



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION III  
ENVIRONMENTAL SCIENCE CENTER  
701 MAPES ROAD  
FORT MEADE, MARYLAND 20755-5350

DATE : November 24, 2009

SUBJECT: Region III Data QA Review

FROM : Colleen Walling *Colleen K. Walling*  
Region III ESAT PO (3EA20)

TO : Michael Towle  
Regional Project Manager (3HS31)

Attached is the PCB Congener data validation report for the Lin Electric Company site (Das #: R33313; SDG#: R33313-01) completed by the Region III Environmental Services Assistance Team (ESAT) contractor under the direction of Region III EAID.

If you have any questions regarding this review, please call me at (410) 305-2763.

Attachments

cc: Gene Nance (Tech Law )

TO: 0021

TDF: 11003

OFFICE OF ANALYTICAL SERVICES AND QUALITY ASSURANCE



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**DATE:** November 24, 2009

**SUBJECT:** Organic Data Validation (Level M3)  
Site: Lin Electric  
DAS: R33313 SDG: R33313-01

**FROM:** Kenneth W. Curry *KWC*  
Senior Data Reviewer

Mahboobeh Mecanic *MM*  
Senior Oversight Chemist

**TO:** Colleen Walling  
ESAT Region 3 Project Officer

### **OVERVIEW**

DAS R33313, Sample Delivery Group (SDG) R33313-01, from the Lin Electric site consisted of four (4) aqueous samples for the determination of all PCB Congeners (mono to deca). The sample set included one (1) field blank and one (1) field duplicate pair. The PCB congener analyses were performed by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS) in accordance with USEPA Method 1668A. All samples were analyzed by AXYS Analytical Services (AXYS). Samples were analyzed through the Delivery of Analytical Services (DAS) program.

The twelve (12) 2005 World Health Organization (WHO) list congeners have toxicity equivalents (TEQ) similar to dioxin. The TEQ for these PCBs are summarized on the Data Summary Forms (DSFs) for these samples. No problems were detected during validation of these data.

### **PCB Congeners Analytical Methodology Comments**

- Two (2) ions were monitored for identification of each PCB congener.
- The laboratory utilized a total of twenty-seven (27) labeled extraction standards for quantitation of native PCB congeners. Three (3) cleanup standards were used to monitor method cleanup procedure efficiencies.

**QA/QC Comments**

- Sample data were reported by the laboratory using reporting limits (RLs) as specified on the Region 3 Analytical Request Form. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (LMCL) or sample specific detection limit (SDL), whichever was greater. The LMCL has been prorated for the extract volume and sample size. Any concentrations below the RLs were flagged "U" (non-detect) and not included in the homologue total and TEF adjusted concentrations by the laboratory. For the purpose of assessing blank contamination, results greater than one-fifth ( $>1/5$ ) of RL were reported on the DSF for the field blank and those results that were  $>1/5$  the RL in the method blank were evaluated and reported below if they would qualify data.
- Maximum concentrations of PCB congeners found in analyses of samples' associated method and field blanks are listed below. Only compounds that were greater than one-fifth ( $>1/5$ ) of the RL were used to qualify data. Samples with concentrations of PCB congeners less than five times ( $<5X$ ) the blank concentration have been qualified "B". Units are in pg/L. TEQs for results qualified "B" are not calculated.

<u>Blank</u>	<u>PCB Congener</u>	<u>Concentration</u>	<u>Associated Samples Affected</u>
Method CBLK01	DiCB (#11)	5.0 J	R33313-04
Field	DiCB (#8)	13 J	R33313-01
R33313-04	TriCB (#16)	7 J	R33313-02, R33313-03
	TriCB (# 18 + #30)	10 J	R33313-02, R33313-03

- The Laboratory Control Sample (LCS) analysis reported recoveries within control limits.
- Congeners detected below RLs were qualified "J" on the DSFs unless superseded by "B".
- Reported results for field duplicate pair, samples R33313-02/R33313-03, were comparable except for TetraCB(#56), HexaCB(#147 + #149), HexaCB(#153 + #168) and HeptaCB(#187).
- Sample volumes other than one (1) liter were used in the analyses of these samples. The dilution factors on the DSFs reflect this variance in sample volumes.

All data for DAS R33313, SDG R33313-01, were reviewed in accordance with Region III Modifications to the National Functional Guidelines for Organic Data Review, September 1994.

**ATTACHMENTS**

- 1) Appendix A - Glossary of Data Qualifiers
- 2) Appendix B - Data Summary Forms
- 3) Appendix C - Chain of Custody (COC) Records
- 4) Appendix D - Laboratory Case Narrative

DCN: R33313PCBM3

## **APPENDIX A**

### **GLOSSARY OF DATA QUALIFIER CODES (PCB CONGENER)**

## **GLOSSARY OF DATA QUALIFIER CODES (PCB CONGENER)**

- B** Blank Contamination
- J** The analyte was positively identified; the associated numerical value is the estimated concentration of the analyte in the sample.
- N** The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
- R** The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
- U** Not detected above the Contract Required Quantitation Limit (CRQL).
- UJ** Not detected, quantitation limit is estimated.

**APPENDIX B**

**DATA SUMMARY FORMS**

Case #: R33313

SDG : R3313-01

Number of Soil Samples : 0

Site :

LIN ELECTRIC

Number of Water Samples : 4

Lab. :

AXYS

Sample Number :		R33313-01		R33313-02		R33313-03		R33313-04			
Sampling Location : Prefix of LE091509-		SW-01		SW-02		SW-03		FB-01			
Field QC:				Field Dup. of R33313-03		Field Dup. of R33313-02		Field Blank			
Matrix :		Aqueous		Aqueous		Aqueous		Aqueous			
Units :		pg/L		pg/L		pg/L		pg/L			
Date Sampled :		9/15/2009		9/15/2009		9/15/2009		9/15/2009			
Time Sampled :		09:30		10:20		10:25		10:30			
Dilution Factor :		1.08		1.06		1.02		1.10			
PCB Chlorination Level	RL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
MoCB (#1)	20							14	J		
MoCB (#2)	20							13	J		
MoCB (#3)	20							11	J		
DiCB (#4)	20	82						15	J		
DiCB (#5)	20										
DiCB (#6)	20										
DiCB (#7)	20										
DiCB (#8)	20	22	B					13	J		
DiCB (#9)	20										
DiCB (#10)	20										
DiCB (#11)	20							19	B		
DiCB (#12 + #13)	40										
DiCB (#14)	20										
DiCB (#15)	20	26		24		22					
TriCB (#16)	20	45		25	B	25	B	7	J		
TriCB (#17)	20	37						4	J		
TriCB (#18 + #30)	40	87		48	B	43	B	10	J		
TriCB (#19)	20	59									
TriCB (#20 + #28)	40	96		65		60					
TriCB (#21 + #33)	40										
TriCB (#22)	20	26									
TriCB (#23)	20										
TriCB (#24)	20										
TriCB (#25)	20										
TriCB (#26 + #29)	40										
TriCB (#27)	20										
TriCB (#31)	20	53		28		26		5	J		
TriCB (#32)	20	63		27		25					
TriCB (#34)	20										
TriCB (#35)	20										
TriCB (#36)	20										

RL = Reporting Limit.

