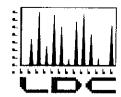
APPENDIX D: DATA VALIDATION REPORTS

ATTACHMENT D-1: SIM ANALYSES

ATTACHMENT D-2: TISSUE CHEMISTRY

ATTACHMENT D-3: SEDIMENT CHEMISTRY



LABORATORY DATA CONSULTANTS, INC.

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

Windward Environmental, LLC 200 West Mercer Street, Suite 401 Seattle, WA 98119 ATTN: Ms. Susie McGroddy LDC #13316 April 13, 2005

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

Rq.

	Job #04-08-06-21				•								Δ.	ttac	hme	nt 1																			
	Level II	LDC	#13316) (V	Vinc	swk	ırd	Enν	/iro	nm	ent	al,					W	A / I	LOV	ver	Du	wai	mis	h W	/ate	rw	ay (Gro	up))					
LDC	SDG#	DATE REC'D	DATE DUE	(82	AHs 70C IM)																		-		· · · · · · · · · · · · · · · · · · ·										
	Tissue/Sediment	1	<u> </u>	Т	_	T	s	Т	s	T	s	Т	s	Т	s	Т	s	Т	s	Т	s	T	s	W	s	W	s	W	s	W	s	W	s	W	s
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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.

Job #04-08-06-21 Attachment 1 LDC #13316 (Windward Environmental, LLC - Seattle WA / Lower Duwamish Waterway Group) Level II **PAHs** DATE DATE (8270C LDC SDG# REC'D DUE -SIM) T S T S T S T S T S T S W S TSTS Matrix: Tissue/Sediment S w s w s w HV45 03/28/05 04/18/05 0 15

0 0 0 0 0 0 0 0

Total

В

Attachment 2

SDG#: HV45				VALID	ATION SAN	MPLET	ABLE				LDC#: 1	3316A
Project Name: Lower	Duwamish Waterway G	roup		Paran	neters/Analy	∕tical M	ethod			Proje	ct #04-0	8-06-24
Client ID#	Lab ID #	Matrix	Date Collected	SVOA (8270C -SIM)								
LDW-C2-S2	HV45A	sediment	08/26/04	Х								
LDW-C3-S1	HV45B	sediment	08/29/04	Х								
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	Х								
LDW-C6-S	HV45F	sediment	08/25/04	Х								
LDW-C9-S	HV45G	sediment	08/15/04	Х				 				
LDW-B1a-\$	HV45H	sediment	08/12/04	X								
LDW-2Ba-S	HV45I	sediment	08/13/04	Х								
LDW-B3a-S	HV45J	sediment	08/26/04	Х								
LDW-B10b-S	HV45K	sediment	08/19/04	Х	****							
LDW-B1b-S	HV45L	sediment	09/27/04	Х					 			
LDW-B8b-S	HV45M	sediment	08/19/04	Х								
LDW-B9b-S	HV45N	sediment	08/11/04	Х								
LDW-B10a-S	HV450	sediment	08/26/04	Х								
LDW-B1a-SMS	HV45HMS	sediment	08/12/04	Х								
LDW-B1a-SMSD	HV45HMSD	sediment	08/12/04	Х								
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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.

SDG #: # DDM 41521 LDC #: 1323843

Routine Calibration Results Verification VALIDATION FINDINGS WORKSHEET

ᢐ Page: 2nd Reviewer: Reviewer:

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 $RRF = (A_x)(C_s)/(A_k)(C_x)$

ave. RRF = initial calibration average RRF Where:

RRF = continuing calibration RRF $A_x = Area$ of compound, $C_x = Concentration$ of compound,

 $A_{\rm k}=$ Area of associated internal standard $C_{\rm k}=$ Concentration of internal standard

	1							
				-	Reported	Recalculated	Reported	Recalculated
*	Standard ID	Calibratio n Date	Compound (Reference Internal Standard)	Average RRF	PPF Con(PRF Com (CC)	0 %	U%
					,	(
-	THE STATES	11	PCB-77 ("C-PCB-77)	0.9	52.	52.5		45
	DB5C-084	3/4/2	-2/05 PCB-105 (19C-PCB-105)	10.83	1 St.0	540	-	7.8
	15:1		PCB-156 (*3C-PCB-156)	86.0	100	101	7	8.0
		<u>.</u>	PCB-184 (13C-PCB-184)	67.0	52.9	4.88	2	70
				/	,		a	
2	1850 074 Sil		PCB-77 (¹⁸ C-PCB-77)	10.0	47.4	47.3	ls,	8-38
		12/2/	PCB-105 (3C-PCB-105)	0.83	20.2	8,0		0.6
			PCB-156 (¹³ C-PCB-156)	0.98	97.3	97.7	×	2.3
			PCB-18q (13C-PCB-18q)	0.79	5/5	51.8	, ,	シンペ
		-		,			S	
ო		·	PCB-77 (¹³ C-PCB-77)					
			PCB-105 (13C-PCB-105)					
			PCB-156 (¹⁶ C-PCB-156)					
			PCB-180 (¹³ C-PCB-180)					

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. Comments:

LDC #:_	3238A)
SDG #:	DPWG15217

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	of/_
Reviewer:	9
2nd reviewer:	70

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

N/A

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

N/A

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

Concentration = (A_)(I_)(DF) (A,)(RRF)(V,)(%S)

Area of the characteristic ion (EICP) for the compound to be measured

Area of the characteristic ion (EICP) for the specific internal standard

Amount of internal standard added in nanograms

Relative response factor of the calibration standard. RRF

Volume or weight of sample pruged in millititers (ml) ٧ or grams (g).

Dilution factor. Df

Percent solids, applicable to soils and solid %S

matrices only.

Example:

 $c = \frac{(1992 + 0)}{(9.82 + 0)} \frac{(2000)}{(0.91)} \frac{1}{(1.44)}$ = 309.29 ns/s

	matrices only.		Penorted	Calculated	
			Reported Concentration	Concentration	
#	Sample ID	Compound	()	()	Qualification
			÷		
			<u> </u>	······································	
			, , , , , , , , , , , , , , , , , , ,		
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LDC #:_	3238A3
SDG #:	DDW615=17

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

Page:	of
Reviewer:	7
	~

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

2nd Reviewer: 6

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory 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 = I LCS - LCSD I * 2/(LCS + LCSD)

LCS = Laboractry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: _OPR

	S	pike	Spiked	Sample	Lo	S	LCS	SD.	1.00	LCSD
Compound	- Ac	ided S/m \)	Concer (N S	tration	Percent I		Percent F			PD .
	LCS	LÇSD	LCS	LCSD	Reported	Recalc.	Reported	Recaic.	Reported	Recalculated
PCB-77	50	NÃ	AT.4	WA	94.9	94.8				Traditation .
PCB-81			47.2		944	94.4		······································		
PCB-105			51.2		102	102				
PCB-114			51.0		102	102				
PCB-118			526		105	105				
PCB-123	,		51.3		103	103				·
PCB-126	V		50.8		102	102				
PCB-156 PCBIST	100		95.1		97.1	97.1				
PCB-157										
PCB-167	50		49.0		97.9	98		······································		
PCB-169	V		27.6		953	95.3		·		
PCB-170										
PCB-180										
PCB-189	50	1	51.3		103	103				
				-						
	ŕ	· · · · · · · · · · · · · · · · · · ·	·							
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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 ^(e)	ion ID	Analyte	Substance
1	289.9224 291.9194 301.9626 303.9597 325.8804 327.8775 [292.9825]	M M+2 M M+2 M+2 M+4 Lock	C12 H6 35Cl4 C12 H6 35Cl3 37Cl4 13C12 H6 35Cl3 37Cl 13C12 H6 35Cl3 37Cl C12 H5 35Cl4 37Cl C12 H5 35Cl3 37Cl2 C7 F11	TCB TCB PeCB PeCB PeCB PeCB PeCB PeCB
2	325.8804 327.8775 337.9207 339.9178 359.8415 361.8385 371.8817 373.8788 393.8025 395.7996 405.8428 407.8398 [354.9892]	M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2 M+4 M+2	C12 H5 35Cl4 37Cl C12 H5 35Cl3 37Cl2 13C12 H5 35Cl3 37Cl2 13C12 H5 35Cl3 37Cl2 C12 H4 35Cl5 37Cl C12 H4 35Cl5 37Cl C12 H4 35Cl4 37Cl2 13C12 H4 35Cl5 37Cl 13C12 H4 35Cl6 37Cl C12 H3 35Cl6 37Cl C12 H3 35Cl5 37Cl2 C12 H3 35Cl5 37Cl2 C12 H3 35Cl5 37Cl2 C12 H3 35Cl5 37Cl2 C9F13	PeCB PeCB PeCB PeCB HxCB HxCB HxCB HxCB HxCB HyCB HpCB HpCB HpCB HpCB PFK
3	509.7229 511.7199 513.7170 [442.9728]	M+4 M+6 M+8 Lock	13C12 35Cl10 37Cl2 13C12 35Cl9 37Cl3 13C12 35Cl8 37Cl4 C10 F17	DCB PFK

S = internal/recovery standard

H = 1.007825 C = 12.000000

³⁵CI = 34,968853 $^{37}C1 = 36.965903$

 $^{19}C = 13.003355$

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-Nitrosodiphenyla <i>mi</i> ne	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-\$2	Diethy/phthalate	6.5 ug/Kg	6.5U ug/Kg
HV45	LDW-B9b-S	Diethylphthalate	6.6 ug/Kg	6.6U ug/Kg

Associated	Sample	Compound	Reported	Modified Final
SDG		TIC (RT in minutes)	Concentration	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-Chiorophenol-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)	Р

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 #: <u>HV45</u>	Level II	Page: /of /
Laboratory: <u>Analytical Resour</u>	ces, Inc.	Reviewer:
5 1	VOA3	2nd Reviewer: X
METHOD: CC/MS Polymedae	Pr Aromatic Hydrogerhana (EDA SIMI 946 Mothod 9270C SIMI)	

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

validation findings worksheets.

