.8415	M+2	C12 H4 35Cl5 37Cl	HxCB
	361.8385	M+4	C12 H4 35Cl4 37Cl2	HxCB
	371.8817	M+2	13C12 H4 35Cl5 37Cl	HxCB
	373.8788	M+4	13C12 H4 35Cl4 37Cl2	HxCB
	393.8025	M+2	C12 H3 35Cl6 37Cl	HpCB
	395.7996	M+4	C12 H3 35Cl5 37Cl2	HpCB
	405.8428	M+2	13C12 H3 35Cl6 37Cl	HpCB
	407.8398	M+4	13C12 H3 35Cl5 37Cl2	HpCB
	[354.9892[	Lock	C9F13	PFK
3	509.7229	M+4	13C12 35Cl10 37Cl2	DCB
	511.7199	M+6	13C12 35Cl9 37Cl3	
	513.7170	M+8	13C12 35Cl8 37Cl4	
	[442.9728]	Lock	C10 F17	PFK

S = interral/recovery standard

H = 1.007825
C = 12.000000
^{13}C = 13.003355
F = 18.9984

^{35}Cl = 34.968853
^{37}Cl = 36.965903

C:\WPDOCS\WRK\PCB\TCI.16A

LDC #: 13238A3
SDG #: OPW615217

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Reviewer: 9
2nd reviewer: 8

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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?

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

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)

RRF = Relative response factor of the calibration standard.

V_o = Volume or weight of sample pruged in milliliters (ml) or grams (g).

Df = Dilution factor.

%S = Percent solids, applicable to soils and solid matrices only.

Example:

Sample I.D. 1 , PCBTT:

Conc. = $\frac{(1.99e+07)(2000)(1)}{(9.82e+07)(0.91)(1.44)(1)}$

= 309.29 ng/kg.

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

Attachment D-3: Sediment Chemistry

CHEMICAL DATA QUALITY REVIEW FOR BENTHIC SEDIMENT SAMPLES

Lower Duwamish Waterway Group
LDC# 13247

This report details the findings of an EPA Level II and Level IV data validation review of Analytical chemistry results generated in support of the Lower Duwamish Waterway Group project. The analyses were performed by Axys Analytical Services, Ltd. Samples were analyzed for HRGC/HRMS Polychlorinated Biphenyl Congeners by modified EPA Method 1668A. Samples are referenced under the following Sample Delivery Group: DPWG15252. See the Sample Analysis Table (Attachment 1) for the number of samples reviewed and the Sample Validation Table (Attachment 2) for the sample identifications and analyses. Sample IDs ending in "**" underwent Level IV review.

The QC guidelines used for data qualification are those specified in the EPA Region 10 SOP for the Validation of 1668 Toxic, Dioxin-like PCB Data (Revision 1.0, December 8, 1995). Specific QC criteria used follows the Final Benthic Invertebrate Sampling of the Lower Duwamish Waterway Quality Assurance Project Plan (July 30, 2004). Where specific guidance is not available, the data has been evaluated in a conservative manner consistent with industry standards using professional experience.

The following items were evaluated during the review:

- Holding Times
- Sample Preservation
- Cooler Temperatures
- Instrument Calibration*
- Blanks
- Matrix Spike/Matrix Spike Duplicates
- Internal Standards
- Laboratory Control Samples
- Target Compound Identifications*
- Compound Quantitation and CRQLs*
- System Performance
- Field Duplicates

*Data were not reviewed for Level II.

Only issues which require comment or action are discussed in this report. Data deficiencies are arranged by method. Potential effects of data anomalies have been described where possible.

The following are definitions of the data qualifiers:

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.

J Indicates an estimated value.

- J1 Blank Contamination: Indicates possible high bias and/or false positives.
- J2 Calibration Range exceeded: Indicates possible low bias.
- J3 Holding times not met: Indicates low bias for most analytes.
- J4 Other QC parameters outside control limits: bias not readily determined.
- J5 Other QC parameters outside control limits. The reported results appear to be biased high. The actual value of target compound in the sample may be lower than the value reported by the laboratory.
- J6 Other QC parameters outside control limits. The reported results appear to be biased low. The actual value of target compound in the sample may be higher than the value reported by the laboratory.