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample reporting limits: (RL \* Dilution Factor)

#12 &amp; #13 coelute, #18 &amp; #30 coelute, #26 &amp; #29 coelute, #20 &amp; #28 coelute, # 21&amp; #33 coelute, #26&amp;#29 coelute



Case #: R33313

SDG : R3313-01

Site :

LIN ELECTRIC

Lab. :

AXYS

Sample Number :		R33313-01		R33313-02		R33313-03		R33313-04			
Sampling Location : Prefix of LE091509-		SW-01		SW-02		SW-03		FB-01			
Field QC:				Field Dup. of R33313-03		Field Dup. of R33313-02		Field Blank			
Matrix :		Aqueous		Aqueous		Aqueous		Aqueous			
Units :		pg/L		pg/L		pg/L		pg/L			
Date Sampled :		9/15/2009		9/15/2009		9/15/2009		9/15/2009			
Time Sampled :		09:30		10:20		10:25		10:30			
Dilution Factor :		1.08		1.06		1.02		1.10			
PCB Chlorination Level	RL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
TriCB (#37)	20	20		22		24					
TriCB (#38)	20										
TriCB (#39)	20										
TetraCB (#40 + #41 + #71)	60	82									
TetraCB (#42)	20	39									
TetraCB (#43)	20										
TetraCB (#44 + #47 + #65)	60	180		66		66					
TetraCB (#45 + #51)	40	56									
TetraCB (#46)	20										
TetraCB (#48)	20										
TetraCB (#49 + #69)	40	98									
TetraCB (#50 + #53)	40	54									
TetraCB (#52)	20	240		81		77		4	J		
TetraCB (#54)	20										
TetraCB (#55)	20										
TetraCB (#56)	20	59				24					
TetraCB (#57)	20										
TetraCB (#58)	20										
TetraCB (#59 + #62 + #75)	60										
TetraCB (#60)	20	24									
TetraCB (#61 + #70 + #74 + #76)	80	310									
TetraCB (#63)	20										
TetraCB (#64)	20	61		24		25					
TetraCB (#66)	20	120		31		38					
TetraCB (#67)	20										
TetraCB (#68)	20										
TetraCB (#72)	20										
TetraCB (#73)	20										
TetraCB (#77)	20										
TetraCB (#78)	20										

RL = Reporting Limit.

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample reporting limits: (RL \* Dilution Factor)

#40, #41 &amp; #71 coelute, #44, #47, #65 coelute, #45 &amp; #51 coelute, #50 &amp; #53 coelute, #49 &amp; #69 coelute, #59, #62, #75 &amp; #61, #70, #74, #76 coelute

Case #: R33313

SDG : R33313-01

Site :

LIN ELECTRIC

Lab. :

AXYS

Sample Number :		R33313-01		R33313-02		R33313-03		R33313-04			
Sampling Location : Prefix of LE091509-		SW-01		SW-02		SW-03		FB-01			
Field QC:				Field Dup. of R33313-03		Field Dup. of R33313-02		Field Blank			
Matrix :		Aqueous		Aqueous		Aqueous		Aqueous			
Units :		pg/L		pg/L		pg/L		pg/L			
Date Sampled :		9/15/2009		9/15/2009		9/15/2009		9/15/2009			
Time Sampled :		09:30		10:20		10:25		10:30			
Dilution Factor :		1.08		1.06		1.02		1.10			
PCB Chlorination Level	RL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
TetraCB (#79)	20										
TetraCB (#80)	20										
TetraCB (#81)	20										
PentaCB (#82)	20	190									
PentaCB (#83 + #99)	40	820									
PentaCB (#84)	20	290		21		25					
PentaCB (#85 + #116 + #117)	60	240									
PentaCB (#86 + #87 + #97 + #108 + #119 + #125)	120	1000									
PentaCB (#88 + #91)	40	130									
PentaCB (#89)	20										
PentaCB (#90 + #101 + #113)	60	1300									
PentaCB (#92)	20	210									
PentaCB (#93 + #95 + #98 + #100 + #102)	100	790									
PentaCB (#94)	20										
PentaCB (#96)	20										
PentaCB (#103)	20										
PentaCB (#104)	20										
PentaCB (#105)	20	1000									
PentaCB (#106)	20										
PentaCB (#107 + #124)	40	84									
PentaCB (#109)	20	110									
PentaCB (#110 + #115)	40	1900		50		58					
PentaCB (#111)	20										
PentaCB (#112)	20										
PentaCB (#114)	20	53									
PentaCB (#118)	20	2200		22		29					
PentaCB (#120)	20										
PentaCB (#121)	20										

RL = Reporting Limit.