<u> </u>	Validation Area	<u> </u>	Comments				
l.	Technical holding times	4	Sampling dates:	State	8/11-9/27/04		
11.	GC/MS Instrument performance check	N		•			
111.	Initial calibration	N					
. IV.	Continuing calibration	N					
V.	Blanks	W					
VI.	Surrogate spikes	TW					
VII.	Matrix spike/Matrix spike duplicates	A					
VIII.	Laboratory control samples	4	205	Sky	THE PARTY OF THE P		
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	• ∌					
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

$\underline{\mathcal{M}}$	Sels						
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-\$2	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. Phenoi**	P. Bis(2-chloroethoxy)methane	EE. 2,6-Dinitrotoluene	TT. Pentachlorophenoi**	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-Chioro-3-methylphenoi**	KK. 2,4-Dinitrotoluene	ZZ. Pyrene	OOO. N-Nitrosodimethylamine
H. 2,2'-Oxybis(1-chloropropane)	W. 2-Methylnaphthalene	LL. Diethylphthalate	AAA. Butyibenzyiphthalate	PPP. Benzoic Acid
I. 4-Methylphenol	X. Hexachtorocyclopentadiene*	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	тт.
M. Isophorone	BB. 2-Nitroaniline	QQ. N-Nitrosodiphenylamine (1)**	FFF. Di-n-octylphthalate**	uuu.
N. 2-Nitrophenoi**	CC. Dimethy/phthalate	RR. 4-Bromophenyl-phenylether	GGG. Benzo(b)fluoranthene	vvv.
O. 2,4-Dimethylphenol	DD. Acenaphthylene	SS. Hexachlorobenzene	HHH. Benzo(k)fluoranthene	www.

LDC #: 133166=	b
SDG #:H V 45	

Blank extraction date:

VALIDATION FINDINGS WORKSHEET Blanks

Page:_	of
Reviewer:	Q/
2nd Reviewer:	1

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".

M N/A Was a method blank analyzed for each matrix?

MON N/A Was a method blank analyzed for each concentration preparation level?

N N/A Was a method blank associated with every sample?

Was the blank contaminated? If yes, please see qualification below. Blank extraction date: 3/17/05 Blank analysis date: 3/18/0 ≤

Blank analysis date:

Compound	Blank ID			ated Samples:_	-	w		-	
***************************************			7		S	ample Identific	ation		
, k	B131705	-	14	15					
24	15	6.5 N	6.6/U	9.6 M					
R.R.	6.7								
	/								 <u> </u>
	·					<u> </u>			
									<u> </u>
								 	

Conc. units:			Associat	ted Samples:				•	
Compound	Blank ID				s	ample Identifica	tion		
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				-	· · · · · · · · · · · · · · · · · · ·			~	
							· ·		

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 #: 13316526	
SDG #: HV45	

VALIDATION FINDINGS WORKSHEET Surrogate Recovery

Page:_	
Reviewer:	9-
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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/A

If 2 or more base neutral or acid surrogates were outside QC limits, was a reanalysis performed to confirm %R?

If any %R was less than 10 percent, was a reanalysis performed to confirm %R?

#	Date	Sample ID	Surrogate	%R (Limits)	Qualifications
		1	DeB	38.0 (40-130)	No anal.
			1,	()	
		8	/	39.2 ()	
				()	
		14	FB-P	37.2 ()	1/W/F
			JAFP	37.2 () 32.5 () 24.8 ()	
	· · · · · · · · · · · · · · · · · · ·		DCB	24.8 ()	
			PHC	33./ () 32.5 ()	
			JCH	32.5 ()	
	····	·	NBZ	30.4 ()	V
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* QC limits are advisory	QC Limits (Soil)	QC Limits (Water)		QC Limits (Soil)	QC Limits (Water)
S1 (NBZ) = Nitrobenzene-d5	23-120	35-114	S5 (2FP)= 2-Fluorophenol	25-121	21-100
S2 (FBP) = 2-Fluorobiphenyl	30-115	43-116	S6 (TBP) = 2,4,6-Tribromophenol	19-122	10-123
S3 (TPH) = Terphenyl-d14	18-137	33-141	S7 (2CP) = 2-Chlorophenol-d4	20-130*	33-110*
S4 (PHL) = Phenof-d5	24-113	10-94	S8 (DCB) = 1,2-Dichlorobenzene-d4	20-130*	16-110*

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.
 - 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.
 - 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.

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DC	SDG#	DATE REC'D	DATE DUE	PC Co (16	Bs ng. 68A)																						olecorranamano		the desired special sp					
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A	DPWG15217	03/07/05	03/28/05	14	0		Π																			-		<u> </u>	Ť			, , , , , , , , , , , , , , , , , , ,		
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13238ST.wpd

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		O. S. O. S. C.					SMOVE SOUTH STREET
SDG#: DPWG15217		***************************************		VALID	ATION SAMP	LE TABLE		***************************************	SEED SAN	507/07/00/03/05/03/03/03/03/03/04/04/04/04/04/04/04/04/04/04/04/04/04/	243	LDC#: 1	3238
Project Name: Lower	Duwamish Waterway	Group		Paran	neters/Analytic	al Method					Proje	ct #04-08	8-06
Client ID #	Lab ID #	Matrix	Date Collected	PCB Cong. (1668A)									
LDW-815-T**	L7510-1	tissue	08/10/04	Х							†		+
LDW-B2a-T	L7510-2	tissue	08/14/04	Х			1						
LDW-B3b-T	_7510-3	tissue	08/10/04	Х								***************************************	T
LDW-B4b-T	L7510-4	tissue	08/17/04	Х								†	1
LDW-B5a-T	L7510-5	tissue	08/22/04	х								***************************************	1
DW-B8a-T	L7510-6	tissue	08/27/04	Х									1
_DW-B9b-T**	L7510-7	tissue	08/11/04	X									1
_DW-B10a-T	L7510-8	tissue	08/25/04	х									1
DW-C1-T	L7510-9	tissue	08/26/04	Х									
_DW-C2-2-T	L7510-10	tissue	08/26/04	х					***************************************				T
_DW-C4-T	L7510-11	tissue	08/27/04	х						***************************************			
-DW-C6-T	L7510-12	tissue	08/26/04	х									T
DW-C7-T1	L7510-13	tissue	08/26/04	Х									
_DW-C8-T	L7510-14	tissue	08/26/04	х							***************************************		1
-DW-C9-T	L7510-15	tissue	08/25/04	Х								en e	
-DW-C10-T1	L7510-16	tissue	08/25/04	х					No contract to the contract of	***************************************	***************************************	······································	T
-DW-C10-T1DUP	L7510-16DUP	tissue	08/25/04	х								C DESCRIPTION OF THE PARTY OF T	1
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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 PCB-2 PCB-3 PCB-4 PCB-6 PCB-8 PCB-11 PCB-15 PCB-16 PCB-15 PCB-16 PCB-17 PCB-18 PCB-19 PCB-20 PCB-21 PCB-22 PCB-25 PCB-25 PCB-25 PCB-26 PCB-37 PCB-31 PCB-32 PCB-37 PCB-40 PCB-42 PCB-43 PCB-44 PCB-45 PCB-48 PCB-49 PCB-50 PCB-52 PCB-56 PCB-59 PCB-60 PCB-61 PCB-64 PCB-66 PCB-77 PCB-83 PCB-84 PCB-85 PCB-86 PCB-88 PCB-90 PCB-92 PCB-93 PCB-105 PCB-110 PCB-118 PCB-128 PCB-129 PCB-135 PCB-136 PCB-141 PCB-146 PCB-147 PCB-153 PCB-156 PCB-156 PCB-156 PCB-157 PCB-157 PCB-157 PCB-157 PCB-157 PCB-170 PCB-177	0.215 ng/Kg 0.245 ng/Kg 0.355 ng/Kg 0.355 ng/Kg 0.371 ng/Kg 0.180 ng/Kg 0.180 ng/Kg 0.704 ng/Kg 0.294 ng/Kg 0.344 ng/Kg 0.452 ng/Kg 0.812 ng/Kg 0.128 ng/Kg 0.128 ng/Kg 0.105 ng/Kg 0.251 ng/Kg 0.251 ng/Kg 0.251 ng/Kg 0.251 ng/Kg 0.251 ng/Kg 0.264 ng/Kg 0.264 ng/Kg 0.374 ng/Kg 0.166 ng/Kg 0.170 ng/Kg 0.166 ng/Kg 0.166 ng/Kg 0.167 ng/Kg 0.168 ng/Kg 0.169 ng/Kg 0.154 ng/Kg 0.154 ng/Kg 0.154 ng/Kg 0.154 ng/Kg 0.155 ng/Kg 0.156 ng/Kg 0.157 ng/Kg 0.158 ng/Kg 0.159 ng/Kg 0.150 ng/Kg 0.151 ng/Kg 0.151 ng/Kg 0.152 ng/Kg 0.153 ng/Kg 0.154 ng/Kg 0.155 ng/Kg 0.156 ng/Kg 0.157 ng/Kg 0.158 ng/Kg 0.159 ng/Kg 0.159 ng/Kg 0.150 ng/Kg 0.151 ng/Kg 0.531 ng/Kg 0.531 ng/Kg 0.531 ng/Kg 0.531 ng/Kg 0.531 ng/Kg 0.531 ng/Kg	All samples in SDG DPWG15217

Associated SDG	Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
DPWG15217	WGK743-101	1/27/05	PCB-177 PCB-179 PCB-180 PCB-183 PCB-187 PCB-190 PCB-194 PCB-195 PCB-196 PCB-198 PCB-201 PCB-202 PCB-203 PCB-204 PCB-205 PCB-206 PCB-208 PCB-209 Total Monochloro Biphenyls Total Dichloro Biphenyls Total Tetrachloro Biphenyls Total Hexchloro Biphenyls Total Octachloro Biphenyls Total Octachloro Biphenyls	0.271 ng/Kg 0.133 ng/Kg 1.07 ng/Kg 0.381 ng/Kg 0.383 ng/Kg 0.144 ng/Kg 0.188 ng/Kg 0.111 ng/Kg 0.117 ng/Kg 0.235 ng/Kg 0.023 ng/Kg 0.023 ng/Kg 0.051 ng/Kg 0.151 ng/Kg 0.151 ng/Kg 0.154 ng/Kg 0.154 ng/Kg 0.154 ng/Kg 0.156 ng/Kg 0.157 ng/Kg 0.460 ng/Kg 1.36 ng/Kg 1.36 ng/Kg 1.36 ng/Kg 1.36 ng/Kg 1.37 ng/Kg 5.37 ng/Kg 5.37 ng/Kg 5.37 ng/Kg 6.24 ng/Kg 7.47 ng/Kg 0.432 ng/Kg 0.133 ng/Kg	All samples in SDG DPWG15217