R Quality control indicates the data is not usable.

N Presumptive evidence of presence of the constituent.

UJ Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

A Indicates the finding is based upon technical validation criteria.

P Indicates the finding is related to a protocol/contractual deviation.

***Overall Data Assessment**

Laboratory duplicate precision exceedances have warranted the qualification of detected results as estimated (J).

*The frequency of matrix spike (MS) and matrix spike duplicate (MSD) was not met as required by the QAPP. MS/MSD analyses are not required for EPA Method 1668A. The laboratory consulted with the client on this discrepancy and was instructed to proceed with the extraction and analysis despite the absence of MS/MSDs.

Field duplicates were not collected for this sampling event.

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.

Attachment 1

LDC #13247 (Windward Environmental, LLC - Seattle WA / Lower Duwamish Waterway Group)

LDC	SDG#	DATE REC'D	DATE DUE	PCBs Cong. (1668A)																															
Matrix:	Tissue/Sediment			T	S	T	S	T	S	T	S	T	S	T	S	T	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S	W	S
A	DPWG15252	03/11/05	04/01/05	0	14																														
A	DPWG15252	03/11/05	04/01/05	0	2																														
Total	B			0	16	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	16

Shaded cells indicate Level IV validation (all other cells are Level II validation). These sample counts do not include MS/MSD, and DUPs

13247ST.wpd

Attachment 2

SDG#: DPWG15252 VALIDATION SAMPLE TABLE **LDC#:** 13247A

Project Name: Lower Duwamish Waterway Group Parameters/Analytical Method Project #04-08-06-21

Client ID #	Lab ID #	Matrix	Date Collected	PCB Cong. (1668A)										
LDW-B1b-S	L7505-1	sediment	08/10/04	X										
LDW-B2a-S	L7505-2	sediment	08/14/04	X										
LDW-B3b-S	L7505-3	sediment	08/10/04	X										
LDW-B4b-S	L7505-4	sediment	08/17/04	X										
LDW-B5a-S	L7505-5	sediment	08/22/04	X										
LDW-B8a-S	L7505-6	sediment	08/27/04	X										
LDW-B9b-S	L7505-7	sediment	08/11/04	X										
LDW-B10a-S**	L7505-8	sediment	08/25/04	X										
LDW-C1-S**	L7505-9	sediment	08/26/04	X										
LDW-C2-S2	L7505-10	sediment	08/26/04	X										
LDW-C4-S	L7505-11	sediment	08/27/04	X										
LDW-C6-S	L7505-12	sediment	08/26/04	X										
LDW-C7-S-1	L7505-13	sediment	08/26/04	X										
LDW-C8-S	L7505-14	sediment	08/26/04	X										
LDW-C9-S	L7505-15	sediment	08/25/04	X										
LDW-C10-S-1	L7505-16	sediment	08/25/04	X										
LDW-B1b-SDUP	L7505-1DUP	sediment	08/10/04	X										

Note: X = Validation was performed.

13247VALA.wpd

HRGC/HRMS Polychlorinated Biphenyl Congeners by modified EPA Method 1668A

I. Technical Holding Times

All technical holding time requirements were met.

The chain-of-custodies were reviewed for documentation of cooler temperatures. All cooler temperatures met validation criteria.

II. GC/MS Instrument Performance Check

Instrument performance was checked at the required daily frequency.

Retention time windows were established for all congeners. The chromatographic resolution was less than or equal to 40% for congeners PCB-23 and PCB-34 and congeners PCB-182 and PCB-187.

The static resolving power was at least 10,000 (10% valley definition).

Instrument performance check data were not reviewed for Level II.

III. Initial Calibration

A five point initial calibration was performed as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0% for compounds.

The minimum S/N ratio for each target compound was greater than or equal to 2.5 and greater than or equal to 10 for each recovery and internal standard compound.

Initial calibration data were not reviewed for Level II.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequencies.

All of the routine calibration percent differences (%D) between the initial calibration RRF and the routine calibration RRF were less than or equal to 25.0% for unlabeled compounds and less than or equal to 35.0% for labeled compounds.

