APPENDIX C DATA VALIDATION REPORT





LABORATORY DATA CONSULTANTS, INC.

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

LDC #15449 September 26, 2006

Windward Environmental, LLC 200 West Mercer Street, Suite 401 Seattle, WA 98119

ATTN: Ms. Marina Mitchell

SUBJECT: Lower Duwamish Waterway Group Fish Tissue Sample Data

Validation

Dear Ms. Mitchell,

Enclosed is our final EPA 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: DPWG19975/WG19626. 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

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

CHEMICAL DATA QUALITY REVIEW FOR FISH TISSUE SAMPLES

Lower Duwamish Waterway Group LDC# 15449

This report details the findings of an EPA 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: DPWG19975/WG19626. 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 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 Fish and Crab Collection and Chemical Analyses Quality Assurance Project Plan (August 27, 2004). Where specific guidance is not available, the data has been evaluated in a conservative manner 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 Replicates

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				Attachme	nt 2										
SDG# : DPWG19975/WG19626			VALIDAT	ION SAM	IPLE T	ABLE							Li	DC#: 1	 5449/
Project Name: Lower Duwamish Wate	rway Group		Paramete	ers/Analy	tical Me	thod							Project		
Client ID #	Lab ID #	Matrix	Date Collected	PCBs Cong. (1668A)											
LDW-05-T2-B-SS-WB-COMP1	L9071-1	tissue	09/01/05	Х										:	
LDW-05-T3-D-SS-WB-COMP1	L9071-2	tissue	08/31/06	Х											
LDW-05-T1-M-ES-WB-COMP3	L9071-3	tissue	08/29/06	Х											
LDW-05-T2-M-ES-WB-COMP3	L9071-4	tissue	09/01/06	Χ			1, .		. :						
LDW-05-T3-M-ES-WB-COMP2	L9071-5	tissue	09/01/06	Х											
LDW-05-T1-B-SS-WB-COMP1	L9071-6	tissue	08/31/06	Х											
LDW-05-T3-M-ES-WB-COMP2DUP	L9071-5DUP	tissue	09/01/06	×											
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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.
 - 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

I. Method Compliance

The ion abundance ratio for PCB-169 in the lowest standard of the initial calibration did meet the method QC limits.

II. Usability

The quality control criteria reviewed were met and are considered acceptable. Based upon the data validation all results are considered valid and usable for all purposes.

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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 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).

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 ion abundance ratios for all PCBs were within validation criteria with the following exceptions:

SDG	Date	Compound	Ion Abundance Ratio (Limits)	Associated Samples	Flag	A or P
DPWG19975/ WG19626	5/5/06 (CS0 Standard)	PCB-169	0.99 (1.05-1.43)	LDW-05-T2-B-SS-WB-COMP1 LDW-05-T3-D-SS-WB-COMP1 LDW-05-T1-M-ES-WB-COMP3 LDW-05-T2-M-ES-WB-COMP3	NA	-

N/A = Not applicable

For the result above flagged "Not applicable", since the ion abundance ratio in the continuing calibration was within the method QC limits, this finding did not warrant the qualification of the data.

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.

IV. Routine Calibration (Continuing)

Routine calibration was performed at the required frequency.

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.

The ion abundance ratios for all PCBs were within validation criteria.

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:

			N 2		
SDG	Method Blank ID	Extraction Date	Compound	Concentration	Associated Samples
	meared Blank is	Date	Compound	Concentration	Associated Samples
DPWG19975/	WG19626-101	7/14/06	PCB-18	0.802 ng/Kg	LDW-05-T2-B-SS-WB-COMP1
WG19626			PCB-20	1.70 ng/Kg	LDW-05-T3-D-SS-WB-COMP1
			PCB-40	0.638 ng/Kg	LDW-05-T1-M-ES-WB-COMP3
			PCB-44	2.67 ng/Kg	LDW-05-T2-M-ES-WB-COMP3
			PCB-45 PCB-49	0.870 ng/Kg 1.66 na/Ka	LDW-05-T3-M-ES-WB-COMP2 LDW-05-T1-B-SS-WB-COMP1
	Alternative Control	1.74	PCB-49	0.381 ng/Kg	LDW-05-T1-B-SS-WB-COMP1
]			PCB-52	2.54 ng/Kg	LEDVV-03-13-WI-LO-VVD-COIVII ZEO(*
			PCB-59	0.362 ng/Kg	
			PCB-60	0.608 ng/Kg	:
			PCB-61	2.81 ng/Kg	· I
	,		PCB-64 PCB-66	0.414 ng/Kg	
		-	PCB-00 PCB-82	1.60 ng/Kg 0.782 ng/Kg	
ļ			PCB-83	1.81 ng/Kg	
1			PCB-86	0.766 ng/Kg	
11 st 55 m		1.	PCB-88	0.472 ng/Kg	
			PCB-90	4.46 ng/Kg	,
		·	PCB-92	0.826 ng/Kg	*
			PCB-93 PCB-105	4.20 ng/Kg	
	egyn, in an		PCB-103	1.07 ng/Kg 0.577 ng/Kg	
			PCB-110	2.05 ng/Kg	
			PCB-118	2.28 ng/Kg	.
			PCB-128	0.498 ng/Kg	
	1		PCB-129	2.46 ng/Kg	
·			PCB-131	0.440 пд/Кд	·
			PCB-132 PCB-135	0.704 ng/Kg	
			PCB-137	1.61 ng/Kg 0.424 ng/Kg	
			PCB-141	0.745 ng/Kg	ĺ
	•		PCB-146	1.02 ng/Kg	
·			PCB-147	1.72 ng/Kg	
ļ			PCB-153	2.72 ng/Kg	
			PCB-156 PCB-167	1.13 ng/Kg	
	İ		PCB-107 PCB-170	0.392 ng/Kg 0.526 ng/Kg	į
			PCB-180	1.47 ng/Kg	
		i	PCB-183	0.412 ng/Kg	
.			PCB-187	0.662 ng/Kg	
. .			PCB-190	0.735 ng/Kg	
			PCB-194 PCB-198	0.757 ng/Kg	į
	ļ		PCB-198 PCB-81	0.575 ng/Kg 0.115 ng/Kg	
			PCB-169	0.115 ng/kg 0.079 ng/Kg	
j			Total tetrachlorobiphenyls	5.05 ng/Kg	•
			Total pentachlorobiphenyls	2.28 ng/Kg	
	•		Total hexachlorobiphenyls	4.44 ng/Kg	
		j	Total octachlorobiphenyls	0.757 ng/Kg	
		İ	Total PCBs	12.5 ng/Kg	

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

No field blanks were identified in this SDG.