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 PCB-3	1.06 ng/Kg 1.60 ng/Kg	1.06U ng/Kg 1.60U ng/Kg
DPWG15217	LDW-B8a-T	PCB-2 PCB-3	0.895 ng/Kg 1.75 ng/Kg	0.895U ng/Kg 1.75U ng/Kg
DPWG15217	LDW-B9b-T**	PCB-2 PCB-3 PCB-11 Total Monochloro Biphenyls	0.259 ng/Kg 0.756 ng/Kg 4.45 ng/Kg 1.56 ng/Kg	0.259U ng/Kg 0.756U ng/Kg 4.45U ng/Kg 1.56U ng/Kg
DPWG15217	LDW-B10a-T	PCB-2 PCB-3 Total Monochloro Biphenyls PCB-1	0.526 ng/Kg 0.442 ng/Kg 1.35 ng/Kg 0.382 ng/Kg	0.526U ng/Kg 0.442U ng/Kg 1.35U ng/Kg 0.382U ng/Kg

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.

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.

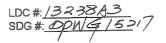
SDG # Labora METH The sa	: DPWG15217 httory: AXYS Analytical Services, Ltd. OD: HRGC/HRMS Polychlorinated Biphe	enyl Conge	PLETENESS WORKSHEET Level II / I / Page: of / Reviewer: 2nd Reviewer: 2
	Validation Area		Comments
I.	Technical holding times	A	Sampling dates: 8/10 - 27/024
11.	GC/MS Instrument performance check	LA	texetit Not leviewed for level !!
111.	Initial calibration	4	70 PSD < 20.
IV.	Routine calibration	A .	7.0525/33 (natine / Kabel)
V.	Blanks	W	
VI.	Matrix spike/Matrix spike duplicates	NA	not regil
VII.	Laboratory control samples	Á	OPR. STELL
VIII.	Regional quality assurance and quality control	N	
IX.	Internal standards	-4	Not reviewed for enell!
Χ.	Target compound identifications	-4	
XI.	Compound quantitation and CRQLs	A	
XII.	System performance	A	√
XIII.	Overall assessment of data	AA	
XIV.	Field duplicates	N	
XV.	Field blanks	N	
Note:	N = Not provided/applicable R = Rins	o compounds sate eld blank	detected D = Duplicate TB = Trip blank EB = Equipment blank

A = Acceptable N = Not provided/applicable SW = See worksheet *x lone IV Validated Samples:

Note:

M	Tissues.	· ·					
1	LDW-B1b-T **	11/1	LDW-C4-T	21	WE14743-101	31	
2/3	LDW-B2a-T	12	LDW-C6-T	22		32	
32/3	LDW-B3b-T	13/51	LDW-C7-T1	23		33	
$\frac{2}{4}/3$	LDW-B4b-T	14 01	_DW-C8-T	24		34	
5/3	LDW-B5a-T	15/01	_DW-C9-T	25		35	
$\frac{2}{6/3}$	LDW-B8a-T	16/5 1	-DW-C10-T1	26		36	
7]	LDW-B9b-T **	14/51	DW-C10-T1DUP	27		37	
₈ 2	LDW-B10a-T	18		28		38	
94	LDW-C1-T	19		29		39	
16/1	LDW-C2-2-T	20		30		40	

PCB-16.wpd

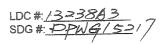


Page:_/of ____ Reviewer:_ 9/___ 2nd Reviewer:_ 9/___

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

Method: HRGC/HRMS Polychionnated Biphenyls (EPA Met Validation Area	Yes	No	NA	Findings/Comments
i. Technical holding limes				
All technical holding times were met.	/			
Cooler temperature criteria was met.	/			
II. GC/M3 Instrument performence shock				V
Was PFK exact mass 380.9780 verified?	1			
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 catibration				Г
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?	/	1		
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?				
M. 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?				W
Was a method blank performed for each matrix and concentration?				MANUSCO 27 27 27 1 4
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 OC limits?				

PCB-1668:IV version 1.0



VALIDATION FINDINGS CHECKLIST

Validation Area	Yes	No	NA	Findings/Comments
VIII Regional Quality Assurance and Quality Control				
Were performance evaluation (PE) samples performed?			<u> </u>	
Were the performance evaluation (PE) samples within the acceptance limits?				
IX Internal standards	T		г—	
Were Internal standard recoveries within the 25-150% criteria?	4			
Was the minimum S/N ratio of all internal standard peaks > 107	Ľ		L	· · · · · · · · · · · · · · · · · · ·
X. Target compound identification			I	
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 <u>+</u> 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.				

PCB-1668.IV version 1.0

LDC #: 13238A3

VALIDATION FINDINGS WORKSHEET Blanks

	Page:
	Reviewer:
2nd	Reviewer: 📐

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

Please see qualifications below for all of	uestions answered "N". N	lot applicable qu	estions are identified as "N/A".

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

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

Was method blank contamination less < CRQL for all target compounds?

Blank extraction date: 1/2/05 Conc. units: 45/45

Associated samples:

Compound	Blank ID	Sample identification								
k)4°H	-T43-101	2	6	7	8	9	10	11	/2	/3
PC 3 2	0.249	1.06/N	0.895/U	0.259 U	0536/n	0.311/4	0.293/11	0.316/4	0.847/U	0.665/4
PCB 3	0.355	1.60 U	1.75/W	0.756 U	0.442/N	0.819/11	0.715/U	0.711/4	1.23/11	1.34/11
PCBIL	1.86			4.45/U	,		, , , , , , , , , , , , , , , , , , , ,	,		/ 1
Total Honockinon Bipho	15 0460			156/4	1.35M		2º1/U	2.00 V		
PCB1	0/215				0.382/11			0.971/11		
PCB204	olda				F054/V	,		0.049/U		
	1				/ /					
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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	#: <u> 3238A3</u>	
SDG	#:0PWG1521/	7

VALIDATION FINDINGS WORKSHEET Blanks

2nd Reviewer:

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".
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Were all samples associated with a method blank?

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: 2/2/05

Conc. units: 10 Associated samples: Conc. units: 1)

Compound	Blank ID	Sample Identification								
WEK	23-10	1715	516							,
2BZ			0.853/U							
PCB 3	0.345	0.781/U								
PCBII	1.36								The second secon	
Total Monochino Bipheny	0.460									
PCBI	0.715									
PCB 204	0/012									
	1/1									
see att	achmer	1								

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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".

CLIENT ID: AXYS METHOD MLA-010 Rev 05 LAB BLANK Form 1A 1668A-S1 209 PCB CONGENER ANALYSIS REPORT Page 1 of 6 Sample Collection: N/A Project Number: N/A Lab Name: AXYS ANALYTICAL SERVICES WG14743-101 Lab Sample ID: 4033 Contract No.: 5.00 CORN OIL Sample Size: Matrix: Initial Calibration Date: 04-Feb-2005 N/A Sample Receipt Date: HR GC/MS Instrument ID: Extraction Date: 27-Jan-2005 GC Column ID: SPB-OCTYL Time: 2:26:42 12-Feb-2005 Analysis Date: Sample Data Filename: PB5C_074 S:7 Extract Volume (µL): 20 Blank Data Filename: PB5C_074 S:7 Injection Volume (µL): 1.0 PB5C_074 S:1 Cal. Ver. Data Filename: Dilution Factor: N/A Concentration Units: ng/kg **CO-ELUTIONS** LAB CONC. DETECTION ION ABUND. RRT COMPOUND IUPAC FOUND LIMIT RATIO FLAG1 NO. 5X PCB 1.075 3.45 1.001 JB 0.215 0.0348 1.225 JΒ 0.245 0.0439 3.42 0.988 - ModB 2 1.TTS 0.355 0.0488 3.71 1.000 K.IB Mor B 3 1.855 KJB 0.371 0,166 1.05 1.001 4 - DICB 0.150 5 U -фсв 0.9 1.61 1.176 PICB 6 кјв 0.180 0.141 2.3 υ 0.139 DICE 7 3.52 KJB 0.704 0.133 1.26 1.207 2,4' DICB 2,5 DICB U 0.138 9 U 0.137 рісв 10 0,969 6.8 0.160 1.61 JB 1.36 DICB 11 12 + 13сυ 0.159 - dice 12 12 + 13 C12 - plcs 13 0.152 U 14 0.294 0.188 1.14 1.000 KJB 15 1.72 JB 0.344 0.0391 1.07 1.166 16 - TriCB 2.26 0.452 0.0355 1.05 1.140 JB 4 - TriC 17 4.06 18 + 30 C JB 0.812 0.0289 1.14 1.113 2,2,5 - TriCB 18 1.002 0.128 0.0320 1.04 0.64 2,2,6 - TriCB 19 JB 0.848 0.0359 20 ÷ 28 C JB 0.982 1.02 - TriCB 27-02-2005 dd-mm-yyyy 14743AD1_1.xis. S2

0) (6 AXYS ANALYTICAL SERVICES LTD R.O. BOX 2219, 2045 MILLS RD. WEST, SIDNEY, B.C., CANADA VBL 358 TEL (250) 655-5800 FAX (250) 655-5811