Continuing calibration data were not reviewed for Level II.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No polychlorinated biphenyl contaminants were found in the method blanks with the following exceptions:

Associated SDG	Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
DPWG15252	WG14745-101	1/25/05	PCB-66	0.108 ng/Kg	All samples in SDG DPWG15252
			PCB-77	0.042 ng/Kg	
			PCB-99+101+113	0.236 ng/Kg	
			PCB-105	0.078 ng/Kg	
			PCB-110+115	0.289 ng/Kg	
			PCB-114	0.044 ng/Kg	
			PCB-118	0.195 ng/Kg	
			PCB-123	0.027 ng/Kg	
			PCB-126	0.041 ng/Kg	
			PCB-129+138+160+163	0.310 ng/Kg	
			PCB-153+168	0.257 ng/Kg	
			PCB-156+157	0.088 ng/Kg	
			PCB-180+193	0.160 ng/Kg	

Sample concentrations were compared to concentrations detected in the method blanks. The sample concentrations were either not detected or were significantly greater (>5X blank contaminants) than the concentrations found in the associated method blanks.

No field blanks were identified in this SDG.

*VI. Matrix Spike/Matrix Spike Duplicates

Matrix spike (MS) and matrix spike duplicate (MSD) analyses were not required by the method.

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

Associated SDG	DUP ID (Associated Samples)	Compounds	RPD (Limits)	Flag	A or P
DPWG15252	LDW-B1b-SDUP (LDW-B1b-S)	PCB-99+101+113	53.7 (≤50)	J4 (all detects)	A
		PCB-129+138+160+163	75.6 (≤50)	J4 (all detects)	
		PCB-153+168	89.5 (≤50)	J4 (all detects)	
		PCB-156+157	60.8 (≤50)	J4 (all detects)	
		PCB-167	59.9 (≤50)	J4 (all detects)	
		PCB-180+193	112 (≤50)	J4 (all detects)	
		PCB-189	91.6 (<50)	J4 (all detects)	

VII. Laboratory Control Samples (LCS)

Laboratory control samples were reviewed for each matrix as applicable. The percent recoveries (%R) were within the QC limits.

Standard reference material was performed at the required frequencies.

VIII. Regional Quality Assurance and Quality Control

Not applicable.

IX. Internal Standards

All internal standard recoveries were within QC limits.

X. Target Compound Identifications

All target compound identifications were within validation criteria.

Target compound identifications data were not reviewed for Level II.

XI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria.

Compound quantitation and CRQLs data were not reviewed for Level II.

XII. System Performance

The system performance was acceptable.

System performance data were not reviewed for Level II.

*Indicates change as the result of report review.

XIII. Overall Assessment of Data

The overall assessment of data was acceptable.

XIV. Field Duplicates

No field duplicates were identified in this SDG.

LDC #: 13247A3 **VALIDATION COMPLETENESS WORKSHEET** Date: 3/16/05
SDG #: DPWG15252 Level II/IV Page: 1 of 1
Laboratory: AXYS Analytical Services, Ltd. Reviewer: A
2nd Reviewer: [illegible]

METHOD: HRGC/HRMS Polychlorinated Biphenyl Congeners (EPA Method 1668A)

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.	Technical holding times	A	Sampling dates: 8/10 - 27/04
II.	GC/MS Instrument performance check	A	NR for level I
III.	Initial calibration	A	%RSD ≤ 20
IV.	Routine calibration	A	%D ≤ 25/35.
V.	Blanks	SW	
VI.	Matrix spike/Matrix spike duplicates /DUP	N SW	not rq'd
VII.	Laboratory control samples	A	OPR. SRM
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	A	NR for level I
X.	Target compound identifications	A	
XI.	Compound quantitation and CRQLs	A	
XII.	System performance	A	
XIII.	Overall assessment of data	A SW	
XIV.	Field duplicates	N	
XV.	Field blanks	N	

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

Validated Samples:
MI seals

1	LDW-B1b-S	11	LDW-C4-S	21	NG14T45-101	31	
2	LDW-B2a-S	12	LDW-C6-S	22		32	
3	LDW-B3b-S	13	LDW-C7-S-1	23		33	
4	LDW-B4b-S	14	LDW-C8-S	24		34	
5	LDW-B5a-S	15	LDW-C9-S	25		35	
6	LDW-B8a-S	16	LDW-C10-S-1	26		36	
7	LDW-B9b-S	17	LDW-B1b-SDUP	27		37	
8	LDW-B10a-S **	18		28		38	
9	LDW-C1-S **	19		29		39	
10	LDW-C2-S2	20		30		40	