VI. Matrix Spike/Matrix Spike Duplicates

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

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

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.

XI. Compound Quantitation and CRQLs

All compound quantitation and CRQLs were within validation criteria with the following exceptions:

SDG	Sample	Compound	Finding	Criteria	Flag	A or P
DPWG19975/ WG19626	LDW-05-T2-M-ES-WB-COMP3	PCB-118	Sample result exceeded calibration range.	Reported result should be within calibration range.	J2 (all detects)	P

XII. System Performance

The system performance was acceptable.

XIII. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

XIV. Field Replicates

No field replicates were identified in this SDG.

Lower Duwamish Waterway Group Polychlorinated Biphenyls - Data Qualification Summary - SDG DPWG19975/WG19626

SDG	Sample	Compound	Flag	AorP	Reason
DPWG19975/ WG19626	LDW-05-T2-M-ES-WB-COMP3	PCB-118	J2 (all detects)	P	Compound quantitation and CRQLs

Lower Duwamish Waterway Group
Polychlorinated Biphenyls - Laboratory Blank Data Qualification Summary - SDG
DPWG19975/WG19626

No Sample Data Qualified in this SDG

	#: 15449A3 VALIDATI #: DPWG19975/WG19626 ratory: AXYS Analytical Services, Ltd.		PLETENESS T Level IV	WORKSHEET	Da Pag Reviewe	
METI	HOD: HRGC/HRMS Polychlorinated Bip	ohenyl Conge	eners (EPA Meth	nod 1668A)	2nd Reviewe	er:
Thee	amples listed below were reviewed for	each of the f	(a. 11 a	on areas. Validation	findings are noted in	
valida	ation findings worksheets.	each of the K	T	JII aleas. Validation	illidings are noted i	1 attached
valida	ation findings worksheets.	each of the R	Ollowing validation	Commer		1 attached
valida	ation findings worksheets.		Sampling dates:			1 attached
valida	Validation Area			Commer	nts	i attachet
Valida	Validation Area Technical holding times		Sampling dates:	Commer 8/29 - 9/1/	nts	1 attached

	Trouble completion		100 5 00 / 35 (WA	ine/ Labeled)
V.	Blanks	w	/	A Street
VI.	Matrix spike/Matrix spike duplicates DUP	NA		
VII.	Laboratory control samples	4	LES. CRM	
VIII.	Regional quality assurance and quality control	N		
IX.	Internal standards	MATA		
Χ.	Target compound identifications	4		tang terminal
XI.	Compound quantitation and CRQLs	W.A.		
XII.	System performance	4		indian distriction of the second
XIII.	Overall assessment of data	1		
XIV.	Field duplicates	N)		
XV.	Field blanks	N		2.30, 120

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:

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1	LDW-05-T2-B-SS-WB-COMP1	11	W419626-101	21	31	
2	LDW-05-T3-D-SS-WB-COMP1	12	1	22	32	
3	LDW-05-T1-M-ES-WB-COMP3	13		23	33	
4	LDW-05-T2-M-ES-WB-COMP3	14		24	34	
5	LDW-05-T3-M-ES-WB-COMP2	15		25	35	
6	LDW-05-T1-B-SS-WB-COMP1	16		26	36	
7	LDW-05-T3-M-ES-WB-COMP2DUP	17		27	37	
8		18		28	38	
9		19		29	39	
10		20		30	40	

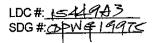
LDC #: 1544913 SDG #: DPWG19975

VALIDATION FINDINGS CHECKLIST

Page: /of 2
Reviewer: 4
2nd Reviewer: A

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

Validation Area	Yes	No	NA	Findings/Comments
t: Technical holding times				
All technical holding times were met.				
Cooler temperature criteria was met.		_		
II; GC/MS instrument performance check				
Was PFK exact mass 380,9760 verified?	_			
Were the retention time windows established for all homologues?	<i></i>			
Is the static resolving power at least 10,000 (10% valley definition)?			·	
Was the mass resolution adequately check with PFK?				_
III. initial calibration				
Was the initial calibration performed at 5 concentration levels?				
Were all percent relative standard deviations (%RSD) ≤ 25% for unlabeled standards and < 30% for labeled standards?		<u>.</u>		The second secon
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?				
fV. Continuing satisfation.				
Was a routine calibration performed at the beginning of each 12 hour period?		_		
Were all percent differences (%D) < 40% for unlabeled and labeled standards?				
Did all routine calibration standards meet the Ion Abundance Ratio criteria?				
V. Blanks				
Was a method blank associated with every sample in this SDG?				
Was a method blank performed for each matrix and concentration?	4			
Was there contamination in the method blanks? If yes, please see the Blanks validation completeness worksheet.		-		
VI. Mairix 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				



VALIDATION FINDINGS CHECKLIST

Page: 2 of 2 Reviewer: 4 2nd Reviewer:

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			The second secon
Were internal standard recoveries within the 25-150% criteria?	/			
Was the minimum S/N ratio of all internal standard peaks > 10?	<u> </u>			
X. Target compound identification	Ι			
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?				The state of the s
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 lons listed in the table attached?				
Was the Ion Abundance Ratio for the two quantitation ions within criteria?				
Was the signal to noise ratio for each target compound and labeled standard > 2.5?		,		
Does the maximum intensity of each specified characteristic ion coincide within ± 2 seconds (includes labeled standards)?				
Was an acceptable lock mass recorded and monitored?				
XI: Compound quantitation/CRQLs				
Were the correct internal standard (IS), quantitation ion and relative response factor (RRF) used to quantitate the compound?	/	_		
Were compound quantitation and CRQLs adjusted to reflect all sample dilutions and dry weight factors applicable to level IV validation?				
X(I. System performance				
System performance was found to be acceptable.		`		
XIII. Overall assessment of data				
Overall assessment of data was found to be acceptable.				
XIV. Field duplicates				
Field duplicate pairs were identified in this SDG.				
Target compounds were detected in the field duplicates.				
XV. Field blanks				
Field blanks were identified in this SDG.				
Target compounds were detected in the field blanks.				