AXYS METHOD MLA	nan Pay 05					CLIENT ID:		LAB BLANK	
Form 1A	4-010 1/64 02							N/A	
Page 2 of 6						Project Numbe Sample Data F		PB5C_074 S:7	
Lab Sample ID:	W	G14743-1	01						
	COMPOUND	IUPAC		CO-ELUTIONS	LAB	CONC. FOUND	DETECTION	ION ABUND. RATIO	RRT
	\sim	NO.	5×		FLAG ¹	, 04/14			
	B		2.25	21 + 33	C JB	0.450	0.0354	0.95	0.857
RB.	2,3,4 - TriCB	21 22	1.38	21+33	JB	0.276	0.0395	1.04	0.871
1 1	2 3,4" - TriCB 2 3,5 - TriQB	23	1		υ		0.0367		
New Contraction	213,6 - TriCB	24			U		0.0252		0.825
	2,8',4 - TriCB	25	0.525		JB	0.105	0.0324 0.0371	1.00 0.96	1.302
***************************************	2,3',5 - Tr CB	26	1.255	20 + 29	C JB	0.251 0.095	0.0371	1.10	1.151
A STATE OF THE STA	2,3',6 - TrICB	27	0.475		JB	CeO.U	0.0240		
	2,44' - TrICB	28		20 + 28	C20				
į.	2,4,5 - TICB	29		26 + 29	C26 C18				
	2,4,6 - TriCB	30	3,975	18 + 30	JB	0.795	0.0362	0.99	0.837
	2,4 5 - TriCB	31	1.32		KJB	0.264	0.0338	1.21	1,198
	2,4',6 - TriCB	32 33	1.7	21 + 33	C21				
	2',3,4 - TriCB 2',3,5 - TriCB	33 34		-	U		0.0369		
	3,3',4 - TriCB	35			U		0.0436		
	3,3',5 TriCB	36			U		0.0361		1.001
	3,4,4 TriCB	37	1.165		KJB	0.233	0.0451	1.26	1,001
ļ	3,4,5 TrICB	38			Ü		0.0374 0.0373		
	3,4',5 TriCB	39			U	0.374	0.0261	0.55	1.338
1:	2,2',3,3' TeCB	40	1.87	40 + 41 + 71	C KJB	0.374	0.0201	0.00	
	2,2',3,4 TeCB	41	0.83	40 + 41 + 71	C40 JB	0.166	0.0277	0.72	1.313
	2,2',3,4 TeCB	42		:	KJB	0.038	0.0308	0.29	1.247
	2,2',3,5 TeCB	43	0.19	44 + 47 + 65	C JB	0.945	0.0243	0.78	1.287
	2,2',3,5' - TeCB	44	4.725	45 + 51	C JB	0.170	0.0256	0.80	1.147
	2,2',3,6 - TeCB	45 46	0.0-	45.01	U		0.0302		
	z,z*,3,6' - TeCB 2,2',4,4' - TeCB	47		44 + 47 + 65	C44	*			4.075
	2,2',4,5 - TeCB	48	0.8!		KJB	0.162	0.0260	0.61	1.275 1.260
İ	2,2',4'5' - TeCB	49	3.34	49 + 69	C JB	0.668	0.0231 0.0247	0.83 0.98	1.112
	2,2',4,6 - TeCB	50	0.83	50 + 53	C KJB	0.166	0.0241	0.00	
	2,2',4,6' - TECB	51		45 + 51	C45	1.40	0.0251	0.81	1.235
	2,2',5,5' - TeCB		T.0		JB C50	1.40	0.0201		
	2,2',5,6' - TeCB	53		50 + 53	U	•	0.0157		
	2,2',6,6' - TeCB	54			Ü		0.0650		
	2,3 3',4 - TeCB	55 56	1.435		KJB	0.287	0.0664	1.16	0.904
	2,3,3',4' - TeCB 2,3,3',5 - TeCB	56 57	1.1.		U		0.0634		
	1 _ 1_	58			U		0,0622		4 000
	2,3,3',5' - TeQB 2,8,3',6 - TeQB	59	0.485	59 + 62 + 75	C KJB	0.097	0.0200	0.48	1.303 0.911
	2,3,4,4' - TeQB	60	0.545		KJB	0.109	0.0640	1.52 0.66	0.875
	2,3,4,5 - TeCB	61	5.15	61 + 70 + 74 + 76	C JB	1.03	0.0597	0.00	3.010
	2,3,4,6 - TeCB	62		59 + 62 + 75	C59 ປ		0.0611		
1	2,3,4',5 - TeC\$	63			U		2.0477		
				Sk		04/00	Chemist		27-02-2005 dd-mm-yyyy
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ONO AXYS ANALYTICAL SERVICES LTD P.O. 80X 2219, 2045 MILLS RO. WEST, SIDNEY, B.C., CANADA VOL 358 TEL (250) 655-5800 FAX (250) 655-5817

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	AXYS METHOD MLA-010 Rev 05			CLIENT ID:		LAB BLANK	
Vind	Form 1A			Project Numbe	r:	N/A	
*****	Page 3 of 6 Lab Sample ID:	WG14743-101		Sample Data F		PB5C_074 S:7	
27546	cab campio in t						
~	COMPOUND	IUPAC CO-ELUTIONS	LAB	CONC.	DETECTION	ION ABUND.	RRT
Peacer	COMPOUND	NO.	FLAG1	FOUND	LIMIT	RATIO	
Section 1	- a - 7	6×	ID.	0.200	0.0404	0.76	1.350
	2,3,4',6 - TeCB 2,3,5,6 - TeCB	64 1. 545 65 44 + 47 + 65	JB C44	0.309	0.0194	0.76	1.330
	2,3',4,4' - TeQB	66 2.815	JB	0.563	0.0617	0.71	0.884
.070	2,8',4,5 • Te¢B	67	U		0.0578		
The state of the s	2,3',4,5' - TeCB	68 69 49 + 69	IJ C49		0.0615		
	2,3',4,6 - TeCB 2,3',4',5 - TeCB	70 61 + 70 + 74 + 76	C61				
ET JUL	2,3141,6 - TeCB	71 40 + 41 + 71	C40				
	2,3'5,5' - TeCB	72	U		0,0646		
	2,3',5',6 - feCB	73 74 61 + 70 + 74 + 76	U C61		0.0192		
-meters	2,4,4,5 - TeCB 2,4,4 6 - TeCB	75 59 + 62 + 75	C59				
*Chapmi	2',3,4,6 TeCB	76 61 + 70 + 74 + 76	C61			0.05	4.004
	3,3',4,4 TeCB	77 0.605	JB U	0.121	0.0727 0.0683	0.85	1.001
.amed	3,3',4,5 TeCB 3,3',4,5' TeCB	78 79	Ü		0.0548		
نہ	3,3',5,5' TeCB	80	U		0.0616		
_	3,4,4',5 - TeCB	81	U U		0.0671 0.0816		
	2,2',3,3',4 - PeCB 2,2',3,3',5 - ReCB	82 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' - P+CB	85 0.65 85 +: 116 + 117	C KJB	0.130	0.0625	2.31	0.920
-	2,2',3,4,5 - PeCB 2,2',3,4,5' - PeCB	86 3 4 7 5 86 + 87 + 97 + 108 + 119 + 125 87 86 + 87 + 97 + 108 + 119 + 125	C KJB C86	0.695	0.0618	1.28	0.901
نب	2,2',3,4,6 - PeGB	88 1.08 88 + 91	C JB	0.216	0.0694	1.32	1.155
	2,2*,3,46' - PeCB	89	U		0.0749	4.00	2.000
	2,2',3,4',5 - PeCB	90 6.4 90 + 101 + 113 91 88 + 91	C JB C88	1.28	0.0637	1.39	0.869
-	2,2',3,4',6 - PeCB 2,2',3,5,5' - PeCB	92 0.77	JB	0.154	0.0743	1.49	0.853
g#Talm	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 2,2',3 6,6' - PeCB	95 93 + 95 + 98 + 100 + 102 96	C93 U		0.142		
an 1	2,2',3',4,5 - PeCB	97 86 + 87 + 97 + 108 + 119 + 125	C86				
	2,2',\$',4,6 - PeCB	98 93 + 95 + 98 + 100 + 102	C93 C83				
	2,2' 4,4',5 - PeCB 2,2',4,4',6 - PeCB	99 83 + 99 100 93 + 95 + 98 + 100 + 102	C93				
25-120	2,2,4,5,5' - PeCB	101 90 + 101 + 113	C90				
	2,2',4,5,6' - PeCB	102 93 + 95 + 98 + 100 + 102	C93		0.0050		
1200	2/2',4,5',6 - PeCB 2,2',4,6,6' - PeCB	103 104	U		0.0652 0.112		
Ene	2,3,3',4,4' - PeCB	105 3.77	KJB	0.754	0.0004	2.60	1,001
248	2,3,3',4,5 - PeCB	106	U		0.0831		
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• 10 AXYS ANALYTICAL SERVICES LTD P.O. BOX 2219, 2045 MILLS RD. WEST, SIDNEY, B.C., CANADA VØL 3S8 TEL (250) 655-5800 FAX (250) 655-5811