LDC #: 13247A3
SDG #: DPWE1525

VALIDATION FINDINGS CHECKLIST

Page: 1 of 3-2
Reviewer: 9
2nd Reviewer: JK

Method: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

Validation Area	Yes	No	NA	Findings/Comments
I. Technical holding times				
All technical holding times were met.	✓			
Cooler temperature criteria was met.	✓			
II. GC/MS instrument performance check				
Was PFK ~~exact mass 380.9760~~ verified?	✓			
Were the retention time windows established for all homologues?	✓			
Is the static resolving power at least 10,000 (10% valley definition)?	✓			
Was the mass resolution adequately check with PFK?	✓			
III. Initial calibration				
Was the initial calibration performed at 5 concentration levels?	✓			
Were all percent relative standard deviations (%RSD) ≤ ~~25%~~ 20% for unlabeled standards and ≤ 30% for labeled standards?	✓			
Did all calibration standards meet the Ion Abundance Ratio criteria?	✓			
Was the signal to noise ratio for each target compound ≥ 2.5 and for each recovery and internal standard > 10?	✓			
IV. Continuing calibration				
Was a routine calibration performed at the beginning of each 12 hour period?	✓			
Were all percent differences (%D) < ~~40%~~ 35 for unlabeled and labeled standards?	✓			
Did all routine calibration standards meet the Ion Abundance Ratio criteria?	✓			
V. Blanks				
Was a method blank associated with every sample in this SDG?	✓			
Was a method blank performed for each matrix and concentration?	✓			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.	✓			
VI. 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
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?			✓	
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 QC limits?	✓			

LDC #: 13247A3
SDG #: DPNG15252

VALIDATION FINDINGS CHECKLIST

Reviewer: Q
2nd Reviewer: [illegible]

Validation Area	Yes	No	NA	Findings/Comments
VIII. Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?		✓		
Were the performance evaluation (PE) samples within the acceptance limits?			✓	
IX. Internal standards				
Were internal standard recoveries within the 25-150% criteria?	✓			
Was the minimum S/N ratio of all internal standard peaks > 10?	✓			
X. Target compound identification				
For polychlorinated biphenyl congeners with associated labeled standards, were the retention times of the two quantitation peaks within -1 to 3 sec. of the RT of the labeled standard?	✓			
For polychlorinated biphenyl congeners without associated labeled standards, were the relative retention times of the two quantitation peaks within 0.005 time units of the RRT measured in the routine calibration?	✓			
For other polychlorinated biphenyl congeners, were the retention times of the two quantitation peaks within RT established in the performance check solution?	✓			
Did compound spectra contain all characteristic ions listed in the table attached?	✓			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	✓			
Was the signal to noise ratio for each target compound and labeled standard > 2.5?	✓			
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?	✓			
Was an acceptable lock mass recorded and monitored?	✓			
XI. Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	✓			
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?	✓			
XII. System performance				
System performance was found to be acceptable.	✓			
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.	✓			
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.		✓		
Target compounds were detected in the field duplicates.			✓	
XV. Field blanks				
Field blanks were identified in this SDG.		✓		
Target compounds were detected in the field blanks.			✓	

Ions Monitored for HRGC/HRMS Analysis of Polychlorinated Biphenyls

Descriptor	Accurate mass[a]	Ion ID	Analyte	Substance
1	289.9224	M	C12 H6 35Cl4	TCB
	291.9194	M+2	C12 H6 35Cl3 37Cl4	TCB
	301.9626	M	13C12 H6 35Cl4	PeCB
	303.9597	M+2	13C12 H6 35Cl3 37Cl	PeCB
	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
	[292.9825]	Lock	C7 F11	PFK
2	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
	337.9207	M+2	13C12 H5 35Cl4 37Cl	PeCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	PeCB
	359.8415	M+2	C12 H4 35Cl5 37Cl	HxCB
	361.8385	M+4	C12 H4 35Cl4 37Cl2	HxCB
	371.8817	M+2	13C12 H4 35Cl5 37Cl	HxCB
	373.8788	M+4	13C12 H4 35Cl4 37Cl2	HxCB
	393.8025	M+2	C12 H3 35Cl6 37Cl	HpCB
	395.7996	M+4	C12 H3 35Cl5 37Cl2	HpCB
	405.8428	M+2	13C12 H3 35Cl6 37Cl	HpCB
	407.8398	M+4	13C12 H3 35Cl5 37Cl2	HpCB
	[354.9892[	Lock	C9F13	PFK
3	509.7229	M+4	13C12 35Cl10 37Cl2	DCB
	511.7199	M+6	13C12 35Cl9 37Cl3	
	513.7170	M+8	13C12 35Cl8 37Cl4	
	[442.9728]	Lock	C10 F17	PFK