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample reporting limits: (RL \* Dilution Factor)

#83& #99 coelute, #85,#116,#117 coelute, #86,#87,#97,#108,#119, #125 coelut, #88 & #91 coelute, #90,#101,#113 coelute, #93, #95, #98, #100 & #102 coel  
 #107,#124 coelute, #110,#115 coelute

Case #: R33313

SDG : R3313-01

Site :

LIN ELECTRIC

Lab. :

AXYS

Sample Number :		R33313-01		R33313-02		R33313-03		R33313-04			
Sampling Location : Prefix of LE091509-		SW-01		SW-02		SW-03		FB-01			
Field QC:				Field Dup. of R33313-03		Field Dup. of R33313-02		Field Blank			
Matrix :		Aqueous		Aqueous		Aqueous		Aqueous			
Units :		pg/L		pg/L		pg/L		pg/L			
Date Sampled :		9/15/2009		9/15/2009		9/15/2009		9/15/2009			
Time Sampled :		09:30		10:20		10:25		10:30			
Dilution Factor :		1.08		1.06		1.02		1.10			
PCB Chlorination Level	RL	Result	Flag	Result	Flag	Result	Flag		Flag	Result	Flag
PentaCB (#122)	20	28									
PentaCB (#123)	20	35									
PentaCB (#126)	20										
PentaCB (#127)	20										
HexaCB (#128 + #166)	40	880									
HexaCB (#129 + #138 + #160 + #163)	60	3800									
HexaCB (#130)	20	240									
HexaCB (#131)	20	45									
HexaCB (#132)	20	1100									
HexaCB (#133)	20	33									
HexaCB (#134 + #143)	40	150									
HexaCB (#135 + #151 + #154)	60	520									
HexaCB (#136)	20	210									
HexaCB (#137)	20	250									
HexaCB (#139 + #140)	40	57									
HexaCB (#141)	20	540									
HexaCB (#142)	20										
HexaCB (#144)	20	90									
HexaCB (#145)	20										
HexaCB (#146)	20	360									
HexaCB (#147 + #149)	40	1700				49					
HexaCB (#148)	20										
HexaCB (#150)	20										
HexaCB (#152)	20										
HexaCB (#153 + #168)	40	2100				45					
HexaCB (#155)	20										
HexaCB (#156 + #157)	40	750									
HexaCB (#158)	20	420									
HexaCB (#159)	20										

RL = Reporting Limit.

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample reporting limits: (RL \* Dilution Factor)

#128, #166 coelute, #129, #138, #160, #163 coelute, #134, #143, #154 coelute, #135 & #151 coelute, #139 & #140 coelute, #147 & #149 coelute, #153 & #168 coelute, #156, #157 coelute

Case #: R33313

SDG : R3313-01

Site :

LIN ELECTRIC

Lab. :

AXYS

Sample Number :		R33313-01		R33313-02		R33313-03		R33313-04			
Sampling Location : Prefix of LE091509-		SW-01		SW-02		SW-03		FB-01			
Field QC:				Field Dup. of R33313-03		Field Dup. of R33313-02		Field Blank			
Matrix :		Aqueous		Aqueous		Aqueous		Aqueous			
Units :		pg/L		pg/L		pg/L		pg/L			
Date Sampled :		9/15/2009		9/15/2009		9/15/2009		9/15/2009			
Time Sampled :		09:30		10:20		10:25		10:30			
Dilution Factor :		1.08		1.06		1.02		1.10			
PCB Chlorination Level	RL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
HexaCB (#161)	20										
HexaCB (#162)	20										
HexaCB (#164)	20	230									
HexaCB (#165)	20										
HexaCB (#167)	20	210									
HexaCB (#169)	20										
HeptaCB (#170)	20	520									
HeptaCB (#171 + #173)	40	150									
HeptaCB (#172)	20	69									
HeptaCB (#174)	20	320									
HeptaCB (#175)	20										
HeptaCB (#176)	20	37									
HeptaCB (#177)	20	180									
HeptaCB (#178)	20	47									
HeptaCB (#179)	20	82									
HeptaCB (#180 + #193)	40	760									
HeptaCB (#181)	20										
HeptaCB (#182)	20										
HeptaCB (#183 + #185)	40	220									
HeptaCB (#184)	20										
HeptaCB (#186)	20										
HeptaCB (#187)	20	280				21					
HeptaCB (#188)	20										
HeptaCB (#189)	20	29									
HeptaCB (#190)	20	90									
HeptaCB (#191)	20										
HeptaCB (#192)	20										
OctaCB (#194)	20	110									
OctaCB (#195)	20	41									
OctaCB (#196)	20	47									

RL = Reporting Limit.

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample reporting limits: (RL \* Dilution Factor)

#171 &amp; #173 coelute, #180 &amp; #193 coelute, #183 &amp; #185 coelute

Case #: R33313

SDG : R3313-01

Site :

LIN ELECTRIC

Lab. :

AXYS

Sample Number :		R33313-01		R33313-02		R33313-03		R33313-04			
Sampling Location : Prefix of LE091509-		SW-01		SW-02		SW-03		FB-01			
Field QC:				Field Dup. of R33313-03		Field Dup. of R33313-02		Field Blank			
Matrix :		Aqueous		Aqueous		Aqueous		Aqueous			
Units :		pg/L		pg/L		pg/L		pg/L			
Date Sampled :		9/15/2009		9/15/2009		9/15/2009		9/15/2009			
Time Sampled :		09:30		10:20		10:25		10:30			
Dilution Factor :		1.08		1.06		1.02		1.10			
PCB Chlorination Level	RL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
OctaCB (#197 + #200)	40										
OctaCB (#198 + #199)	40	97									
OctaCB (#201)	20										
OctaCB (#202)	20										
OctaCB (#203)	20	58									
OctaCB (#204)	20										
OctaCB (#205)	20										
NanoCB (#206)	20	33									
NanoCB (#207)	20										
NanoCB (#208)	20										
DecaCB (#209)	20										

RL = Reporting Limit.

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample reporting limits: (RL \* Dilution Factor)

, #197 &amp; #200 coelute, #198 &amp; # 199 coelute



Case #: R33313

SDG : R3313-01

Site :

LIN ELECTRIC

Lab. :

AXYS

Sample Number :	R33313-01	R33313-02	R33313-03	R33313-04							
Sampling Location : Prefix of LE091509-	SW-01	SW-02	SW-03	FB-01							
Field QC:		Field Dup. of R33313-03	Field Dup. of R33313-02	Field Blank							
Matrix :	Aqueous	Aqueous	Aqueous	Aqueous							
Units :	pg/L	pg/L	pg/L	pg/L							
Date Sampled :	9/15/2009	9/15/2009	9/15/2009	9/15/2009							
Time Sampled :	09:30	10:20	10:25	10:30							
Dilution Factor :	1.08	1.06	1.02	1.10							
PCB Chlorination Level (IUPAC#)	RL	Result	Flag	Result	Flag	Result	Flag	Result	Flag	Result	Flag
Total Mono-CB								38			
Total Di-CB		108		24		22		47			
Total Tri-CB		510		147		132		26			
Total Tetra-CB		1300		200		230		4			
Total Penta-CB		10000		93		110					
Total Hexa-CB		14000				94					
Total Hepta-CB		2800				21					
Total Octa-CB		350									
Total Nano-CB		33									
Total Deca-CB											
TOTAL		29000		460		610		115			