orm 1A age 4 of 6 ab Sample ID: COMPOUND	WG14743-101		Project Numbe Sample Data F	e:		
COMPOUND	IUPAC CO-ELUTIONS				N/A PB5C_074 S:7	
	NO. 5X	LAB FLAG ¹	CONC. FOUND	DETECTION LIMIT	ION ABUND. RATIO	RRT
PCP 4,3,3',4',5 - PeCB	107 107 + 124	СU		0.0842		
2/3,3',4,5' - PeCB	108 86 + 87 + 97 + 108 + 119 + 125 109	C86 U		0,0820		
2 3,3',4,6 - PeQB	110 6.45 110 + 115	СЈВ	1.29	0.0546	1.49	0.92
2,3 3'.5,5' - PeCB	111	U		0.0548		
2,3 3',5,6 - PeCB	112			0.0560		
1 1 1				0.0866		
2,3,4,4',6 - PeCB	115 110 + 115	C110				
2,3,4,5,6 - FeCB	116 85 + 116 + 117	C85				
2,3,415,6 - PeCB	117 85 + 116 + 117		1 //1	0.0822	1 40	1.001
		C86	1.441	0.0022	UPP,1	7.00
2,3',4,5 5' PeCB	120	U		0.0554		
2,3',4,5'6 PeCB	121	U		0.0545		
1 11						
1	124 107 + 124	C107		-		
2',3,4,5, 6 ' - PeCB	125 86 + 87 + 97 + 108 + 119 + 125	C86				
3,3',4,4' - PeCB	126					
1 11	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		
	131 132 2.56 5		0.513		1.50	1.175
	133	U		0.0926		
2,2',3,8',5,6 - HxCB	134 + 143	CU		0.0937		
2,2',3,5',5,6' - HxCB	135 2.6 135 + 151 + 154					1,103 1,024
		U	0,107	0.0738	1.00	1.024
2,2',4,4,4',5' - HXCB	138 129 + 138 + 160 + 163	C129				
2,2° 3,4,4°,6 - HxCB	139 139 + 140	CU		0.0877		
			0.337	0.0931	0.73	0.903
	142	υ	•	0.0979	•	
2,2',3,4,5,6' - Hx¢B	143 134 + 143	C134				
2/2',3,4,5',6 - HxCB	144					
2/2',3,4,6,6" - HXQB 2/2',3.4',5.5' - HXQB	146 1.53	KJB	0.306	0.0790	1.66	0.885
2.2'.3.4'.5.6 - HXOB	147 A .985 147 + 149	C JB	0.997	0.0866	1.22	1.133
	148	U		0.111		
2,2',3,4',5,6' - H×CB 2,2',3,4',5',6 - H×CB	149 147 + 149	C147				
	2,3,3',4',6 - PeGB 2,3,3',5,6 - PeGB 2,3,4',5 - PeGB 2,3',4,5 - PeGB 2,3',4,5 - PeGB 2,3',4,5 - PeGB 2,3',4,5 - PeGB 2',3,4,5 - PeGB 2,2',3,3',4 - HxGB 2,2',3,3',4,5 - HxGB 2,2',3,3',4,6 - HxGB 2,2',3,3',5,6 - HxGB 2,2',3,3',5,6 - HxGB 2,2',3,4',5 - HxGB 2,2',3,4',5 - HxGB 2,2',3,4',5 - HxGB 2,2',3,4,4',5 - HxGB	2.33'.4',6 - PeGB 110 6.45 110+115 2.33'.5.5' - PeGB 111 2.33'.5.6 - PeGB 112 2.3.4',5 - PeGB 113 90+101+113 2.3.4',5 - PeGB 114 2.3.4',5 - PeGB 115 110+115 2.3.4,5 - PeGB 116 85+116+117 2.3.4,5 - PeGB 117 85+116+117 2.3'.4,5 - PeGB 118 85+116+117 2.3'.4,5 - PeGB 119 86+87+97+108+119+125 2.3'.4,5 - PeGB 120 2.3'.4,5 - PeGB 121 2'.3.3',4,5 - PeGB 122 2'.3,4,4' - PeGB 123 2'.3,4,5 - PeGB 124 107+124 2'.3,4,5 - PeGB 125 86+87+97+108+119+125 3.3',4,5 - PeGB 126 3.3',4,5 - PeGB 127 2.2'.3,3',4,5 - PeGB 127 2.2'.3,3',4,5 - PeGB 127 2.2'.3,3',4,5 - PeGB 128 2.2'.3,3',4,5 - PeGB 129 2.2'.3,3',4,5 - PeGB 127 2.2'.3,3',4,5 - PeGB 127 2.2'.3,3',4,5 - PeGB 128 2.2'.3,3',4,5 - PeGB 129 2.2'.3,4,4',5 - PeGB 133 2.2'.3,4,4',5 - PhGB 134 2.2'.3,4,4',5 - PhGB 136 139 139+140 2.2'.3,4,5,6 - HxGB 139 139+140 2.2'.3,4,5,6 - HxGB 144 2.2'.3,4,5,6 - HxGB 145 2.2'.3,4,5,6 - HxGB 145 2.2'.3,4,5,6 - HxGB 145 2.2'.3,4,5,6 - HxGB 144 2.2'.3,4,5,6 - HxGB 145 2.2'.3,4,5,5 - HxGB 145	2.3 3.4 5.6 - PedB 110 6.4 5 110 + 115 CJB 2.3 3.5.5 - PedB 111 U 2.3 3.5.5 - PedB 111 U 2.3 3.5.5 - PedB 111 U 2.3 3.5.5 - PedB 113 90 + 101 + 113 C90 2.3 4.5.5 - PeCB 114 U 2.3 4.5.5 - PeCB 115 110 + 115 C110 2.3 4.5.5 - PeCB 116 85 + 116 + 117 C85 2.3 4.5.5 - PeCB 117 85 + 116 + 117 C85 2.3 4.5.5 - PeCB 118 7.0 5 JB 2.3 4.5.5 - PeCB 120 2.3 4.5.5 - PeCB 120 2.3 4.5.5 - PeCB 121 U 2.3 3.4.5 - PeCB 121 U 2.3 3.4.5 - PeCB 122 U 2.3 3.4.5 - PeCB 124 107 + 124 C107 2.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.4 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 2.3 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 U U 2.2 3.3 4.5 - PeCB 125 86 + 87 + 97 + 108 + 119 + 125 C86 U U 2.2 3.3 4.5 - PeCB 126 U U 2.2 3.3 4.5 - PeCB 126 U U 2.2 3.3 4.5 - PeCB 127 U U 2.2 3.3 4.5 - PeCB 128 128 129 + 138 + 160 + 163 C JB 2.2 3.3 4.5 - HxCB 131 2.2 3.3 4.5 - HxCB 132 U U 2.2 3.3 4.5 - HxCB 132 U U 2.2 3.3 4.5 - HxCB 134 134 1443 C U U 2.2 3.4 4.5 - HxCB 139 139 140 C U 2.2 3.4 4.5 - HxCB 139 139 140 C U 2.2 3.4 4.5 - HxCB 139 139 140 C U 2.2 3.4 4.5 - HxCB 139 139 140 C U 2.2 3.4 4.5 - HxCB 140 140 139 140 C 130 139 140 C 130 134 140 140 140 140 140 140 140 140 140 14	2,3,4',6',6 - PedB 110 6.45 110 + 115 CJB 1.29 2,3'*15.5' - PedB 111 U 2,3,1'5.5' - PedB 112 2,3,1'5.5' - PedB 113 90 + 101 + 113 C90 2,3,1'5.5' - PedB 115 100 + 115 C110 2,3,1'5.5' - PedB 115 100 + 115 C110 2,3,1'5.5' - PedB 116 85 + 116 + 117 C85 2,3,1'5.5' - PedB 117 85 + 116 + 117 C85 2,3,1'5.5' - PedB 118 705 JB 1.41 2,3,1'5.5' - PedB 120 U 2,3'1,1'5' - PedB 120 U 2,3'1,1'5' - PedB 120 U 2,3'1,1'5' - PedB 121 U 2,3,1'4,1' - PedB 122 U 2,3,1'4,1' - PedB 123 U 2,3,1'4,1' - PedB 124 107 + 124 C107 2',3,1'5' - PedB 125 86 + 87 + 97 + 108 + 119 + 125 C86 3,3'1,1' - PedB 126 U 2,2',3,1',5' - PedB 127 U 2,2',3,1',5' - PedB 128 U 2,2',3,1',5' - PedB 129 \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	2,3,3,4,6,6,PeGB 110 6.4.5 110 + 115	2.33-4.6 - PedB 1110 6.45 110 115 C.JB 1.29 0.0546 1.49 2.34-5.5 - PedB 1112 U 0.0546 1.49 0.0548 1.29 U 0.0546 1.49 0.0548 1.29 U 0.0546 1.49 0.0548 1.29 1.29-1.34-5.5 - PedB 112 U 0.0560 1.34-5.5 - PedB 113 90 + 101 + 113 C90 U 0.0866 1.23-4.5.5 - PedB 116 S5 + 116 + 117 C85 1.34-5.5 - PedB 116 S5 + 116 + 117 C85 1.34-5.5 - PedB 118 7.05 JB 1.41 0.0822 1.49 1.39 1.49 1.49 1.49 1.49 1.49 1.49 1.49 1.4

AXYS METHOD MLA-010 Rev 05 CLIENT ID: LAB BLANK Form 1A Page 5 of 6 Project Number: N/A WG14743-101 Lab Sample ID: Sample Data Filename: PB5C_074 S:7 COMPOUND **IUPAC** DETECTION **CO-ELUTIONS** LAB CONC. ION ABUND. RRT NO. FOUND RATIO FLAG1 5 X 2,2',3,4',6,6' - HxCB 150 u 0.0787 ,2',3,5,5',6 - HxCB 135 + 151 + 154 C135 1,2',3,5,6,6' - HxdB 152 11 0.0762 22',4,4',5,5' - Hx¢B 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 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' - HKCB 156 ÷ 157 157 C156 158 1.25 2,3 3',4,4',6 - HXCB JB 0.250 0.0720 1.12 0.938 2,3,3',4,5,5' - HXCB 159 11 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', 5',5',6 HXCB 164 0.535 JB 0.107 0.0740 1.12 0.921 2,3,3',5,5',6 - HxCB 165 U 0.0771 2,3,4, 1,5,6 - HxCB 166 128 + 166C128 167 0.83 2,3',4,4,5,# - HxCB KJB 0.166 0.0664 1.47 1.001 2,3',4,4' 5' 6 - HxCB 3,3',4,4' 5.5' - HxCB 168 153 + 166 C153 169 u 0.0786 2,2',3,3',4,4',5 - HpCB 2,2',3,3',4,4',6 - HpCB 3,38 170 JB 0.676 0.169 1.00 0.936 171 171 + 173CU 0.156 2,2',3,3',4,\$5' - HpCB 172 U 0.159 2,2',3,3',4|5,6' - HpCB 2,2',3,3',4|5,6' - HpCB 173 171 + 173C171 1.855 174 JB 0.371 0.140 1.02 1.133 2,2',3,3',4,5',6 - HpCB 2,2',3,3',4,6,6' - HpCB 175 U 0.135 176 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 0.665 2,2',3,3,5,6,6' HpCB 179 KJB 0.133 0.0963 2.25 1.010 2,2',3,4',5,5' HpCB 5.35 180 C JB 180 + 1931.07 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' B,4,5,5',6 - HPCB 185 183 + 185C183 2,2 3,4,5,6,6 - HpCB 186 U 0.101 1.965 2,2'3,4',5,5',6 - HDCB 187 KJB 0.393 0.126 1.39 1.110 2,2,3,4',5,6,6' - HCB 188 U 0.0770 2,3(3',4,4',5,5' - HpCB 189 U 0.108 2,8,3',4,4',5,6 - HpCB 190 O.T2 **KJB** 0.144 0.129 0.947 2,8,3',4,4',5',6 - Hp¢B 191 U 0.125 2,3,3',4,5,5',6 - HpCB 192 U 0 127

