Determination and Comparison of Planar Chlorinated Compounds in Lobster Tissue and Tomalley

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Abstract

The background levels of polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and co-planar polychlorinated biphenyls (CoPCBs) in lobsters was made by analysis of lobster meat and tomalley (liver) samples. The edible meat portion of the samples showed very low levels of these compounds. By comparison, in all cases the tomalley tissues showed significantly elevated levels of some isomers of PCDDs, PCDFs, and Co-PCBs. For 2,3,7,8-TCDD/TCDF isomers, lobster tomalleys presented elevated levels of 2,3,7,8-TCDF isomer ranging from 51.5 to 147 fold with a mean of 99.6 fold and elevated levels of 2,3,7,8-TCDD isomer ranging from 6.3 to 78.5 fold with a mean of 26.4 fold, as compared to the lobster meat. Lobster tomalleys also presented elevated levels of Co-PCB 126 as high as 262 fold.

Introduction

Lobster meat and tomalley (liver) samples from 12 lobsters were extracted and analyzed for polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzo-furans (PCDFs) and co-planar polychlorinated biphenyls (CoPCBs). Lobster tissues and tomalleys were prepared and analyzed using a method similar to the isotope dilution technique specified in US EPA Method 8290 with a modification to include Co-PCBs. Quality control (QC) samples such as matrix spike, matrix spike duplicate, laboratory control spike, and laboratory method blank were prepared and analyzed for each of the matrices. QC samples were used to test the laboratory performance, recovery, precision, accuracy, and background levels. A comparison of the results from each matrix was performed and is summarized below.

Sample Preparation

The lobster samples were received frozen and the meat was removed from the tail and claw. The meat was homogenized and chopped on a solvent-rinsed stainless steel sheet. The individual tomalleys also were homogenized prior to extraction.

Approximately 50 g of each meat or 20 g of tomalley was mixed with 150 g of sodium sulfate and the mixture was placed into a pre-extracted thimble and then into a Soxhlet extractor. Two additional portions of each sample matrix were prepared as a matrix spike/matrix spike duplicate (MS/MSD). A method blank consisting of pre-extracted sand

was prepared as well. A mixture of PCDD/F and CoPCB carbon 13-labeled internal quantitation standards were added to each thimble and extracted for 12 to 16 hr with 750 mL of 1:1 dichloromethane:hexane. The MS/MSD portions also were spiked with a known solution of native dioxins, furans, and coplanar PCBs to determine method precision and accuracy. After extraction, the solvent was evaporated and the percent lipid determined by weighing the flask. The lipid was re-dissolved in hexane and the extract was subjected to a 2 hr acid silica slurry (40% H_2SO_4 on silica gel) to remove bulk lipids. The extract was then eluted through a 6 g/1 g acid/neutral silica column. The eluent was further cleaned up by eluting through a neutral alumina column. The final fraction was eluted through a 0.6g column of AX-21/Celite 545®. The extract was then concentrated using prepurified nitrogen to approximately 100uL. Ten microliters of a recovery internal standard solution in tridecane was added, and the evaporation continued to a final volume of 10uL.

Sample Analysis

HRGC/HRMS sample analysis was performed using a Hewlett-Packard 5890 gas chromatograph interfaced to a VG70S-250 high resolution mass spectrometer. A DB-5MS gas chromatography column (60 meter, 0.25 mm ID, 0.25 μm film thickness) was used under conditions specific for 2,3,7,8-TCDD. Mass spectrometry analysis was performed operating at 10,000 resolving power (resolution=100 ppm at 5% peak height) in the selected ion monitoring mode (SIM).

The initial calibration consisted of a series of five calibration standards ranging from 0.25 to 40 pg/μL for tetra-substituted isomers, 1.25 to200 pg/μL for penta through hepta-substituted isomers, and 2.5 to 400 pg/μL for octa-substituted isomer. Co-PCB isomers ranged from 1 to 250 pg/μL. The initial calibration passed the criteria of less than 20% relative standard deviation for all compounds.

Continuing calibration using a mid-level standard passed the criteria of less than 20% difference from the initial calibration.

Data acquisition for PCDD/F and Co-PCB were achieved simultaneously in a single HRGC/HRMS analysis.

Results and Discussion

The sample results presented in Table 1 are given in pg/g on a whole weight basis. The results from the lobster meat is presented with the data for the lobster tomalley from the corresponding sample. The method blank results are presented first, followed by the sample results. Detection limits were based upon the low calibration standard. The method blanks showed no 2,3,7,8-substituted isomers above the detection limit. A small amount of Co-PCBs #80 and #77 were detected but at concentrations less than half of the lowest value found any of the samples.

Overall, few PCDD/F isomers were detected in the meat samples, but most isomers were present at detectable concentration levels in the tomalley samples. Each tomalley showed low levels of 2,3,7,8-TCDD ranging from 0.466 to 8.01 pg/g. Most meat samples showed low levels (around 0.5 ppt) of 2,3,7,8-TCDF. However, in the corresponding tomalley sample the concentration was typically more than 51 times higher. This elevated level is

exaggerated to 147 fold in PRST-2 sample. In addition, each tomalley sample contained low levels of non 2,3,7,8-substituted isomers.