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

To calculate sample quantitation limits: (RL \* Dilution Factor)

## DATA SUMMARY FORM: TOX

Page 8 of 8

Case #: R33313

SDG : R33313-01

Number of Soil Samples : 0

Site :

LIN ELECTRIC

Number of Water Samples : 4

Lab. :

AXYS

Sample Number :	R33313-01	R33313-02	R33313-03	R33313-04												
Sample Location : Prefix of LE091509-	SW-01	SW-02	SW-03	FB-01												
Field QC:		Field Dup. of R33313-03	Field Dup. of R33313-02	Field Blank												
Matrix:	Aqueous	Aqueous	Aqueous	Aqueous												
Units:	pg/L	pg/L	pg/L	pg/L												
Date Sampled :	9/15/2009	9/15/2009	9/15/2009	9/15/2009												
Time Sampled :	09:30	10:20	10:25	10:30												
Dilution Factor :	1.08	1.06	1.02	1.10												
Analyte / TEF	RL	CONC	TEQ	Q	CONC	TEQ	Q	CONC	TEQ	Q	CONC	TEQ	Q	CONC	TEQ	Q
TetraCB #77 (0.0001)	20		0			0			0			0			0	
TetraCB #81 (0.0003)	20		0			0			0			0			0	
PeCB #105 (0.00003)	20	1000	0.03			0			0			0			0	
PeCB #114 (0.00003)	20	53	0.00159			0			0			0			0	
PeCB #118 (0.00003)	20	2200	0.066		22	0.00066		29	0.00087			0			0	
PeCB #123 (0.00003)	20	35	0.00105			0			0			0			0	
PeCB #126 (0.1)	20		0			0			0			0			0	
HxCB #156/157 (0.00003)	40	750	0.0225			0			0			0			0	
HxCB #167 (0.00003)	20	210	0.0063			0			0			0			0	
HxCB #169 (0.03)	20		0			0			0			0			0	
HpCB #189 (0.00003)	20	29	0.00087			0			0			0			0	
TOTAL TEQ			0.12831			0.00066			0.00087			0			0	

RL = Reporting Limit.

To calculate sample quantitation limits: (QL \* Dilution Factor)

**APPENDIX C**

**CHAIN OF CUSTODY (COC) RECORDS**



Reference Case:  
Client No: R33313

**R**

Region: 3		Date Shipped: 9/29/2009		Chain of Custody Record	
Project Code: 09TO3N302DC6CA3CNRV00		Carrier Name: FedEx		Sampler Signature:	
Account Code: WVN000306141		Airbill: 7969 8389 8902		Relinquished By (Date / Time)	
Spill ID: A3CN		Shipped to: AXYS Analytical Services Ltd.		Received By (Date / Time)	
Site Name/State: Lin Electric Company/WW		2045 Mills Rd. W.			
Project Leader: Gene Nance		Sidney BC V8L 5X2			
Action: Removal Action		(250) 655-5800			
Sampling Co: Techlaw, Inc.					

SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
R33313-01	Storm Water/ Gene Nance	L/G	PCB_C (30)	31025 (Ice Only), 31026 (Ice Only), 31027 (Ice Only), 31028 (Ice Only), 31029 (Ice Only), 31030 (Ice Only) (6)	LE091509-SW-01	S: 9/15/2009 9:30	Lab QC
R33313-04	Field QC/ Gene Nance	L/G	PCB_C (30)	31035 (Ice Only), 31036 (Ice Only) (2)	LE091509-FB-01	S: 9/15/2009 10:30	Field Blank

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC: R33313-01	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: PCB_C = PCBs (CONGENERs) _Aqueous	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

Reference Case:  
Client No: R33313

**R**

Region: Project Code: Account Code: CERCLIS ID: Spill ID: Site Name/State: Project Leader: Action: Sampling Co:	3 09TO3N302DC6CA3CNRV00 WVN000306141 A3CN Lin Electric Company/WV Gene Nance Removal Action Techlaw, Inc.	Date Shipped: Carrier Name: Airbill: Shipped to:	9/29/2009 FedEx 7979 7158 0905 AXYS Analytical Services Ltd. 2045 Mills Rd. W. Sidney BC V8L 5X2 (250) 655-5800
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SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	QC Type
R33313-02	Storm Water/ Gene Nance	L/G	PCB_C (30)	31031 (Ice Only), 31032 (Ice Only) (2)	LE091509-SW-02	S: 9/15/2009 10:20	Fld Dup of LE091509-SW-03
R33313-03	Storm Water/ Gene Nance	L/G	PCB_C (30)	31033 (Ice Only), 31034 (Ice Only) (2)	LE091509-SW-03	S: 9/15/2009 10:25	Fld Dup of LE091509-SW-02

Shipment for Case Complete? Y	Sample(s) to be used for laboratory QC:	Additional Sampler Signature(s):	Chain of Custody Seal Number:
Analysis Key: PCB_C = PCBs (CONGENERs) _Aqueous	Concentration: L = Low, M = Low/Medium, H = High	Type/Designate: Composite = C, Grab = G	Shipment Iced? _____