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Approved by: QA/QC Chemist

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■ AXYS ANALYTICAL SERVICES LTD R.O. BOX 2219, 2045 MILLS RD. WEST, SIDNEY, B.C., CANADA VOL 358 TEL (250) 655-5800 FAX (250) 655-5811

AXYS METHOD MLA-010 Rev 05 CLIENT ID: LAB BLANK Form 1A Page 6 of 6 Project Number: N/A Lab Sample ID: WG14743-101 Sample Data Filename: PB5C_074 S:7 COMPOUND IUPAC CO-ELUTIONS LAB CONC. DETECTION ION ABUND. RRT FOUND FLAG1 LIMIT 5× RATIO 2,3,3',4',5,5',6 - HpCB 77 193 180 + 193 C180 0.94 2,213,31,4,41,5,51 - OccB 194 KJB. 0.188 0.0067 1.11 0.991 2,2',8,3',4,4',5,6 - 9ccB 195 0.555 JΒ 0.0075 0.111 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',3,8',4,4',6,9' - OccB 197 197 + 200 CU 0.0052 2,2',3,8',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 198 + 199 199 C198 2,2',3,3 4,5,6,6' - Occs 200 197 + 200 C197 2,2',3,3',5',6,6' - OcCB 2,2',3,3',5',6,6' - OcCB 201 0.115 202 0.43 203 0.755 **KJB** 0.023 0.0053 5.00 1.023 JB 0.086 0.0051 0.79 1.000 2,2',3,4,4\5,5',6 - OcCB K.IB 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 ,3',4,4',5\5',6 - OcCB 205 0.255 KJB 0.051 0.0061 3.40 1.000 ,3,3',4,4',5,3',6 - NoCB 206 O.T45 **KJB** 0.149 0.111 0.96 1.001 2/2',3,3',4,4',5,6,4' - NoCB 207 U 0.0915 2,2',3,3',4,5,5',6,6' NoCB 2,2',3,3',4,4',5,5',6,6' DeCB 208 0.665 JB 0.133 0.0922 0.82 1.001 209 0.77 **KJB** 0.154 0.0076 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.

_QA/QC Chemist

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Approved by:

27-02-2005

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AXYS METHOD MLA-010 Rev 05 CLIENT ID: PCB-TOTAL_209 LAB BLANK Page 1 of 1 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 CORN OIL Matrix: 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 N/A PB5C_074 S:7 **Dilution Factor:** Sample Datafile(s): Concentration Units: ng/kg **PCB HOMOLOGUE GROUP** LAB CONC. DETECTION FOUND LIMIT FLAG1 **Total Monochloro Biphenyls** 0.460 0.0488 **Total Dichloro Biphenyls** 1.36 0.188 23.45 **Total Trichloro Biphenyls** 4.69 0.0451 26.85 **Total Tetrachloro Biphenyls** 5.37 0.0727 31.2 Total Pentachloro Biphenyls 6.24 0.142 3T.35 **Total Hexachloro Biphenyls** 7.47 0.111 12,5 **Total Heptachloro Biphenyls** 2.50 0.169 2.16 **Total Octachloro Biphenyls** 0.432 0.0079 0 665 **Total Nonachtoro Biphenyls** 0.133 0.111 U Decachloro Biphenyl 0.0076 TOTAL PCBs (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. 4.39 Hawy Moral DAVOC Chemist 02-03-2005 14743PCBTOTAL_1.xls, S3 dd-mm-yyyy NZE!

VALIDATION FINDINGS WORKSHEET nitial Calibration Calculation Verification

	Page:
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2nd	Reviewer: X

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:

 $\mathsf{RRF} = (\mathsf{A}_{\mathsf{x}})(\mathsf{C}_{\mathsf{b}})/(\mathsf{A}_{\mathsf{b}})(\mathsf{C}_{\mathsf{x}})$ average RRF = sum of the RRFs/number of standards

 $\begin{array}{ll} A_{\tt x} = \text{Area of compound,} & A_{\tt k} = \text{Area of associated internal standard} \\ C_{\tt x} = \text{Concentration of compound,} & C_{\tt k} = \text{Concentration of internal standard} \\ S = \text{Standard deviation of the RRFs,} & X = \text{Mean of the RRFs} \end{array}$

%RSD = 100 * (S/X)

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (Initial)	Average RRF (initial)	RRF (std)	RRF (/ std)	%RSD	%RSD
1	ICAZ =	2/1/05	PCE-77 (¹⁸ C-PCB-77)	0.91	0.91	0.87	0.86	3.54	3,20
	/	/"/ [PCE-105 (¹³ C-PCB-105)	0.83	0.83	0.82	0.82	3.00	3.19
		· [PCB-156 (¹⁹ C-PCB-156)	0.98		0.96	0.96	1.52	1.61
	and the state of t			0.79	0.79	0.79	0.79	2.01	1.65
					7				
2			PCB-77 (¹⁸ C-PCB-77)						
		ſ	PCB-105 (19C-PCB-105)						**************************************
			PCB-156 (¹³ C-PCB-156)				COLUMN TO THE PARTY OF THE PART		
			PCB180 (¹³ C-PCB-180)						
							24.H	A Miles Company of the Company of th	
3			PCB-77 (¹² C-PCB-77)						Market Control
		ľ	PCB-105 (¹⁸ C-PCB-105)						
		ľ	PCB-156 (¹³ C-PCB-158)						
			PCB-180 (¹³ C-PCB-180)						
								PPGGGALASTANIA STANIS STANIS STANIS AND	

Comments:	Heter to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the
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VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

	Page:_	
	Reviewer:	9-
2nd	Reviewer:	24

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 $RRF = (A_i)(C_{in})/(A_{in})(C_i)$

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

A, = Area of compound,

A. = Area of associated internal standard

C_k = Concentration of compound,

C_h = Concentration of internal standard

					Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibratio n Date	Compound (Reference internal Standard)	Average RRF (initial)	RRF Conc (CC)	88 F@m((CC)	%D	%D
	PBSC BAS	, -7	PCB-77 (¹³ C-PCB-77)	0.91	52.	52.3	1.	4.5
1	PB5C-084	2/11/05	PCB-105 (¹⁹ C-PCB-105)	0.83	54.0	540		7.8
	5-1	/ / / "	PCB-156 (¹³ C-PCB-156)	0.98	100	101	, X	0.8
			PCB-180 (13C-PCB-180)	0.79	52.9	53.2	The state of the s	6.4
	p	- CONTRACTOR STATEMENT OF THE STATEMENT		//	/			
2	DBSC_074 5-1		PCB-77 (13C-PCB-77)	0.91	AT.A	47.3	19,	5-38
		17/2/05	PCB-105 (¹³ C-PCB-105)	0.83	50.d	50,3	1	0.6
		/ /-	PCB-156 (¹³ C-PCB-156)	0.98	97.3	97.7	X	· 3
			PCB-189 (13C-PCB-189)	0.79	51.5	51.8	9	Za La Diamenta de la Companya del Companya de la Companya del Companya de la Comp
		CONTROL OF THE CONTRO					8	
3			PCB-77 (1°C-PCB-77)					
	Demonstration of the control of the		PCB-105 (¹³ C-PCB-105)				And the second section of the second section of the second	
			PCB-156 (¹³ C-PCB-156)				And the second s	По-филосон в веней на беспективности беспективности по до него постоя в контрактивности в него постоя в контрактивности по него постоя в контрактивности в него постоя в контрактивности в него постоя в него посто
			PCB-180 (¹³ C-PCB-180)					дення на
							The state of the s	AND THE RESIDENCE OF THE PROPERTY OF THE PROPE

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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VALIDATION FINDINGS WORKSHEET <u>Laboratory Control Sample Results Verification</u>

	Page:_	of
	Reviewer:	<u> </u>
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METHOD: HRGC/HRMS Polychlorinated Biphenyls (EPA Method 1668)

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory 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 = ILCS - LCSD | * 2/(LCS + LCSD)

LCS = Laboractry control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: OF

		oike	Spiked S		LC	s	LCS	LCSD		CSD
Compound		ded (m \)	Concent (N S	tration	Percent F	Recovery	Percent R	ecovery	RF	D
	LCS	LCSD	LCS	LCSD	Reported	Recaic.	Reported	Recalc.	Reported	Recalculated
PCB-77	50	NA	AT.4	WA	94.9	94.8				
PCB-81			47.2		944	94.4			Omnepation in the Control of the Con	
PCB-105			4.2		102	102			от при	
PCB-114			51.0		102	102			Commence of the Commence of th	
PCB-118			52.6		105	105		MARKET DE LEGISLATION	MATERIAL PROPERTY OF THE PROPE	
PCB-123			51.3		103	103			endan-dautaroara actronomonada et a communica (e 4 - y 4 - 6 y 4 - 6 y 4 - 6 y 4 - 6 y 4 - 6 y 4 - 6 y 4 - 6 y	умовна неб-пов-тов под издухну доставляет под конфинулији не од адам од недост
PCB-126	V		50.8		102	102			Personal Section (Section 1998) Annual photographic participation of the Company	о-164-46-1840 ж.
PCB-156 PCB (ST	100		97.1		97.1	97.1				
PGB-167									The second secon	autot tääntäiteksin on oli kinnepyysellysellystä säärikkikoivoin yva assayseet tataattava
PCB-167	50		49.0		97.9	98			ANTO TO COST THE PROPERTY OF T	
PCB-169	V		AT.6		as.3	95.3	 			
POB-170								ORANGE WATER TO THE TAXABLE WA		**************************************
PC8-160-									la anticipa la companya de la compa	MMMM An experimental position of the state o
PCB-189	50	V	51.3		103	103	White committee or agreement consumer	- Walter and the second	mmilyan-kontooli soosiiniidii ilikkin ahvahoossa saasaa qaaaa ahka ahka milka	**************************************
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person (every section of the delivery section construction and appropriate delivery section and the section of the every section of the							W	NNSP-50 Access to relay a store difficult we were experienced.	Auszanomen (1900) des sembrares estates (1900) des semblacións estates (1900) des sembrares (1900) de semb	
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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.