In general, the Co-PCBs detected in the tomalley samples were 10 to 200 times the concentration found in the corresponding meat sample. Co-PCB #80 was detected in both the meat and tomalley samples, and notably in each tomalley sample a saturated peak response was observed.

International Toxicity Equivalents (I-TEs) were calculated for each sample using I-TEs for PCDD/F as found in *Chemosphere*, 20(7-9):751-757 (1990). International Toxicity Equivalents (I-TEs) were calculated for each sample using I-TEs for CoPCB #77, 126, and 169 as found in *Chemosphere*, 28(6):1049-1067 (1994). The detection limits were used in the calculation for any undetected isomers.

Labeled analog recoveries averaged between 59.3 and 91.0 %, with a standard deviation between 7 and 31. High recoveries for 13C-HPCDD and 13C-OCDD were observed in a few samples and were believed to be due to sample matrix interferences, which also caused some disruptions in the reference compound perfluorokerosene (PFK), which was monitored to illustrate any changes in the stability and sensitivity of the instrument during the analysis.
Results for the meat and tomalley MS/MDS showed good method precision and accuracy. The relative percent difference was less than 17 for all isomers. Results for Co-PCB #80 in the tomalley are not reported due to saturated peak responses for this isomer. In addition, the results for Co-PCB #127 in the tomalley are not reported due to a co-eluting peak interference.

Acknowledgments
Midwest Research Institute would like to thank the Maine Department of Environmental Protection and Mr. Barry Mower of the Surface Water Ambient Toxics Monitoring Program for their support in conducting this study.

Table 1. SAMPLE RESULTS SUMMARY
Lobster Meat and Tomalley (PG/G, whole weight)

Isomer	Sample ID	Method Blank Meat	Method Blank Tomalley	BVB-1Meat	BVB-1Tomalley	BVB-2Meat	BVB-2Tomalley	KNB-1Meat	KNB-1Tomalley
2378TCDF		ND(.114 cdl)(a)	ND(.217 cdl)	0.232	21.7	0.226	17.8	0.673	82.7
2378TCDD		ND(.096 cdl)	ND(.204 cdl)	ND(.0775 cdl)	1.43	ND(.0838 cdl)	1.13	ND(.102 mpc)	8.01
12378PECDF		ND(.542 cdl)	ND(.847 cdl)	ND(.52 cdl)	ND(6.89 mpc)	ND(.513 cdl)	ND(4.83 mpc)	ND(.39 cdl)	ND(12.1 mpc)
23478PECDF		ND(.542 cdl)	ND(.847 cdl)	ND(.52 cdl)	8.14	ND(.513 cdl)	6.38	ND(.39 cdl)	12.6
12378PECDD		ND(.465 cdl)	ND(1.02 cdl)	ND(.444 cdl)	4.63	ND(.346 cdl)	3.67	ND(.384 cdl)	6.13
123478HXCDF		ND(.509 cdl)	ND(1.11 cdl)	ND(1.38)	0.936	ND(.934)	ND(1.05)	ND(.487 cdl)	ND(.959 cdl)
123678HXCDF		ND(.509 cdl)	ND(1.11 cdl)	ND(1.27)	ND(11.6 mpc)	ND(.86)	ND(8.24 mpc)	ND(.487 cdl)	ND(17.1 mpc)
234678HXCDF		ND(.509 cdl)	ND(1.11 cdl)	ND(1.25 mpc)(b)	ND(1.86 mpc)	ND(.829)	ND(2.38 mpc)	ND(.549 mpc)	ND(4.19 mpc)
123789HXCDF		ND(.509 cdl)	ND(1.11 cdl)	ND(1.79)	ND(.926 cdl)	ND(1.21)	ND(1.18 mpc)	ND(.487 cdl)	ND(2 mpc)
123478HXCDD		ND(.427 cdl)	ND(.941 cdl)	ND(.444 cdl)	1.47	ND(.375 cdl)	ND(.899 cdl)	ND(.391 cdl)	ND(.984 cdl)
123678HXCDD		ND(.427 cdl)	ND(.941 cdl)	ND(.444 cdl)	5.17	ND(.375 cdl)	4.01	ND(.391 cdl)	11.3
123789HXCDD		ND(.427 cdl)	ND(.941 cdl)	ND(.444 cdl)	1.35	ND(.375 cdl)	1.49	ND(.391 cdl)	3.51
1234678HPCDF		ND(.551 cdl)	ND(1.03 cdl)	ND(.734 cdl)	ND(9.29 mpc)	ND(.557 cdl)	ND(3.62 mpc)	ND(.564 cdl)	ND(9.89 mpc)
1234789HPCDF		ND(.551 cdl)	ND(1.03 cdl)	ND(.734 cdl)	ND(1.14)	ND(.557 cdl)	ND(.513 cdl)	ND(.564 cdl)	ND(.586 cdl)
1234678HPCDD		ND(.471 cdl)	ND(.929 cdl)	ND(.283 cdl)	5.26	ND(.423 cdl)	3.44(c)	ND(.455 cdl)	11.8(c)
12346789OCDF		ND(1.08 cdl)	ND(2.24 cdl)	ND(.344 cdl)	ND(1.69 cdl)	ND(.909 cdl)	ND(.55 cdl)(c)	ND(.761 cdl)	ND(.404 cdl)(c)
12346789OCDD		ND(1.08 cdl)	ND(2.24 cdl)	ND(.427 mpc)	2.99	ND(.909 cdl)	1.79(c)	0.927	8.3(c)
80-TPCB		16.3	16.4	65.7	2130(d)	77.1	1740(d)	151	2250(d)
79-TPCB		ND(.441 cdl)	ND(.718 cdl)	ND(.343 cdl)	23.3	ND(.3 cdl)	19.4	ND(.292 cdl)	30.4
78-TPCB		ND(.441 cdl)	ND(.718 cdl)	ND(.343 cdl)	ND(.651 cdl)	ND(.3 cdl)	ND(.595 cdl)	ND(.292 cdl)	ND(.563 cdl)
81-TPCB		ND(.441 cdl)	ND(.718 cdl)	0.346	27	0.368	20.9	0.559	59
77-TPCB		1.59	1.11	6.95	624	6.25	456	11.2	940
127-PePCB		ND(.355 cdl)	ND(.692 cdl)	ND(.342 cdl)	ND(.633 cdl)	ND(.299 cdl)	ND(.605 cdl)	ND(.292 cdl)	ND(.577 cdl)
126-PePCB		ND(.355 cdl)	ND(.692 cdl)	1.09	161	1.24	118	1.62	275
169-HxPCB		ND(.287 cdl)	ND(.726 cdl)	ND(.298 cdl)	30.5	ND(.194 cdl)	21.3	ND(.249 cdl)	34.8
PCDD/F I-TE		0.99	1.96	1.33	12.82	1.07	10.18	0.91	30.49
CoPCB I-TE		0.04	0.08	0.12	16.72	0.13	12.24	0.17	28.32
Total I-TE		1.03	2.04	1.44	29.54	1.20	22.42	1.08	58.80