# U.S. EPA Region III Analytical Request Form

Revision 10.06

QTS 9-11-09

ASQAB USE ONLY	
RAS#	Analytical TAT
DAS#	R33313
NSF#	30

Date: 09/10/09		Site Activity: Removal	
Site Name: Lin Electric Company		Street Address: 1400 Bluefield Avenue	
City: Bluefield		State: WV	
Latitude: 37.262839		Longitude: -81.240273	
Program: Superfund		Acct. #: 2009 TO3N302DC6CA3CNRV00	
Site ID: A3CN		CERCLIS #: WVN000306141	
Spill ID:		Operable Unit:	
Site Specific QA Plan Submitted: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes		Title: Sampling QA/QC Work Plan	
Date Approved: June 2009			
EPA Project Leader: Mike Towle		Cell Phone #: 215-287-2443	
Request Preparer: Gene Nance		E-mail: towle.michael@epa.gov	
Site Leader: Gene Nance		Cell Phone #: 740-867-0968	
		E-mail: gnance@techlawinc.com	
Contractor: TechLaw, Inc		Cell Phone #: 740-867-0968	
		E-mail: gnance@techlawinc.com	
EPA CO/PO: Lorrie Murray/Denise Jones/ Karen Wodarczyk			
#Samples	Matrix:	Parameter:	Method:
#Samples 4	Matrix: Surface water	Parameter: PCB Congeners (209 List)	Method: EPA 1668A or equiv 31194
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
#Samples	Matrix:	Parameter:	Method:
Ship Date From: Sept 15, 2009	Ship Date To: Sept 18, 2009	Org. Validation Level M3 equiv	Inorg. Validation Level
Unvalidated Data Requested: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes	If Yes, TAT Needed: <input type="checkbox"/> 14days <input type="checkbox"/> 7days <input type="checkbox"/> 48hrs <input type="checkbox"/> 24hrs	<input checked="" type="checkbox"/> Other (Specify) 30 day by lab.	
Validated Data Package Due: <input type="checkbox"/> 42 days <input type="checkbox"/> 30 days <input type="checkbox"/> 21days <input type="checkbox"/> 14 days	<input checked="" type="checkbox"/> Other (Specify) 45 day TAT (flexible to accommodate ASQA schedule) 30/15		
Electronic Data Deliverables Required: <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes (EDDs will be provided in Region 3 EDD Format)			
Special Instructions:			
Extension of TAT for PCB congeners is acceptable to accommodate ASQA Lab schedule, if ASQA can accept the samples.			
Detection Levels/Target Compound List: PCB Congeners by 1668A - 20 pg/L as per Case R33090 which were analyzed by OASQA. Fact sheet for CBC01.1 (Attachment 1 in Lin Electric Sampling and Analysis Plan Addendum 3 for Groundwater/Surface Water) provided to list Target compounds only.			

## **APPENDIX D**

### **LABORATORY CASE NARRATIVE**

# **PCB CONGENER ANALYSIS**

## **AQUEOUS SAMPLES**

**AXYS METHOD: MLA-010**

**EPA METHOD: CBC01.0**

**Contract No.: EP05W001655**

**Contract Requisition/Reference No.: SD0069/QT-DC-  
09-003258 and PR-DC-09-02167/SD006**

**Task Order No.: EP09W002073**

**Project/Case No.: R33313**

**SDG No.: R33313-01**

**Client No: 4125**

**Data Package Identification: DPWG30669**

**Analysis WG30393**

**Prepared for:  
USEPA Region 3**

**Prepared by:  
AXYS Analytical Services Ltd.  
2045 Mills Rd West  
Sidney, British Columbia V8L 5X2  
CANADA**

**Contact: Angie Whetung  
Project Manager**

**29 October 2009**



**USEPA REGION 3**

**LAB NAME: AXYS ANALYTICAL SERVICES LTD.**

**PCB CONGENER ANALYSIS**

**EPA Method: 1668A and CBC01.0**

**AXYS Method: MLA-010**

**4125: L13654-1 to -4**

**Contract Number: EP05W001655**

**Task Order Number: EP09W002073**

**Project/Case No: R33313**

**Requisition/Reference Number: SD0069/QT-DC-09-003258 & PR-DC-09-02167/SD006**

**SDG Number: R33313-01**

**Regional Tracking Number: N/A**

**Sample Number:**

**R33313-01, R33313-02, R33313-03, R33313-04**

**Matrix: WATER**

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**29 October 2009**

**SDG NARRATIVE**

This narrative describes the analysis of four aqueous samples for the determination of PCB congeners using high-resolution gas chromatography / high-resolution mass spectrometry (HRGC / HRMS).

**1. SAMPLE RECEIPT AND STORAGE**

Samples were received on September 30<sup>th</sup>, 2009. The temperature of the samples upon receipt was between 4 °C and 6 °C, exceeding the requirement of 4 °C. This is judged not to significantly impact the data accuracy and the analysis was allowed to proceed. Details of sample conditions on receipt are provided on the Sample Receiving Record forms included in this data package. Samples were stored at 4°C, in the dark, prior to extraction and analysis.

**2. SAMPLE PREPARATION**

The aqueous samples were pre-treated as described on the Water Pretreatment Record forms included in this data package.

Approximately 1L of sample was spiked with labeled quantification standards, allowed to equilibrate, and then liquid-liquid extracted using dichloromethane. The raw extract was spiked with labeled cleanup standards and cleaned up using standard chromatographic columns as documented on the laboratory worksheets included in this data package. Following cleanup, the final extract was reduced in volume and spiked with labeled recovery (internal) standards prior to instrumental analysis.

**3. ANALYSIS**

- i. Samples and QC samples were analyzed in one batch named WG30393. The composition of the batch is shown on the Cover Page and Correlation Table, and on the Batch list forms included among the extraction workup sheets.
- ii. Analysis procedures were in general accordance with **USEPA Method CBC01.0** as documented in **AXYS Method MLA-010: Analytical Method for the Determination of 209 PCB Congeners by EPA Method 1668A or EPA Method CBC01.0**. Method summary (MSU-020) of **AXYS Method MAL-010** is included following this narrative.
- iii. Instrumental analysis was conducted by high-resolution gas chromatography/high resolution



mass spectrometry (HRGC/HRMS) on an AUTOSPEC ULTIMA high resolution MS equipped with an HP 6890 gas chromatograph, a CTC auto-sampler, and an Alpha data system running Micromass software. An SPB-Octyl (30 m, 0.25 mm i.d., 0.25  $\mu$ m film thickness) chromatography column was coupled directly to the MS source. The MS was operated at 10,000 (static) mass resolution in the electron impact ionization mode using multiple ion detection, acquiring the ions listed in Table 8 of 'USEPA Method 1668, Revision A'.