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ions Monitored for HRGC/HRMS Analysis of Polychlorinated Biphenyls

Descriptor	Accurate mass ^(a)	lon ID	Analyte	Substance
1	289.9224	М	C12 H6 35Cl4	тсв
	291.9194	M+2	C12 H6 35Cl3 37Cl4	тсв
	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	FFK
2	325.8804	M+2	C12 H5 35Cl4 37Cl	PeCB
	327.8775	M+4	C12 H5 35Cl3 37Cl2	PeCB
TO THE PARTY OF TH	337.9207	M+2	13C12 H5 35Cl4 37Cl	PeCB
	339.9178	M+4	13C12 H5 35Cl3 37Cl2	PeCB
denomination of the control of the c	359,8415	M+2	C12 H4 35Cl5 37Cl	HxCB
	361.8385	M+4	C12 H4 35Cl4 37Cl2	HxCB
Control	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	НрСВ
	395,7996	M+4	C12 H3 35Cl5 37Cl2	HpCB
	405.8428	M+2	13C12 H3 35Cl6 37Cl	НрСВ
	407.8398	M+4	13C12 H3 35Cl5 37Cl2	HpCB
	[354.9892[Lock	C9F13	FFK
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	C10F17	PFK

S = internal/recovery standard

H = 1.007825 C = 12.000000 ¹³C = 13.003355 F = 18.9984

³⁵Cl = 34.968853 ³⁷Cl = 36.965903

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

LDC	#:_	3238A3
SDG	#;	OPW61521

matrices only.

VALIDATION FINDINGS WORKSHEET <u>Sample Calculation Verification</u>

Page:	of
Reviewer:	9
2nd reviewer:	M/

v 1	V/A	HRGC/HRMS Polychlorinated Biphenyls (EPA Were all reported results recalculated and Were all recalculated results for detected	Method 1668) d verified for all level IV samples? target compounds agree within 10.0% of the reported results?
Concen	tration		Example:
		(A,)(RRF)(V,)(%S)	Sample I.D. / POBTT:
A_x	200	Area of the characteristic ion (EICP) for the compound to be measured	Sample I.D, PUS (] :
A _b	222	Area of the characteristic ion (EICP) for the specific internal standard	1292127
i,	=	Amount of internal standard added in nanograms (ng)	Conc. = $(1.992+0)$, (2000) , $(9.82+0)$, (0.91) , (1.44) , (0.91)
RRF	=	Relative response factor of the calibration standard.	,
٧.	=	Volume or weight of sample pruged in milliliters (ml) or grams (g).	= 309.29 ns/F8.
Df	=	Dilution factor.	
%S	=	Percent solids, applicable to soils and solid	

#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
				-	
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RECALC.16



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.
 - 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.
 - 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.

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рс	SDG#	DATE REC'D	DATE DUE	PC Co	Bs ng. 68A)										***************************************												***************************************				almärläkistäken avaiha		***	
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1	DPWG15252	03/11/05	04/01/05	0	14																					<u> </u>	Ī		<u> </u>	† '		**********		
4	DPWG15252	03/11/05	04/01/05	0	2																													
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Constitution of the second of	WWW.				Attach	ment 2							
SDG#: DPWG15252				VALID	ATION S	AMPLE	TABLE					LDC#: 1	3247A
Project Name: Lower	Duwamish Waterway (Group		Paran	neters/Ar	alytical	Method					ct #04-08	
Client ID #	Lab ID #	Matrix	Date Collected	PCB Cong.									
LDW-B1b-S	L7505-1	sediment	08/10/04	Х								о при	
LDW-B2a-S	L7505-2	sediment	08/14/04	х								***************************************	
LDW-B3b-S	L7505-3	sediment	08/10/04	Х							and the second second second second		
LDW-B4b-S	L7505-4	sediment	08/17/04	Х					-	**************************************			
LDW-B5a-S	L7505-5	sediment	08/22/04	Х									
LDW-B8a-S	L7505-6	sediment	08/27/04	Х							-		
LDW-B9b-S	L7505-7	sediment	08/11/04	Х							***************************************		
LDW-B10a-S**	L7505-8	sediment	08/25/04	х								**************************************	
LDW-C1-S**	L7505-9	sediment	08/26/04	Х									
LDW-C2-S2	L7505-10	sediment	08/26/04	Х									
LDW-C4-S	L7505-11	sediment	08/27/04	Х									
LDW-C6-S	L7505-12	sediment	08/26/04	х									
LDW-C7-S-1	L7505-13	sediment	08/26/04	X									
LDW-C8-S	L7505-14	sediment	08/26/04	х									
LDW-C9-S	L7505-15	sediment	08/25/04	Х									
LDW-C10-S-1	L7505-16	sediment	08/25/04	Х		***							
LDW-B1b-SDUP	L7505-1DUP	sediment	08/10/04	Х									
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PROTECTION AND ACTION OF THE PROTECTION OF THE P													

Note: X = Validation was performed.

13247VALA.wpd

Revision 1

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 PCB-77 PCB-99+101+113 PCB-105 PCB-110+115 PCB-114 PCB-118 PCB-123 PCB-126 PCB-129+138+160+163 PCB-153+168 PCB-156+157 PCB-180+193	0.108 ng/Kg 0.042 ng/Kg 0.236 ng/Kg 0.078 ng/Kg 0.289 ng/Kg 0.044 ng/Kg 0.195 ng/Kg 0.027 ng/Kg 0.041 ng/Kg 0.310 ng/Kg 0.257 ng/Kg 0.088 ng/Kg 0.160 ng/Kg	All samples in SDG DPWG15252

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 PCB-129+138+160+163 PCB-153+168 PCB-156+157 PCB-167 PCB-180+193 PCB-189	53.7 (≤50) 75.6 (≤50) 89.5 (≤50) 60.8 (≤50) 59.9 (≤50) 112 (≤50) 91.6 (<50)	J4 (all detects)	Α

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.

XIII. Overall Assessment of Data

The overall assessment of data was acceptable.

XIV. Field Duplicates

No field duplicates were identified in this SDG.

SDG Labo	#: 13247A3 #: DPWG15252 ratory: AXYS Analytical S	Servic	es, Ltd.		Level IJ			Date: <u>3/16/05</u> Page: /of/ Reviewer: 4 2nd Reviewer: **
The s		e rev	-					on findings are noted in attached
<u> </u>	Validation	Area		 	<u> </u>		Comm	ents
1.	Technical holding times			1	Sampling	dates: 8/10 -	27/0	4
11.	GC/MS Instrument perform	ance o	check	4		,	NRti	r lene 11
111.	Initial calibration			1	76 PS	> < 20	Į.	
IV.	Routine calibration			1	7000	525/35	•	
ν.	Blanks			W		are a constructive of the		
VI.	Matrix spike/Matrix spike du	plicat	es toub	N AW	not	Moil		
VII.	Laboratory control samples			-	OPR	SRM		
VIII.	Regional quality assurance	and q	uality control	N		1		
IX.	Internal standards			A		,	IR for	[enel]
X.	Target compound identifica	tions		A		<u></u>		1
XI.	Compound quantitation and	CRQ	Ls	A				,
XII.	System performance			Á	/			/
XIII.	Overall assessment of data			Kaw				
XIV.	Field duplicates			N				
XV.	Field blanks			N				
Note:	A = Acceptable N = Not provided/applicable SW = See worksheet ed Samples:	•	R = Rin:	o compounds sate eld blank	s detected	D = Duplic TB = Trip EB = Equi		
1	LDW-B1b-S	11	LDW-C4-S	· · · · · · · · · · · · · · · · · · ·	1 21	NE14745-	101	31
2	LDW-B10-S	12	LDW-C4-S		22	IN-TITITO	-/	32
3	LDW-B3b-S	13	LDW-C7-S-1		23			33

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LDW-B4b-S

LDW-B5a-S

LDW-B8a-S

LDW-B9b-S

LDW-C2-S2

LDW-B10a-S **

LDW-C1-S ##

LDW-C8-S

LDW-C9-S

LDW-C10-S-1

LDW-B1b-SDUP

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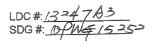
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VALIDATION FINDINGS CHECKLIST

Page: /of 3-2
Reviewer: d
2nd Reviewer:

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

Method: HRGC/HRMS Polychionnated Biphenyls (EPA Met	T		T	
Validation Area	Yes	No	NA NA	Findings/Comments
i. Technical holding times	T /	l	T	1
All technical holding times were met.	\vdash	 	├─	
Cooler temperature criteria was met.	14_	L	L	
# GC/MS Instrument performance check	T -		Ī	
Was PFK exact mass 380:9760 verified?	 		├	
Were the retention time windows established for all homologues?	1		-	
Is the static resolving power at least 10,000 (10% valley definition)?	14		_	
Was the mass resolution adequately check with PFK?	<u> </u>		L	
III: Initial calibration	T		I	I
Was the initial calibration performed at 5 concentration levels?	14	· · · ·	-	
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?	1			
Was the signal to noise ratio for each target compound \geq 2.5 and for each recovery and internal standard > 10?			<u> </u>	
IV. Continuing calibration				
Was a routine calibration performed at the beginning of each 12 hour period?	/			
Were all percent differences (%D) < 46% for unlabeled and labeled standards?	/		<u> </u>	
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.				out
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				