LDC #:<u>15449A3</u>

VALIDATION FINDINGS WORKSHEET Initial Calibration

Page:_	_/of/_
Reviewer:	9-
nd Reviewer:_	A

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".

N N/A

Was the initial calibration performed at 5 concentration levels?

Were all percent relative standard deviations (%RSD) < 85% for unlabeled standards and labeled standards?

Did all calibration standards meet the Ion Abundance Ratio criteria? YN_N/A YAN N/A

Was the signal to noise ratio for each target compound \geq 2,5 and for each recovery and internal standard \geq 10? N/A

#	Date	Standard ID	Compound	Finding %RSD		Finding Ion Abundace Ratio		ociated Samples		Qualifications
	\$506		PCB169			0.99 (1.05-1.4	3)	1-4, zek	NA.	Fox + No lava
	<u> </u>	(CSO)								1 - 700 100001
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		Halogen §	Selected ions (m/z)	Ion Abundace Ratio		Halogen		Selected ions (n	n/z)	Ion Abundace Ratio
	1 CI		M/M+2	2.66-3,60		7 CI		M/M+2	<u> </u>	0.37-0.51
	2 CI		M/M+2	1.33-1.81		7 CI		M+2/M+4		0.88-1.20
	3 CI		M/M+2	0.88-1.20		8 CI		M+2/M+4		0.76-1.02
	4 CI		M/M+2	0,65-0,89		9 CI		M/M+2		1.14-1.54
	5 CI		M+2/M+4	1.32-1.78		9 CL		M/M-2		0.66-0.90
	6 CI		M/M+2	0.43-0,59		10 CL		M/M+2		0.99-1.35
	6 CI		M+2/M+4	1.05-1.43		<u> </u>				1.00

LDC #:15	41913
	PW&19975

VALIDATION FINDINGS WORKSHEET **Blanks**

	Page:_	_/of /
	Reviewer:	
2nd	Reviewer:	N

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? ON N/A

Was a method blank performed for each matrix and whenever a sample extraction was performed? Was method blank contamination less < CRQL for all target compounds? AM N(X)

Blank extraction date: 7/14/06

Conc. units: notes

Blank analysis date: 7/24/06 Associated samples:

Compound	Blank ID					ample Identifi				
uf	19626-101	All			T = 	ambie ideutiti	ation			
see attachment		75X					<u> </u>			
		- (5/	 	<u> </u>						
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	.									
		 								
ED RESULTS WERE NOT QU					_ [

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".

VANG	METHOD	MIL A.	ስ4ሰ	Day 00

Form 1A PCB CONGENER ANALYSIS REPORT CLIENT SAMPLE NO. Lab Blank Sample Collection:

AXYS ANALYTICAL SERVICES

P.O. Box 2219, 2045 MILLS RD. WEST, SIDNEY, B.C., CANADA V8L 3S8 TEL (250) 655-5800 FAX (250) 655-5811

4033

Project No.

N/A

Contract No.:

Lab Sample I.D.:

WG19626-101

Matrix:

Sample Size:

5.00 g

Sample Receipt Date:

N/A

Initial Calibration Date:

05-Jun-2006

Extraction Date:

14-Jul-2006

CORN OIL

instrument ID:

HR GC/MS

Analysis Date:

24-Jul-2006 Time: 11:52:21

GC Column ID:

SPB OCTYL

Extract Volume (uL):

400

Sample Data Filename:

PB6C_327 S: 5

injection Volume (uL):

1.0

Blank Data Filename:

PB6C 327 S: 5

Dilution Factor:

20

Cal. Ver. Data Filename:

PB6C_327 S: 1

Concentration Units:

ng/kg

COMPOUND	IUPAC NO.	CO-ELUTIONS	5x	LAB FLAG ¹	CONC. FOUND	REPORTING LIMIT	IQN ABUND.	RRT
and the sale		•					RATIO	
	200		:	4.0				
2-MoCB	1			UD		0.472		
3-MoCB	2	**		UD		0.546	-	
4-MoCB	3			UD		0.664		
2,2'-DiCB	4			UD	+ 1 +	2.97		1.
2,3-DICB	5			UD		2.46		
2,3'-DICB	6			UD		2.26		
2,4-DiCB	7	to the second		ÜD		2.32		
2,4'-DICB	. 8			UD		2.14		
2,5-DiCB	9			UD.		2,24		
2,6-DiCB	10			· U D		2.31		
3,3'-DICB	- 11	•		UD		2.37		
3,4-DiCB	.12	12 + 13		CUD		2.47		
3,4'-DICB	13	12 + 13		C12				
3,5-DICB	14			UD		2.31		
4,4'-DICB	15		-	UD		3.15		
2,2',3-TriCB	16			UD		1.09		
2,2',4-TriCB	17		_	UD		0.922		
2,2',5-TriCB	18	18 + 30	4.01	CKDJ	0.802	0.771	1.51	1.112
2,2',6-TriCB	19			UD	•	1.07		
2,3,3'-TriCB	20	20 + 28	8,50	CKDJ	1.70	0.982	0.83	0.848
2,3,4-TrICB	21	21 + 33		CUD		0.955		
2,3,4'-TriCB	22		•	ŲD		1.07		
2,3,5-TrICB	23			UÐ		1.02		
2,3,6-TrICB	- 24	•		UВ		0.693		
2,3',4-TriCB	25			UD.		0.879		
2,3',5-TriCB	26	26 + 29		CUD		0.980		
2,3',6-TriCB	27			UD		0.634		
2,4,4'-TriCB	28	20 ÷ 28		C20			•	
2,4,5-TriCB	29	26 + 29		C26				
2,4,6-TriCB	30	18 + 30	٠.	C18				
2,4',5-TriCB	31	-		UD		0.934		
2,4',6-TriCB	32			ŲD		0.962		
2',3,4-TrICB	33	21 + 33		C21		÷		
2',3,5-TriCB	34			UD		0.999		
3,3',4-TriCB	35			UD		1.10		
3,3',5-TriCB	36			UD		0.987		
3,4,4'-TriCB	37			ŲĎ		1.18		
3,4,5-TrICB	38			ŲΦ		1.03		