- iv. On the chromatograms and quantification reports, the fourth character in the datafile name identifies the instrument (e.g. PB9C\_306 S: A - the fourth character is "C"). The following is a correlation between the single character and the formal name:
  - A, B, C, 1, 2, 3 - AUTOSPEC ULTIMAs.
- v. In this data package each chromatogram is attached to its quantification report pages as produced by the instrument's Micromass OPUSQuan software. The qualitative identification procedures followed the criteria as set out in Exhibit D of Method CBC01.0, Sections 11.1.1 through 11.1.6.1; the specifications refer to absolute and relative retention time, signal to noise ratio and relative abundance ratio. Chromatograms were visually examined and evaluated against the areas and ratios of PCB peaks as determined by the OPUSQuan software. If the ratio determined by the software was just marginally out of specification, the chromatogram was visually inspected to ensure that peaks were correctly integrated and peak areas were adjusted where correction was necessary.
- vi. Homologue totals were obtained by summing the concentration of all detected congeners at each level of chlorination. Congener peaks that did not meet the method ion abundance ratio criteria were excluded from in the homologue totals.
- vii. Target concentrations were determined by Isotope dilution using Micromass OPUSQUAN software. Sample data are reported using reporting limits (RLs) as specified by the client. The reporting limit (RL) was defined as the concentration equivalent to the lowest calibration standard (LMCL) or the sample specific detection limit (SDLs), whichever was greater. The LMCL has been prorated for the extract volume and sample size. Any concentrations below the reporting limits have been flagged 'U' (non-detect) and not included in the homologue total and TEF adjusted concentrations.
- viii. Sample specific detection limits (SDLs) were determined from the analysis data by converting the average noise signal to a concentration following the same procedures used to convert target peak responses to concentrations.

#### 4. REPORTING CONVENTIONS

- i. The AXYS contract number assigned for internal tracking was 4125. Samples were assigned a unique laboratory identifier of the form L13654-X, where X = numeral; all data reports reference both the AXYS ID and the client sample identifier. A table co-correlating AXYS IDs with client's sample numbers is included in this data package.
- ii. Results are reported in concentration units of picograms per litre (pg/L).
- iii. The following laboratory, contract specific, qualifiers were used for the database:

C	= co-elution of congeners
Cx	= co-elutes with the indicated congener 'x'. Data are reported against the indicated congener (which is always the lowest IUPAC-designated congener of the co-elution).
U	= identifies a target analyte that was not detected



iv. The following laboratory, contract specific, qualifiers were used for the reports:

U = identifies a target analyte that was not detected

## 5. QA/QC NOTES

QC samples were prepared alongside the client samples, and were carried through the same analytical procedures. The sample data were evaluated in relation to the batch QC samples. Note that sample analyte concentrations are not blank corrected.

### Lab Blank (AXYS ID WG30393-101)

No analytes were detected in the lab blank above the reporting limits specified.

### Laboratory Control Sample (AXYS ID WG30393-102)

Recoveries for all target analytes were within the range specified in Table 6 Exhibit D of Analytical Methods for Chlorinated Biphenyl Congeners.

## 6. ANALYTICAL DISCUSSION

### General

A summary of the method modifications to the EPA Method CBC01.0 follows this narrative.

Signal-to-noise values for the initial calibration data are provided on a separate printout. This is included with the chromatograms and is attached to the signed quantification report pages.

No analytical difficulties were encountered.

## 7. SAMPLE CALCULATION

### I. Example RRF calculation

Relative Response Factors (RRFs) were calculated by applying the area response of the analytes using the following formula.

$$RRF = \left( \frac{\text{area of Target}}{\text{area of labeled compound}} \right) \times \left( \frac{\text{concentration of labeled compound}}{\text{concentration of Target}} \right)$$

An example is given for PCB 4. Initial calibration data file PB9C\_274A S: 6 = CS3

$$\begin{aligned} RRF \text{ for PCB 4} &= \frac{(\text{area of PCB 4}) (\text{concentration of labeled PCB 4})}{(\text{area of labeled PCB 4}) (\text{concentration of PCB 4})} \\ &= \frac{(4.07 \times 10^7) (100 \text{ ng/mL})}{(8.79 \times 10^7) (50 \text{ ng/mL})} \\ &= \underline{0.927} \end{aligned}$$

Mean relative response factors (RRF) determined from the initial calibration runs were used to convert raw peak areas in sample chromatograms to final concentrations using the formulae applied below. The mean RRF for PCB 4 from the initial calibration equals 0.926.





## II. Sample target concentration calculation.

Sample calculation is provided for PCB 4 in sample R33313-01 (AXYS ID: L13654-1) (datafile: PB9C\_306A S: 5).

$$\text{Concentration of Target} = \left( \frac{\text{area of Target}}{\text{area of labeled compound}} \right) \times \left( \frac{\text{weight of labeled compound}}{\text{RRF}} \right) \times \left( \frac{1}{\text{sample size}} \right)$$

$$\text{Concentration of PCB 1} = \frac{(\text{area of PCB 1}) (\text{weight of labeled PCB 1}) (1)}{(\text{area of labeled PCB 1}) (\text{RRF}) (\text{weight of sample})}$$

$$\begin{aligned} \text{Concentration of PCB 1} &= \frac{(1.48 \times 10^6) (2000\text{pg}) (1)}{(4.18 \times 10^7) (0.926) (0.9295 \text{ L})} \\ &= \underline{\underline{82.3 \text{ pg/L}}} \end{aligned}$$

## 8. DATA PACKAGE

This data package is assigned a unique identifier DPWG30669 shown on the front page of this Data Package. Included in the data package following the narrative is the following documentation:

- method summary
- sample cover page and correlation table
- sample data reports followed by sample raw data, organized by AXYS ID
- laboratory QC summaries
- instrumental QC data reports and raw data
- laboratory QC data reports, followed by raw data
- laboratory extraction logs for each sample
- sample receiving documentation

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.



Signed: Shelley Facchin, QC Chemist



Date Signed



**AXYS ANALYTICAL SERVICES LTD**

**Analysis of PCB Congeners  
By USEPA Method 1668A**

Samples are spiked with isotopically labelled surrogate standards, solvent extracted and cleaned up on a series of chromatographic columns which may include silica, Florisil, alumina, carbon/Celite and gel permeation columns. The final extract is spiked with isotopically labelled recovery (internal) standards prior to instrumental analysis. Analysis of the extract is performed on high-resolution mass spectrometer (HRMS) coupled to a high-resolution gas chromatograph (HRGC) equipped with a SPB-Octyl chromatography column (30 m, 0.25 mm i.d., 0.25 µm film thickness). Resolution of the PCB 156/157 coelution may be achieved by high resolution GC/MS using a DB-1 chromatography column (30 m, 0.25 mm id, 0.25 µm film thickness). The method is carried out in accordance with the protocols described in EPA Method 1668A *with changes and correction through to August 20, 2003*, incorporating the AXYS modifications described below. Details of all procedures are documented in AXYS method MLA-010, *Analytical Method for Determination of 209 PCB Congeners by EPA Method 1668A*.