S = internal/recovery standard

H = 1.007825
C = 12.000000
^{13}C = 13.003355
F = 18.9984

^{35}Cl = 34.968853
^{37}Cl = 36.965903

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LDC #: 13247A3
SDG #: DPW61525-2

VALIDATION FINDINGS WORKSHEET
Blanks

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

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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

Y N N/A Were all samples associated with a method blank?

Y N N/A Was a method blank performed for each matrix and whenever a sample extraction was performed?

Y N N/A Was method blank contamination less < CRQL for all target compounds?

Blank extraction date: 1/25/05 **Blank analysis date:** 2/8/05 **Associated samples:** All

Conc. units: pg/L

Compound	Blank ID	Sample Identification								
WF14745-101		All								
PCB 66	0.108									
TT	0.042	> 5X								
90+101+113	0.336									
105	0.078									
110+115	~~0.090~~ 0.289									
114	0.044									
118	0.195									
123	0.027									
126	0.041									
129+138+160+163	0.310									
153+168	0.257									
156+157	0.088									
180+193	0.160									

CIRCLED RESULTS WERE NOT QUALIFIED. ALL RESULTS NOT CIRCLED WERE QUALIFIED BY THE FOLLOWING STATEMENT:
All contaminants within five times the method blank concentration were qualified as not detected, "U".

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LDC #: 13247A3
SDG #: DPWG15252

VALIDATION FINDINGS WORKSHEET

Matrix Spike/Matrix Spike Duplicates

Page: 1 of 1
Reviewer: Q
2nd Reviewer: 4

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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 concentrations and the relative percent differences (RPD) within the QC limits stated below?

#	Date	MS/MSD ID	Compound	MS %R (Limits)	MSD %R (Limits)	RPD (Limits)	Associated Samples	Qualifications
		1/17	PCB 90+101+113	( )	( )	53.7 (≤50)	1	J / dets / A
			PCB 129+138+160+163	( )	( )	75.6 ( )		↓
			PCB 153+168	( )	( )	89.5 ( )		
			PCB 156+157	( )	( )	60.8 ( )		
			PCB 167	( )	( )	59.9 ( )		
			PCB 180+193	( )	( )	112 ( )		
			PCB 189	( )	( )	91.6 (↓)		↓
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
				( )	( )	( )		
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				( )	( )	( )		

MSD.16A

LDC #: 13347A3
SDG #: JPJW615252

VALIDATION FINDINGS WORKSHEET
Initial Calibration Calculation Verification

Page: 1 of 1
Reviewer:
2nd Reviewer:

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

The Relative Response Factor (RRF), average RRF, and percent relative standard deviation (%RSD) were recalculated for the compounds identified below using the following calculations:

$RRF = (A_x)(C_{is})/(A_{is})(C_x)$
average RRF = sum of the RRFs/number of standards
$\%RSD = 100 * (S/X)$

A_x = Area of compound, A_{is} = Area of associated internal standard
C_x = Concentration of compound, C_{is} = Concentration of internal standard
S = Standard deviation of the RRFs, X = Mean of the RRFs