1.759

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COMPOUND	IUPAC NO.	J 7 1	LAB CONC.	REPORTING LIMIT	ION ABUND.	RRT
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1					RATIO	
3,4",5-TriCB	.39	* .	UD	4.03		
2,2',3,3'-TeCB	40	40+41+71 319	CDJ 0.638	1.02 0.269	0.70	4 224
2,2',3,4-TeCB	41		C40	0.209	0.70	1.334
2,2',3,4'-TeCB	42		UD	0.282		
2,2',3,5-TeCB	43		IID.	0.328		
2,2',3,5'-TeCB	44	44+47+65 (3.35°C		0.245	0.63	1.285
2,2',3,6-TeCB	45	45+51 4.35 C	KDJ 0.870	0.274	1.08	1.145
2,2',3,6'-TeCB 2,2',4,4'-TeCB	46		UD	0.316		
2,2',4,5-TeCB	47 48		C44			
2,2',4,5'-TeCB	49		UD AGG	0.274		
2,2',4,6-TeCB	50	50+53 1.905C	KDJ 1.66 KDJ 0.381	0.229 0.266	0.58 3.90	1.258
2,2',4,6'-TeCB	51	45 + 51	C45	0.200	3.50	1.110
2,2',5,5'-TeCB	52		(DJ 2.54	0.258	1.24	1.233
2,2',5,6'-TeCB	53		C50	1,44		,,,,,,
2,2',6,6'-TeCB	54		סט	0.244		
2,3,3',4-TeCB	55		UD '	0.533		
2,3,3',4'-TeCB	56 =7		UD	0.518		
2,3,3',5-TeCB 2,3,3',5'-TeCB	57 58		UD	0.494		
2,3,3',6-TeCB	59	1	UD	0.507		
2,3,4,4'-TeCB	60		KDJ 0.362 CDJ 0.608	0.204	0.96	1.300
2,3,4,5-TeCB	61		DJ 2,81	0.535 0.492	1.24 0.73	0.912 0.875
2,3,4,6-TeCB	62		C59	0.402	0.73	0.075
2,3,4',5-TeCB	63	· .	JD	0.476		
2,3,4',6-TeCB	64	2.07 K	DJ 0.414	0.196	3.38	1.347
2,3,5,6-TeCB	65		244			
2,3',4,4'-TeCB	. 66		DJ 1.60	0.499	0.82	0.885
2,3',4,5-TeCB	67		ם נ	0.431		
2,3',4,5'-TeCB 2,3',4,6-TeCB	68 69		J D	0.477		
2,3',4',5-TeCB	70	· ••	249	-		
2,3',4',6-TeCB	71		261 240			
2,3',5,5'-TeCB	72	· · · · · · · · · · · · · · · · · · ·	JD.	0.469		
2,3',5',6-TeCB	73		J D	0.199		
2,4,4',5-TeCB	74	**	61	******		
2,4,4',6-TeCB	75	59 + 62 + 75 C	359	*		
2',3,4,5-TeCB	76	61 + 70 + 74 + 76 C	61			
3,3',4,4'-TeCB	77 70	•	I D	0.534		
3,3',4,5-TeCB	78 79		I.D	0.532		
3,3',4,5'-TeCB 3,3',5,5'-TeCB	80		ID	0.437		
3,4,4',5-TeCB	81		D X	0.463		
2,2',3,3',4-PeCB	82	<i>-</i> 3 ·	A DJ 0.782	0.464	0.62	0.024
2,2',3,3',5-PeCB	83	83+99 <i>9.05</i> CK		0.404	0.62 0.73	0.934 0.885
2,2',3,3',6-PeCB	84	U	D	0.471		0.000
2,2',3,4,4'-PeCB	85	85 + 116 + 117 86 + 87 + 97 + 108 + 119 + 125 38 3 CK 86 + 87 + 97 + 108 + 119 + 125 CK	סע	0.345		
2,2',3,4,5-PeCB	86	86 + 87 + 97 + 108 + 119 + 125 30 CK	DJ 0.766	0.358	1.87	0.899
2,2',3,4,5'-PeCB	87					
2,2',3,4,6-PeCB	88	88 + 91 2.36 CK		0.417	0.38	1.151
2,2',3,4,6'-PeCB 2,2',3,4',5-PeCB	89 90	ں کے چے 3 ck		0.436		
2,2',3,4',6-PeCB	91			0.363	1.28	0.869
2,2',3,5,5'-PeCB	92	4.13 KI	88 D.I. 0.926	0.424	224	A 0E2
2,2',3,5,6-PeCB	93	93+95+98+100+102 ⊃ CK		0.421 0.404	2.34 1.82	0.853 1.123
2,2',3,5,6'-PeCB	94	U		0.455	1.02	1.120
2,2',3,5',6-PeCB	95	93 + 95 + 98 + 100 + 102 C		JJJ		
2,2',3,6,6'-PeCB	96	ឋ		0.247		
2,2',3',4,5-PeCB	97	86 + 87 + 97 + 108 + 119 + 125 C8				
2,2',3',4,6-PeCB	98	93 + 95 + 98 + 100 + 102 CS				
2,2',4,4',5-PeCB	99 400	83 + 99 CS				
	100 101	93 + 95 + 98 + 100 + 102 CS				
	102	90 + 101 + 113 CS 93 + 95 + 98 + 100 + 102 CS				
,- ; ·,-,- 1 00D		93 + 95 + 98 + 100 + 102 CS				