**Method Modifications:**

*Section 4.2.1, 4.2.2:* The protocol for washing reusable glassware includes a detergent wash, water rinse and baking at a minimum of 300°C for 8 hours. Immediately prior to use, glassware is solvent rinsed with toluene and hexane.

*Section 4.7:* The first cleanup column for tissue extracts is a gravity gel permeation column (SX-3 Biobeads). An anthropogenic isolation column 7.5.3 is not used.

*Section 6.5.1:* Glass wool is cleaned by rinsing twice with toluene and twice with hexane.

*Section 7.12, 7.13, 9.0, 11.0:* The concentration of the labeled toxics/LOC and the cleanup standard spiking solutions is 100 ng/mL and the sample spiking volume is 20 µL. The resulting final concentrations in the extracts are as specified in the method.

*Section 7.14:* Concentration of the labeled injection internal standard spiking solution (recovery standard) is modified so that a volume of 5 µL is added. The resulting amount of standard added to the final extract is the same as specified in the method. The solution is spiked into a 15 µL extract volume for a final extract volume of 20 µL.

*Section 7.2.1:* Powdered, not granular, sodium sulphate is baked at a minimum of 300°C for 8 hrs rather than at 600°C for 24 hrs.

*Section 7.5.1:* Silica is activated by baking at 450°C in a muffle oven for at least 8 hrs.

*Section 7.5.4.1.1:* Florisil is baked at 450°C in a muffle oven for at least 8 hrs, and then deactivated with water to 2.1% deactivation.

*Section 10.3.3, 15.3.3:* A S:N ratio of 3:1 for di-PCBS and nona-PCBs in CS0.2 calibration solution is acceptable.

*Section 11.5.6:* Unless requested by the client, the aqueous portion after filtration of aqueous samples with >1% solids is not discarded but is extracted.

*Section 11.5, 11.5.2, 11.5.5 12.3* Solid samples are dried by mixing with anhydrous sodium sulphate. The dried solid is extracted using a soxhlet extraction apparatus. The surrogate



## AXYS ANALYTICAL SERVICES LTD

spike is incorporated after the drying step. Equilibration time is 30 minutes. The extracting solvent for solids is dichloromethane.

*Section 11.8, 12.4:* The surrogate spike is incorporated into the sample after the drying step to eliminate the possibility of disproportional loss of volatile labeled and target compounds.

*Section 12.4.2:* The pre-cleaning of the soxhlet apparatus is carried out using toluene instead of dichloromethane.

*Section 12.4.9:* Lipid analysis is carried out by sub-sampling two 2g portions of the extract from a total 30 g extract weight. The cleanup standard is spiked into the extract after soxhlet extraction and before any lipid analysis or rotary evaporation is done. The percent recoveries are corrected for the amount of extract used for lipid analysis.

*Section 12.6.1.1:* Rotary evaporation is done at 30°C. Daily cleaning of the rotary evaporators include dismantling and rinsing/soaking in solvent. Mimic proofs are run periodically but are not archived daily.

*Section 12.7.4:* Before Florisil or alumina cleanup procedures, a solvent exchange is done by reducing under nitrogen to 300 uL and bulking up to 1mL in hexane. If toluene is present the extract is reduced to 50 uL under nitrogen and bulked up to 1mL.

*Section 12.7.7:* Toluene (1 mL) is added to the eluate from the final column prior to rotary evaporation and nitrogen blow down concentration steps.

*Section 13.1.1:* GPC chromatography, by a gravity column, is routinely used only for tissue extracts. The GPC cleanup is optional for all other matrices.

*Section 13.3.1:* Routine layered silica column is as follows: 0.5 g neutral silica, 2 g 28% basic silica, 0.5 g neutral silica, 4 g 44% acidic silica, 4 g 22% acidic silica, 1 g neutral silica.

*Section 13.3.4:* The sample is loaded onto the column followed by 2-3 rinses of a least 1 mL, and eluted with 100 mL of hexane.

*Section 14.2:* The volume of labeled injection internal standard (recovery standard) added to the extract is 5 µL, for a final extract volume of 20 µL. Hexane rather than nonane is used as the solvent to bring extract back to volume for re-analysis or to dilute extracts.

*Section 15.3:* The calibration solution containing all 209 PCB congeners is used as the CAL/VER solution.

*Section 17.5:* Extracts are diluted with hexane. The concentration of the labeled injection internal (recovery) standard is not re-adjusted to 100 pg/uL when dilutions are performed.

### Section 17.0

*Conc<sub>i</sub>* - the concentrations of target analytes, and the labelled compound concentrations and recoveries, are calculated using the equations below. These procedures are equivalent to those described in the method but are more direct.



## AXYS ANALYTICAL SERVICES LTD

$$Conc_i = \frac{A_i}{A_{si}} \times \frac{M_{si}}{RRF_{i,si}} \times \frac{1}{M_x}$$

- where  $A_i$  = summed areas of the primary and secondary m/z's for the analyte peak of interest (compound  $i$ )
- $A_{si}$  = summed areas of the primary and secondary m/z's for the labelled surrogate peak used to quantify  $i$ )
- $M_x$  = mass of sample taken for analysis
- $M_{si}$  = mass of labelled surrogate (compound  $si$ ) added to sample as calculated by the concentration of standard spiked (pg/mL) multiplied by the volume spiked (mL)
- $RRF_{i,si}$  = mean relative response factor of  $i$  to  $si$  from the five-point calibration range and defined individually as:

$$\frac{A_i}{A_{si}} \times \frac{M_{si}}{M_i}$$

Calculation of Surrogate Standard Concentrations and Percent Recoveries:  
Concentrations of surrogate standards are calculated using the following equation:

$$Conc_{si} = \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{RRF_{si,rs}}$$

and, the percent recoveries of the surrogate standards are calculated using the following equation:

$$\% Recovery = \frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{RRF_{si,rs}} \times \frac{1}{M_{si}} \times 100$$

where  $A_{rs}$  and  $A_{si}$  are the summed peak areas (from the primary and secondary m/z channels) of recovery standard and labelled surrogate added to the sample;  $M_{rs}$  and  $M_{si}$  are the masses of recovery standard and labelled surrogate added to the sample, and;

$RRF_{si,rs}$  is the mean relative response factor of the labelled surrogate to the recovery standard as determined by the five-point calibration range and defined individually as:

$$\frac{A_{si}}{A_{rs}} \times \frac{M_{rs}}{M_{si}}$$



## AXYS ANALYTICAL SERVICES LTD

Table 1. QC Acceptance Criteria for PCBs in CAL/VER, IPR, OPR, and Samples<sup>1</sup>