#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Reported Average RRF (initial)	Recalculated Average RRF (initial)	Reported RRF (/ std)	Recalculated RRF (/ std)	Reported %RSD	Recalculated %RSD
1	ICAL	2/1/05	PCB-77 (^{13}C-PCB-77)	0.91	0.91	0.87	0.86	3.54	3.20
			PCB-105 (^{13}C-PCB-105)	0.83	0.83	0.82	0.82	3.00	3.19
			PCB-156 (^{13}C-PCB-156)	0.98	0.98	0.96	0.96	1.52	1.61
			PCB-180 (^{13}C-PCB-180)	0.79	0.79	0.79	0.79	2.01	1.65
2			PCB-77 (^{13}C-PCB-77)						
			PCB-105 (^{13}C-PCB-105)						
			PCB-156 (^{13}C-PCB-156)						
			PCB-180 (^{13}C-PCB-180)						
3			PCB-77 (^{13}C-PCB-77)						
			PCB-105 (^{13}C-PCB-105)						
			PCB-156 (^{13}C-PCB-156)						
			PCB-180 (^{13}C-PCB-180)						

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

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LDC #: 13247A3
SDG #: DPW615252

VALIDATION FINDINGS WORKSHEET
Routine Calibration Results Verification

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

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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 RRF (CC)	Recalculated RRF (CC)	Reported %D*	Recalculated %D
1	PBSC-069	2/8/05	PCB-77 (^{13}C-PCB-77)	0.91	48.3	48.2		3.6
			PCB-105 (^{13}C-PCB-105)	0.83	49.3	49.2		1.6
			PCB-156 (^{13}C-PCB-156)	0.98	98.6	98.5		1.5
			PCB-180 (^{13}C-PCB-180)	0.79	49.8	50.1		0.2
2	PBSC-069	2/9/05	PCB-77 (^{13}C-PCB-77)	0.91	48.8	48.7		2.6
			PCB-105 (^{13}C-PCB-105)	0.83	50.5	50.2		0.4
			PCB-156 (^{13}C-PCB-156)	0.98	100	100		0
			PCB-180 (^{13}C-PCB-180)	0.79	49.2	49.6		0.7
3			PCB-77 (^{13}C-PCB-77)					
			PCB-105 (^{13}C-PCB-105)					
			PCB-156 (^{13}C-PCB-156)					
			PCB-180 (^{13}C-PCB-180)					

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

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LDC #: 13247A3
SDG #: DPW615252

VALIDATION FINDINGS WORKSHEET
Laboratory Control Sample Results Verification

Reviewer: 2nd Reviewer:

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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

% Recovery = 100 * SSC/SA Where: SSC = Spiked sample concentration; SA = Spike added

RPD = | LCS - LCSD | * 2/(LCS + LCSD) LCS = Laboraotry control sample percent recovery LCSD = Laboratory control sample duplicate percent recovery

LCS ID: OPR

Compound	Spike Added (ng/mL)		Spiked Sample Concentration (ng/mL)		LCS Percent Recovery		LCSD Percent Recovery		LCS/LCSD RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
PCB-77	50.0	NA	48.8	NA	97.6	97.6				
PCB-81	↓	↓	48.5	↓	97.0	97.0				
PCB-105	↓	↓	49.4	↓	98.9	98.8				
PCB-114	↓	↓	49.1	↓	98.1	98.2				
PCB-118	↓	↓	50.0	↓	100	100				
PCB-123	↓	↓	50.1	↓	100	100				
PCB-126	↓	↓	48.6	↓	97.2	97.2				
PCB-156 + 157	100	↓	97.9	↓	97.9	97.9				
~~PCB-157~~		↓		↓						
PCB-167	50.0	↓	49.6	↓	99.1	99.2				
PCB-169	↓	↓	49.3	↓	98.6	98.6				
~~PCB-170~~		↓		↓						
~~PCB-180~~		↓		↓						
PCB-189	50.0	↓	49.7	↓	99.5	99.4				

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

LDC #: 13247A3
SDG #: 0PN615252

VALIDATION FINDINGS WORKSHEET
Sample Calculation Verification

Page: 1 of 1
Reviewer: 9
2nd reviewer: R

METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

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?

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

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)

RRF = Relative response factor of the calibration standard.

V_o = Volume or weight of sample pruged in milliliters (ml) or grams (g).

Df = Dilution factor.

%S = Percent solids, applicable to soils and solid matrices only.

Example:

Sample I.D. 8 , PCB77:

Conc. = $\frac{(3.31e+07)(2000)(1)}{(1.36e+08)(0.91)(10.8)(\quad)}$

= 49.53 ng/kg

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

RECALC.16