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COMPOUND	IUPAC NO.	CO-ELUTIONS	5X LAB	CONC. FOUND	REPORTING LIMIT	ION ABUND. RATIO	RRT
2,2',4,5',6-PeCB	103		UD		0.378		
2,2',4,6,6'-PeCB	104		UD		0.277		
2,3,3',4,4'-PeCB	105		5,35 KDJ	1.07	0.458	2.47	1.000
2,3,3',4,5-PeCB	106	40m - 404	UD		0.415		
2,3,3',4',5-PeCB 2,3,3',4,5'-PeCB	107 108	107 + 124 86 + 87 + 97 + 108 + 119	CUD +125 C86		0.428		
2,3,3',4,6-PeCB	109	00 . 07 . 37 + 108 + 113	_3.88€kDJ	0.577	0.384	3.41	0.997
2,3,3',4',6-PeCB	110	110 + 115	O. ZSCKDJ	2.05	0.303	2.24	0.925
2,3,3',5,5'-PeCB	111		UD	•	0.312		0.040
2,3,3',5,6-PeCB	112	00 . 404 . 446	UD		0.303		
2,3,3',5',6-PeCB 2,3,4,4',5-PeCB	. 113 114	90 + 101 + 113	C90 U D		0.444		
2,3,4,4',6-PeCB	115	110 + 115	C110		0.414		
2,3,4,5,6-PeCB	116	85 + 116 + 117	C85	•			
2,3,4',5,6-PeCB	117	85 + 116 + 117	C85				
2,3',4,4',5-PeCB 2,3',4,4',6-PeCB	118 119	86 + 87 + 97 + 108 + 119 +	11.4 DJ	2.28	0.428	1.60	1.000
2,3',4,5,5'-PeCB	120	00 + 01 + 91 + 100 + 119 +	+ 125 C86 U D		0.295		
2,3',4,5',6-PeCB	121		ÜD		0.317		
2',3,3',4,5-PeCB	122		ם ט		0.451		
2',3,4,4',5-PeCB	123	407 . 404	U D		0.561		•
2',3,4,5,5'-PeCB 2',3,4,5,6'-PeCB	124 125	107 + 124 86 + 87 + 97 + 108 + 119 +	C107 + 125 C86				
3,3',4,4',5-PeCB	126	. 00 -01 + 91 + 100 + 119 +	7 125 Cob X		al .		
3,3',4,5,5'-PeCB	127	2.475	UD		0.440		
2,2',3,3',4,4'-HxCB	128	128 + 166	P.47 CKDJ	0.498	0.253	5.15	0.959
2,2',3,3',4,5-HxCB 2,2',3,3',4,5'-HxCB	129 130	129 + 138 + 160 + 163		2.46	0.254	0.93	0.929
2,2',3,3',4,6-HxCB	131		≥,2 KDJ	0.440	0.317 0.301	4 52	1 160
2,2',3,3',4,6'-HxCB	132		3 ₹5 KDJ	0.704	0.301	1.52 0.51	1.160 1.174
2,2',3,3',5,5'-HxCB	133		UD		0.289		****
2,2',3,3',5,6-HxCB	134	134 + 143	a o CUD		0.297		
2,2',3,3',5,6'-HxCB 2,2',3,3',6,6'-HxCB	135 136	135 + 151 + 154	& ~ > CKD1	1.61	0.338	0.96	1.102
2,2',3,4,4',5-HxCB	137		≥ 1 ≥ KDJ	0.424	0.264 0.291	0.21	0.918
2,2',3,4,4',5'-HxCB	138	129 + 138 + 160 + 163		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	0.201		0.010
2,2',3,4,4',6-HxCB	139	139 + 140	CUD		0.265		
2,2',3,4,4',6'-HxCB 2,2',3,4,5,5'-HxCB	140 141	139 + 140	272€ C139	0.745	0.070		
2,2',3,4,5,6-HxGB	142		S (Z KD)	0.745	0.273 0.307	0.88	0.904
2,2',3,4,5,6'-HxCB	143	134 + 143	C134		0.001		
2,2',3,4,5',6-HxCB	144		· UD		0.347		
2,2',3,4,6,6'-HxCB 2,2',3,4',5,5'-HxCB	145 146		UD VO		0.277		
2,2',3,4',5,6-HxCB	147	147 + 149	5.1 KDJ 8.6 CDJ	1.02 1.72	0.249 0.262	1.03 1.20	0.885 1.133
2,2',3,4',5,6'-HxCB	148	(10	8.6 CDJ	1,12	0.356	1.20	1.100
2,2',3,4',5',6-HxCB	149	147 + 149	C147		•		
2,2',3,4',6,6'-HxCB 2,2',3,5,5',6-HxCB	150	405 - 454 - 454	UD		0.265		
2,2',3,5,6,6'-HxCB	151 152	135 + 151 + 154	C135 U D	•	0.257		
2,2',4,4',5,5'-HxCB	153	153 + 168	13.6cbj	2.72	0.257 0.222	1.21	0.899
2,2',4,4',5,6'-HxCB	154	135 + 151 + 154	C135				5,000
2,2',4,4',6,6'-HxCB	155	450 . 450	- 65 UD		0.248		
2,3,3',4,4',5-HxCB 2,3,3',4,4',5'-HxCB	156 157	156 + 157 156 + 157	5,65 CKDJ	1.13	0.294	0.88	1.000
2,3,3',4,4',6-HxCB	158	156 + 157	C156 U D		0.198		
2,3,3',4,5,5'-HxCB	159		āŪ		0.138		
	160	129 + 138 + 160 + 163	C129				
2,3,3',4,5',6-HxCB 2,3,3',4',5,5'-HxCB	161 162		UD		0.209		
2,3,3',4',5,6-HxCB	163	129 + 138 + 160 + 163	U D C129		0.222		
2,3,3',4',5',6-HxCB	164		UD		0.212		
2,3,3',5,5',6-HxCB	165		UD		0.233		
2,3,4,4',5,6-HxCB	166	128 + 166	C128				