Congener	Cong. No. <sup>2</sup>	Test conc ng/mL	CAL/VER <sup>3</sup> (%)		IPR <sup>3</sup> (%)		OPR <sup>3</sup> (%)		Labelled compound recovery in samples	
			Warning Limit	Acceptance Limit	RSD	X	Warning Limit	Acceptance Limit (%)	Warning Limit	Acceptance Limit
2-MoCB	1	50	75-125	70-130	40	60-140	70-130	50-150	-	-
4-MoCB	3	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2'-DiCB	4	50	75-125	70-130	40	60-140	70-130	50-150	-	-
4,4'-DiCB	15	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',6-TrCB	19	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,4,4'-TrCB	37	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',6,6'-TeCB	54	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4'-TeCB	77	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,4,4',5-TeCB	81	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',4,6,6'-PeCB	104	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4'-PeCB	105	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,4,4',5-PeCB	114	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3',4,4',5-PeCB	118	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2',3,4,4',5-PeCB	123	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4',5-PeCB	126	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5-HxCB <sup>3</sup>	156	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5'-HxCB <sup>3</sup>	157	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	70-130	40	60-140	70-130	50-150	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',5,5',6,6'-OoCB	202	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,3,3',4,4',5,5',6-OoCB	205	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	70-130	40	60-140	70-130	50-150	-	-
2,2',3,3',4,5,5',6,6'-NoCB	208	50	75-125	70-130	40	60-140	70-130	50-150	-	-
DeCB	209	50	75-125	70-130	40	60-140	70-130	50-150	-	-
<b>Labelled Compounds</b>										
<sup>13</sup> C <sub>12</sub> -2-MoCB	1L	100	65-135	50-150	50	20-135	15-140	15-140	15-130	15-150
<sup>13</sup> C <sub>12</sub> -4-MoCB	3L	100	65-135	50-150	50	20-135	15-140	15-140	15-130	15-150
<sup>13</sup> C <sub>12</sub> -2,2'-DiCB	4L	100	65-135	50-150	50	35-135	30-140	30-140	25-130	25-150
<sup>13</sup> C <sub>12</sub> -4,4'-DiCB	15L	100	65-135	50-150	50	35-135	30-140	30-140	25-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',6-TrCB	19L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
<sup>13</sup> C <sub>12</sub> -3,4,4'-TrCB	37L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',6,6'-TeCB	54L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
<sup>13</sup> C <sub>12</sub> -3,3',4,4'-TCB	77L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
<sup>13</sup> C <sub>12</sub> -3,4,4',5-TeCB	81L	100	65-135	50-150	50	35-135	30-140	30-140	30-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',4,6,6'-PeCB	104L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4'-PeCB	105L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3,4,4',5-PeCB	114L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3',4,4',5-PeCB	118L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2',3,4,4',5-PeCB	123L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -3,3',4,4',5-PeCB	126L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',4,4',6,6'-HxCB	155L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5-HxCB <sup>3</sup>	156L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5'-HxCB <sup>3</sup>	157L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3',4,4',5,5'-HxCB	167L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -3,3',4,4',5,5'-HxCB	169L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',3,4',5,6,6'-HpCB	188L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5,5'-HpCB	189L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',3,3',5,5',6,6'-OoCB	202L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,3,3',4,4',5,5',6-OoCB	205L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',3,3',4,4',5,5',6-NoCB	206L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',3,3',4,4',5,5',6,6'-NoCB	208L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<sup>13</sup> C <sub>12</sub> -2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	65-135	50-150	50	35-135	30-140	30-140	40-130	25-150
<b>Cleanup Standard</b>										
<sup>13</sup> C <sub>12</sub> -2,4,4',4'-TriCB	28L	100	60-130	60-130	45	45-120	40-125	40-125	40-130	30-135
<sup>13</sup> C <sub>12</sub> -2,3,3',5,5'-PeCB	111L	100	60-130	60-130	45	45-120	40-125	40-125	40-130	30-135
<sup>13</sup> C <sub>12</sub> -2,2',3,3',5,5',6-HpCB	178L	100	60-130	60-130	45	45-120	40-125	40-125	40-130	30-135

1. QC acceptance criteria for IPR, OPR, and samples based on a 20 µL extract final volume
2. Suffix "L" indicates labelled compound.
3. PCBs 156 and 157 are tested as the sum of two concentrations



## AXYS ANALYTICAL SERVICES LTD

4. CAL VER: Calibration Verification test run at least every 12 hours
5. IPR: Initial Precision and Recovery demonstration
6. OPR: Ongoing Precision and Recovery test run with every batch of samples.

Table 1 con't

QC Parameter	Specification
Analysis Duplicate	Must agree to within $\pm 20\%$ of the mean (applicable to concentrations $> 10$ times the DL) <sup>1</sup>
Procedural Blank	Analyte concentrations in blank samples for PCB congeners 77, 81, 114, 123, 126 and 169 must be less than 2 pg/congener/sample, and concentrations of PCB congeners 156, 157, 167 and 189 must be less than 10 pg/congener/sample. Concentrations of all other individual PCB congeners or coelutions must be less than 50 pg/congener/sample in blank samples. The sum of all 209 congeners must be less than 300 pg/sample. Higher levels are acceptable where sample concentrations exceed 10x the blank levels.
Detection Limit	Typical sample specific detection limits for individual congeners, determined from chromatographic noise, range from 0.5 to 2.0 pg.
Initial Calibration	For 6-point calibration, a relative standard deviation of the RRF's $\leq 20\%$ for all compounds. Ion ratios for all congeners must be within $\pm 15\%$ of theoretical for CS 0.2. Minimum S:N ratio 10:1 for all calibration standards, except for CS0.2, where the S:N may be as low as 3:1 for di-PCBs and nona-PCBs.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Ion Ratios	Ion ratios must fall within $\pm 15\%$ of the theoretical values for positive identification of all targets in the calibration standards and samples.
Sensitivity	Minimum S:N ratio 10:1 for all calibration standards except for CS0.2, where the S:N may be as low as 3:1 for di-PCBs and nona-PCBs.

<sup>1</sup> Duplicate criterion is a guideline, final assessment depends upon sample characteristics, overall batch QC and on-going lab performance.