Page ≥of ≥ Reviewer: ↓ 2nd Reviewer: ▶

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?				<u> </u>
IX Internal standards				
Were internal standard recoveries within the 25-150% criteria?	1		 -	
Was the minimum S/N ratio of all internal standard peaks > 10?		l		
X Target compound identification		I	Γ	I
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?	4			
Was the Ion Abundance Ratio for the two quantitation ions within criteria?	4		<u> </u>	
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?	<u></u>			
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.			/	

PCB-1668.IV version 1.0

Ions Monitored for HRGC/HRMS Analysis of Polychlorinated Biphenyls

Descriptor	Accurate mass ^(a)	lon ID	Analyte	Substance
1	289.9224	м	C12 H6 35Cl4	тсв
	291.9194	M+2	C12 H6 35Cl3 37Cl4	TCB
	301.9626	М	13C12 H6 35CH	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	PeC8
	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	НрСВ
	395.7996	M+4	C12 H3 35Ci5 37Ci2	НрСВ
	405.8428	M+2	13C12 H3 35Cl6 37Cl	HpCB
	407.8398	M+4	13C12 H3 35CI5 37CI2	HpCB
	[354.9892[Lock	C9F13	PFK
3	509.7229	M+4	13C12 35Cl10 37Cl2	DCB
	511.7199	M+6	13C12 35CI9 37CI3	
	513.7170	M+8	13C12 35CI8 37CI4	
	[442.9728]	Lock	C10 F17	PFK

S = internal/recovery standard

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

³⁵Cl = 34.968853 ³⁷Cl = 36.965903

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

VALIDATION FINDINGS WORKSHEET <u>Blanks</u>

2nd Reviewer:_

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".

Were all samples associated with a method blank?

Was a method blank performed for each matrix and whenever a sample extraction was perform

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

Was method blank contamination less < CRQL for all target compounds? Blank extraction date: 1/25/05

Blank analysis date: 2/8/05 Conc. units: h 5/68

Associated samples:	management of the second secon
Sample Identification	

Compound	Blank ID								
W#1-	1745-101	All		T	Sample Identific	ation		and the second s	
PCB 66	0.108						Harton and the state of the sta		
- Company	0.042								A CONTRACTOR OF THE PROPERTY O
90+101+113	0.236	\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \	(
105	0.078								
110+(15	0.07c.28				 				
14	0.044		 	 	 				
118	0.195					***************************************		- Anna Salah Malayan Angada (Angada)	Bann (Alberta de Sancia de Caración de
123	0.027				<u> </u>				
126	0.041							- COMMITTEE - COMM	
1=9+138+160+163	0310								
153+168	0.257			<u> </u>				CO - CO	
. 1	0.088		-						All and the second seco
li 'II	0.160								*
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CIRCLED RESULTS WEDE NOT SU									

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 #: 1324 7A3	
SDG #: DPW415>52	-

VALIDATION FINDINGS WORKSHEET Matrix Spike/Matrix Spike Duplicates

	Page:_	
	Reviewer:	Ct.
2nd	Reviewer:	4
		7

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

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

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 PCF	90+101+113	()	()	53.T (550))	J4adots/A
		10B1=	7+138+160+	63 ()	()	756(1)		
		L PCB	153+168	())	89.5(1)		
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	20000001000000000000000000000000000000	per per		()	()	59.91		
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		þc þc	394189	()	()	91.61		V
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VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

	Page:
2nd	Reviewer: p

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:

 $RRF = (A_x)(C_{ts})/(A_{ts})(C_x)$ average RRF = sum of the RRFs/number of standards A_x = Area of compound,

 $A_{\mathbf{k}}$ = Area of associated internal standard

 $\hat{C_k}$ = Concentration of compound, $\hat{C_h}$ = Concentration of internal standard

%RSD = 100 * (S/X)

S = Standard deviation of the RRFs, X = Mean of the RRFs

				Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibration Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (initial)	RRF (std)	RRF (/ std)	%RSD	%RSD
1	1CAZ	2/4/05	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
		1	PCB-156 (¹⁵ C-PCB-156)	0.98		0.96	0.96	1.52	1.61
	***************************************		PCB-180 (13C-PCB-180)	0.79		0.79	0.79	2.0/	1.65
2			PCB-77 (¹³ C-PCB-77)					A STATE OF THE STA	
			PCB-105 (¹³ C-PCB-105)						
			PCB-156 (¹³ C-PCB-156)				**************************************		
	~~		PCB-180 (¹⁹ C-PCB-180)				and the state of t		
					-			AND CONTROL OF THE PROPERTY OF	
3			PCB-77 (¹³ C-PCB-77)					CONTROL OF THE PROPERTY OF THE	
			PCB-105 (¹³ C-PCB-105)					A CONTRACTOR OF THE PROPERTY O	
			PCB-156 (¹³ C-PCB-156)					- Commence of the contract of	Q-1020022200449000000000000000000000000000
	***************************************		PCB-180 (¹³ C-PCB-180)					***************************************	
				/L	<u> </u>				

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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VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

	Page:	
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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 $RRF = (A_{\kappa}/(C_{k})/(A_{k})(C_{\kappa})$

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

A = Area of compound,

A_s = Area of associated internal standard

 $C_v = Concentration of compound,$

C_k = Concentration of internal standard

		***************************************	**************************************		Reported	Recalculated	Reported	Recalculated
#	Standard ID	Calibratio n Date	Compound (Reference Internal Standard)	Average RRF (initial)	RRF-AW + (CC)	RAF SHI (CC)	, %D,	%D
1	PBSC-06T	2/9/	PCB-77 (¹³ C-PCB-77)	0.91	48.3	18.2		26
	1	2/8/15	PCB-105 (¹³ C-PCB-105)	0.83	49.3	49.2	- Q	1.6
			PCB-156 (¹³ C-PCB-156)	0.98	986	98.5	K	L =
			PCB-18q (13C-PCB-18q)	0.78	49.8	50.1	7	0.2
		47.400.000.000.000	•				[.Q'	
2	PBSC-069 .	- / ·	PCB-77 (¹³ C-PCB-77)	0.91	18.8	48.7	9	2:6
		2/9/05	PCB-105 (¹³ C-PCB-105)	0.83	505	50.2		0.4
		<i>'</i>	PCB-156 (¹³ C-PCB-156)	0.98	100	100		0
			PCB-189 (13C-PCB-189)	0.79	49.2	49.6	2	0.7
				1				A Commence of the Commence of
3			PCB-77 (¹³ C-PCB-77)					
	And the state of t		PCB-105 (¹³ C-PCB-105)			от на поставления на при н При на при н		antill kommune mengiring geografische statische statische in der den der en unsgewoorde statische der
			PCB-156 (¹³ C-PCB-156)				general menter del del legio e di servizio de acconstructivo con trataca del se acconstructivo en establista d	988 derth a deineanna a' deann i agu faigh ag dag 1990 deithill a 1989 a dhidh a ch ann a cein tu ag ag ann ag Tha ann ann ann an ann ann ann ann ann an
			PCB-180 (¹³ C-PCB-180)				A SECTION AND A SECTION ASSESSMENT OF THE PROPERTY OF THE PROP	reconstruction de la dissertation de la version de la construction de
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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 #: 13247A3 SDG #: 0PW&15252

VALIDATION FINDINGS WORKSHEET <u>Laboratory Control Sample Results Verification</u>

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2nd	Reviewer:	20

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

The percent recoveries (%R) and Relative Percent Difference (RPD) of the laboratory 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 = Laboractry control sample percent recovery

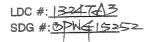
LCSD = Laboratory control sample duplicate percent recovery

LCS ID: OPR

Spike		Spiked S	Sample	mple LCS		LCSD		LCS/LCSD		
Compound	(NS	ded w ()	Concentration (N 5 M) Percent Recovery		Percent Recovery		RPD			
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated
PCB-77	50!	NĂ	48.8	NA	97.6	97.6			200000000000000000000000000000000000000	The second secon
PCB-81			48.5		97.0	at.0		***************************************		De-OPENIER RECOGNISION RECOGNISION CONTRACTOR CONTRACTO
PCB-105			49.4		98.9	98.8			AND THE RESIDENCE OF THE PARTY	an een Pilokata dista tiista marka sanna ayna tasaa sanaa ahaa ahaa siira dhaanna dh
PCB-114			49.1		98.1	98.2		and the consequence of the conse		······································
PCB-118			50.0		100	100		~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~		
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PCB-126	V		48.6		97.2	97. ²		***************************************		
PCB-156 + 185T	100		aT.9		97.9	97.9			petite et filler studen for til år stelste som produkt i 4-4-4-4-4-4-4-4-6-asset siddokusen.	MOTOR AND AND STATEMENT OF COURT AND STATEMENT AND
PCB-157									ymatus al Tawan nyayayi iyoo aa oo o	
PCB-167	50,0		19.6		99.1	99.2		***************************************	and An reco logic scotter concept a view to high the state the control of the state of the stat	SACONIA DILA CERA CERA CERA CERCA CONTRA CONTRA CONTRA CERCA CONTRA CERCA CERC
PCB-169	lacksquare		49.3		98.6	98.6		THE COMMENTS ASSESSED TO SECURITY OF THE COMMENTS OF THE COMME		**************************************
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PCB-189	50.0	V	49.7		99.5	99.4		***************************************		
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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.

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VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

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2nd reviewer:	

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 = $\frac{(A_{\bullet})(I_{\bullet})(DF)}{(A_{\bullet})(RRF)(V_{\bullet})(\%S)}$

A_x = Area of the characteristic ion (EICP) for the compound to be measured

 A_{la} = Area of the characteristic ion (EICP) for the specific internal standard

 $l_a = Amount of internal standard added in nanograms (ng)$

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 , POST 7:

Conc. = (3.3 + 0.7)(2000)(1). (1.360 + 0.0)(0.9)(10.8)(0.9)

= 49.53 ng/s

matrices or uy.					
#	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
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