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COMPOUND	IUPAC NO.		CO-ELUTIONS	5X	LAB FLAG ¹	CONC. FOUND	REPORTING LIMIT	ION ABUND. RATIO	RRT
2,3',4,4',5,5'-HxCB	. 167			1.96	ĶDJ	0.392	0.210	0.54	1.001
2,3',4,4',5',6-HxCB	168		153 + 168	1 - 4 -	C153			0.04	1.001
3,3',4,4',5,5'-HxCB	169			:	X				
2,2',3,3',4,4',5-HpCB	170			> 63	KDJ	0.526	0.407	2.94	0.936
2,2',3,3',4,4',6-HpCB	171		171 + 173		CUD	51010	0.389	2.04	0.830
2,2',3,3',4,5,5'-HpCB	172		•		UD		0.393		
2,2',3,3',4,5,6-HpCB	173		171 + 173		C171		0.000		
2,2',3,3',4,5,6'-HpCB	174				UD		0.356		
2,2',3,3',4,5',6-HpCB	175				UD		0.343		
2,2',3,3',4,6,6'-HpCB	176				UD		0.262		
2,2',3,3',4',5,6-HpCB	177		* .		UD		0.370		
2,2',3,3',5,5',6-HpCB	178			*	UD		0.365		
2,2',3,3',5,6,6'-HpCB	179				UD		0.255		
2,2',3,4,4',5,5'-HpCB	180		180 + 193	735	CKDJ	1.47	0.309	2.04	0.910
2,2',3,4,4',5,6-HpCB	181			(* -	מט		0.367	2.04	0.510
2,2',3,4,4',5,6'-HpCB	182				ÜÞ	_	0.336		100
2,2',3,4,4',5',6-HpCB	183		183 + 185	206	CKDJ	0.412	0.349	0.65	1.127
2,2',3,4,4',6,6'-HpCB	184				UD		0.254	0.00	1.121
2,2',3,4,5,5',6-HpCB	185		183 + 185		C183		******		
2,2',3,4,5,6,6'-HpCB	186		· **		III D		0.271		
2,2',3,4',5,5',6-HpCB	187	*.		3319	KDJ	0.663	0.326	0.43	1.109
2,2',3,4',5,6,6'-HpCB	188	7.	Tage 1	٠,٠	UD		0.267	0.10	1.103
2,3,3',4,4',5,5'-HpCB	189		e e e e e e e e e e e e e e e e e e e		U.D.	-	0.529	•	
2,3,3',4,4',5,6-HpCB	190			3.675	>KDJ	0.735	0.302	1.52	0.947
2,3,3',4,4',5',6-HpCB	191			7 Th	סט		0,291	1102	5.047
2,3,3',4,5,5',6-HpCB	192			to the second	ם ט		0.316		-
2,3,3',4',5,5',6-HpCB	193		180 + 193	9.	_ C180				
2,2',3,3',4,4',5,5'-OcCB	194			3.785	DJ	0.757	0.572	0.92	0.992
2,2',3,3',4,4',5,6-OcCB	195				UD		0.595		0.002
2,2',3,3',4,4',5,6'-OcCB	196				UD		0.445		
2,2',3,3',4,4',6,6'-OcCB	197		197 + 200	97.	-CUD		0.317		
2,2',3,3',4,5,5',6-OcCB	198		198 + 199	ع) ۵ ز د	CKDJ	0.575	0.461	2.04	1.115
2,2',3,3',4,5,5',6'-OcCB	199		198 + 199	1, 14	C198	3.3.,5			
2,2',3,3',4,5,6,6'-OcCB	200		197 + 200		C197				
2,2',3,3',4,5',6,6'-OcCB	201				UD		0.313		
2,2',3,3',5,5',6,6'-OcCB	202				UD		0.403		
2,2',3,4,4',5,5',6-OcCB	203		•		UD		0.430		
2,2',3,4,4',5,6,6'-OcCB	204				UD		0.315		
2,3,3',4,4',5,5',6-OcCB	205			•	UD		0.472		
2,2',3,3',4,4',5,5',6-NoCB	206				UD		2.12		
2,2',3,3',4,4',5,6,6'-NoCB	207				ΩŪ		1.48		
2,2',3,3',4,5,5',6,6'-NoCB	208				UD		1.69		1.
2,2',3,3',4,4',5,5',6,6'-DeCB	209				UD		0.705		
					00		U.705		

(1) Where applicable, custom lab flags have been used on this report; U = not detected; K = peak detected but did not meet quantification criteria, result reported represents the estimated maximum possible concentration; D = dilution data; J = concentration less than LMCL; C = co-eluting congener; X =

Approved by:	Teresa	Rawsthorne	QA/QC Chemist
			CACC CREITISI

For Axys Internal Use Only [XSL Template: Form16681 A.xst; Created: 28-Aug-2006 10:00:20; Application: XMLTransformer-1.7.9; Report Filename: 1668_PCB1668_PCBTF_WG19626-101_Form1A_SJ575956.html; Workgroup: WG19626; Design ID: 240]

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



AXYS METHOD MLA-010 Rev 08 CLIENT SAMPLE NO. Lab Blank Sample Collection: N/A PCB CONGENER ANALYSIS REPORT **AXYS ANALYTICAL SERVICES** P.O. Box 2219, 2045 MILLS RD. WEST, SIDNEY, B.C., CANADA V8L 3S8 TEL (250) 655-5800 FAX (250) 655-5811 NΑ Project No. Contract No.: 4033 Lab Sample I.D.: WG19626-101 Matrix: CORN OIL Sample Size: 5.00 g Sample Receipt Date: NΑ 08-May-2006 Initial Calibration Date: **Extraction Date:** 14-Jul-2006 HR GC/MS Instrument ID: Analysis Date: 01-Aug-2006 Time: 01:12:21 GC Column ID: DB1 Extract Volume (uL): 20 Sample Data Filename: DT63_189B S: 4 Injection Volume (uL): 2.0 Blank Data Filename: DT63_189B S: 4 **Dilution Factor:** N/A Cal. Ver. Data Filename: DT63_189B S: 1 Concentration Units: ng/kg

COMPOUND	IUPAC NO.	CO-ELUTIONS 5 X	LAB FLAG 1	CONC. FOUND	REPORTING LIMIT	ION ABUND. RATIO	RRT
3,3',4,4'-TeCB	77		¥				
3,4,4',5-TeCB	81	0575	ĸĴ	0.115	0.0634	23.96	1.000
2,3,3',4,4'-PeCB	105	V/-	X		0.000	20.00	1.000
2,3,4,4',5-PeCB	114		X				
2,3',4,4',5-PeCB	118		X				
2',3,4,4',5-PeCB	123		X				
3,3',4,4',5-PeCB	126		U	•	0.157		
2,3,3',4,4',5-HxCB	156		Х				
2,3,3',4,4',5'-HxCB	157		Х				
2,3',4,4',5,5'-HxCB	167		х				
3,3',4,4',5,5'-HxCB	169	0.393	ΚJ	0.079	0.0427	0.92	1.000
2,2',3,3',4,4',5-HpCB	170	- 12	Х				
2,2',3,4,4',5,5'-HpCB	180		X				
2,3,3',4,4',5,5'-HpCB	189	•	. X				

(1) Where applicable, custom lab flags have been used on this report; U = not detected; K = peak detected but did not meet quantification criteria, result reported represents the estimated maximum possible concentration; J = concentration less than LMCL; X = result reported separately.

Approved by:	Teresa	Rawsthorne	QA/QC Chemis

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

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AXYS METHOD MLA-010 R	ev 08			CLIENT SAMPLE NO. Lab Blank	
		Form 1A		Sample Collection:	
	HOMOLOGUE TO	TAL PCB	ANALYSIS REPORT	N/A	
P.O. Box 2219, 2045 MILLS RD.	WEST, SIDNEY, B.C., CANADA		Project No.	N/A	
V8L 3S8 TEL (250) 655-5800 FA Contract No.:	4033		Lab Sample I.D.:	WG19626-101	
Matrix:	CORN OIL		Sample Size:	5.00 g	
Sample Receipt Date:	N/A	<i>:</i>	Initial Calibration Date:	05-Jun-2006	war in the second
Extraction Date:	14-Jul-2006		Instrument ID:	HR GC/MS	
Analysis Date:	24-Jul-2006 Time: 11:52:21		GC Column ID:	DB1, SPB OCTYL	
Extract Volume (uL):	400		Sample Data Filename(s):	DT63_189B S: 4, PB6C_	327 S: 5
Injection Volume (uL):	1.0		Blank Data Filename:	PB6C_327 S: 5	
Dilution Factor:	20		Cal. Ver. Data Filename:	PB6C_327 S: 1	
Concentration Units:	ng/kg				
PCB HOMOLOGUE GROUP		LAB FLAG ¹	CONC. FOUND	<u>5</u> X	
Total Monochloro Biphenyls		U	•	•	
Total Dichloro Biphenyls		บ			
Total Trichloro Biphenyls		Ů			en en en en en en en en en en en en en e
Total Tetrachioro Biphenyis			5.05	25.25	
Total Pentachioro Biphenyls			2.28	-4	
Total Hexachioro Biphenyis			4.44 2:	2,2	
Total Heptachloro Biphenyls		ប			
Total Octachioro Biphenyls			0.757 3	785	· · · · · · · · · · · · · · · · · · ·
Total Nonachloro Biphenyls		U			
Decachioro Biphenyi		U			
TOTAL PCBs			12.5 <i>6.2</i>	.5	
(1) Where applicable, custom la (2) All header information pertaithe sample extract.	ib flags have been used on this ins to the initial instrumental an	report; U : alysis of th	= not detected. e sample extract. Additional sam	ole datafiles listed refer to s	econdary analysis of
· A	pproved by:Te	resa I	Rawsthorne	QA/QC Chemist	

For Axys Internal Use Only [XSL Template: xsi; Created: 28-Aug-2006 10:04:12; Application: XMLTransformer-1.7.9; Report Filename: 1668_PCB1668_HomTotals-TEQs_WG19626-101_Form1AHT_S1575956.html; Workgroup: WG19626; Design ID: 240]

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

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LDC #: 12449425 SDG #: DNPVG19975/WG19625

VALIDATION FINDINGS WORKSHEET Compound Quantitation and Reported CRQLs

Page: _	lof
Reviewer:	_ (r
2nd Reviewer:	1

METHOD: HRGC/HRMS PCB Congeners (EPA Method 1668)

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

N N/A Y N N/A

Were the correct internal standard (IS), quantitation ions and relative response factors (RRF) used to quantitate the compound? Compound quantitation and CRQLs were adjusted to reflect all sample dilutions and dry weight factors (if necessary).

#	Date	Sample ID	Finding	Associated Samples	Qualifications
		Ц	Finding PCB 1/8 > Calib Nange		J2 deta/P
			0	-	7 1
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			1300000		

<u> </u>		***************************************			
-					
			·		

Comments: _	See sample calculation verification worksheet for recalculations	

LDC #:<u>/64.1943</u> SDG #: <u>OPNS/1997</u>5

VALIDATION FINDINGS WORKSHEET Initial Calibration Calculation Verification

	Pa	age:_	<u>/</u> _of_/	_
	Revie		9-	_
2nd	Revie	wer:_	*	_

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

RRF = $(A_a)(C_b)/(A_b)(C_a)$ average RRF = sum of the RRFs/number of standards %RSD = 100 * (S/X)

A_x = Area of compound,

 A_{is} = Area of associated internal standard C_{is} = Concentration of internal standard

l

C_x = Concentration of compound, S = Standard deviation of the RRFs,

X = Mean of the RRFs

		Calibration		Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
#	Standard ID	Date	Compound (Reference Internal Standard)	Average RRF (initial)	Average RRF (Initial)	RRF (CS3 std)	RRF (८≤⊰ std)	%RSD	4,555
1	ICAL	5/9/a6 5/30/06	PCB-77 (°C-PCB-169) PCB-189 (°C-PCB-169) PCB-189 (°C-PCB-189)	1.04 0.94 0.96 0.93	1.04 0.94 0.96 0.93	1.11	1.10	3.72 5.08 4.53	3.8° 4,91 4.8
2	ICAL	5/8/06	PCB-77-28 (*C-PCB-777-81) PCB-156 (*C-PCB-166) [-26) PCB-156 (*C-PCB-166) [69) PCB-180 (*C-PCB-180)	0.88 0.91	0.88 0.91 0.86	0.94 1.00 0.92	0.98	3,8/ 4.0/ 7.64 5.00	3.96 7.42 4.84
3		446	PCB-77 (**C-PCB-77) PCB-105 (**C-PCB-105) PCB-156 (**C-PCB-156) PCB-180 (**C-PCB-180)						

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

LDC :	#:15	119	₩.	<u>>_</u>	
SDG	#:#	NG	-/	99	TS

VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

	Page:	
	Reviewer:	
2nd	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 $RRF = (A_x)(C_x)/(A_{sx})(C_x)$

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

 $A_x =$ Area of compound,

Ak = Area of associated internal standard

C_x = 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)	CC)	marfaut (CC)	%D	%D
1	PB6C-37	76.14	PCB-77 (¹³ C-PCB-77)	1.04	473	47.2		/
	5:1	7/24/06	PCB-105 (¹³ C-PCB-105)	0.94	51.5	5.5		
			PCB-156 (¹⁸ C-PCB-156)	0.96	104	104		
	6/5		PCB-180 (13C-PCB-180)	0.93	47.7	AT.9		
2	1763_18913	a/	PCB-72 (1°C-PCB-77)-81)	0.88	19.6	49.8	:	
	5-	8/	PCB-106 (1°C-PCB-105) (=6)	091	42.1	42,6		
			PCB-166 (13C-PCB-156) (69)	0.86	49.3	196		
	5/8		PCB-180 (13C-PCB-180)					·
3	PB6c_330		PCB-77 (¹³ C-PCB-77)	1.04	47.4	47.3		
	521	7/26/06	PCB-105 (¹⁹ C-PCB-105)	0.94	47.4 52.8	53.0		
		,	PCB-156 (¹⁸ C-PCB-156)	0.96	107	107		
	3/30		PCB-180 (13C-PCB-180)	093	49.3	49.6		
							7	

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.

VALIDATION FINDINGS WORKSHEET Routine Calibration Results Verification

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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 $RRF = (A_s)(C_h)/(A_h)(C_s)$

Where: ave. RRF = initial calibration average RRF

RRF = continuing calibration RRF

A_x = Area of compound,

A_k = Area of associated internal standard

 $C_x =$ 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)	RRFALC(-)	nnr And (cc)	%D	%D
1	PB&C3=84		PCB-77 (3C-PCB-77)	1.04	A O			7
	5=2	7/24/06	PCB-105 (19C-PCB-105)	0.94	58/9			
			PCB-156 (1°C-PCB-156)	2.96	107			
	6/5		PCB-180 (13C-PCB-180)	0.93	405	er grand garage		
Щ	'							
2	136C_33/C S:		PCB-77 (¹³ C-PCB-77)	1.04	46.9	46.9	·	
	5.	7/27/06	PCB-105 (1°C-PCB-105)	0.94	52.6	52.7		/
		=	PCB-156 (1°C-PCB-156)	0.96	106	106		/
			PCB-18q (1°C-PCB-18q)	0.93	48.2	48.4		/
					- '			
3	PB6C-3-8A	,	PCB-77 (1°C-PCB-77)	1.04	466	46.7		
	5:2	7/24/06	PCB-105 (^{f3} C-PCB-105)	1.94	53.9	54.0		
	-	/ /	PCB-156 (13C-PCB-156)	0.96	106	106		
			PCB-180 (1°C-PCB-180)	0.93	47.8	180		
				<i>F</i>			_/	

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 #: 15449A3 SDG #: DDW # 19975

VALIDATION FINDINGS WORKSHEET Laboratory Control Sample Results Verification

	Page:_	
	Reviewer:	9
nd	Reviewer:	A

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 = I LCS - LCSD I * 2/(LCS + LCSD)

LCS = Laboratory control sample percent recovery

LCSD = Laboratory control sample duplicate percent recovery

LCS ID: W&19626-103

	Spike Added (ഗ≤/wL)		Spiked S	Spiked Sample		LCS		LCSD		LCS/LCSD	
Compound			Concentration		Percent Recovery		Percent Recovery		RPD		
	LCS	rčep	LCS	LOSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalculated	
PCB-77	100	RA	97.0	/	aT.0	900					
PCB-81			101		(0)	(0)					
PCB-105			99.9		999	99.9					
PCB-114			104		104	104					
PCB-118			104		104	102					
PCB-123			104		104	104					
PCB-126	4		108		108	108					
PCB-156	200		218		109	109					
PCB-157_ -		. "									
PCB-167	100		105		105	105					
PCB-169	₩ /		101		(0)	10					
PGB-170_		·			/						
P CB-18 0									-		
PCB-189	100		97.5		a7.5	QT5					

Comments: Refer to Laboratory Control Sample findings worksheet for list of qualifications and associated samples when reported results do not agree within 10,0% of the recalculated results.

Ions Monitored for HRGC/HRMS Analysis of Polychlorinated Biphenyls

Descriptor	Accurate mass ^(a)	lon ID	Analyte	Substance
1	289.9224 291.9194 301.9626 303.9597 325.8804 927.8775 [292.9825]	M M+2 M M+2 M+2 M+4 Lock	C12 H6 35Cl4 C12 H6 35Cl3 37Cl4 13C12 H6 35Cl4 13C12 H6 35Cl3 37Cl C12 H5 35Cl4 37Cl C12 H5 35Cl3 37Cl2 C7 F11	TCB TCB PeCB PeCB PeCB PeCB PFK
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 35Cl5 37Cl 13C12 H4 35Cl5 37Cl 13C12 H4 35Cl6 37Cl C12 H3 35Cl6 37Cl C12 H3 35Cl6 37Cl C12 H3 35Cl5 37Cl2 13C12 H3 35Cl5 37Cl2 C15 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 ¹⁰C = 13.003355

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

F = 18.9984

LDC #: 15449A3
SDG #: DPWS 19975

VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page:	
Reviewer:_	9-
2nd reviewer:	*/

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

Y N N/A Y N N/A Were all reported results recalculated and verified for all level IV samples?

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

Concentration = (A.)(L)(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 l, Relative response factor of the calibration standard. RRF Volume or weight of sample pruged in milliliters (ml) ٧, or grams (g). Dilution factor. Df Percent solids, applicable to soils and solid %S

1	matrices only.			T	
# .	Sample ID	Compound	Reported Concentration ()	Calculated Concentration ()	Qualification
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