(a)-Not detected, with the curve based detection limit in pg/g.
(b)-Not detected due to an interference. An estimated maximum possible concentration (MPC) is given as the detection limit in pg/g.
(c)-Corresponding labeled analog recovery outside criteria.
(d)-Peak saturated.

Isomer	Sample ID	KNB-2Meat	KNB-2Tomalley	MCH-1Meat	MCH-1Tomalley	MCH-2Meat	MCH-2Tomalley	PRST-1Meat	PRST-1Tomalley	PRST-2Meat
2378TCDF		0.836	62.4	0.123	17.6	ND(.134 mpc)	10.3	0.265	31.2	ND(.266 mpc)
2378TCDD		ND(.123 mpc)	5.06	ND(.0629 cdl)	0.949	ND(.0745 cdl)	0.466	ND(.0677 cdl)	1.88	ND(.0951 cdl)
12378PECDF		ND(.328 cdl)	ND(9.48 mpc)	ND(.258 cdl)	ND(5.26 mpc)	ND(.405 cdl)	2.78	ND(.356 cdl)	ND(10.5 mpc)	ND(.651 cdl)
23478PECDF		ND(.328 cdl)	8.87	ND(.258 cdl)	5.3	ND(.405 cdl)	4.44	ND(.356 cdl)	8.47	ND(.651 cdl)
12378PECDD		ND(.302 cdl)	4.86	ND(.265 cdl)	2.77	ND(.357 cdl)	1.74	ND(.325 cdl)	4.8	ND(.4 cdl)
123478HXCDF		ND(3.63)	ND(.906 cdl)	ND(.603)	ND(1.65 mpc)	ND(.366 cdl)	ND(1.16)	ND(.464 mpc)	ND(1.85)	ND(.867)
123678HXCDF		ND(3.34)	ND(19.9 mpc)	ND(.555)	ND(6.16 mpc)	ND(.366 cdl)	ND(2.37 mpc)	ND(.384 mpc)	ND(16.7 mpc)	ND(.798)
234678HXCDF		ND(3.22)	ND(.906 cdl)	ND(.926 mpc)	ND(2.84 mpc)	ND(.366 cdl)	ND(1.37 mpc)	ND(.789 mpc)	ND(3.89 mpc)	ND(.705 mpc)
123789HXCDF		ND(4.71)	ND(3.6 mpc)	ND(.784)	ND(1.09 cdl)	ND(.366 cdl)	ND(1.51)	ND(.215 mpc)	ND(2.4)	ND(1.13)
123478HXCDD		ND(.472 cdl)	ND(1.09 cdl)	ND(.348 cdl)	ND(1.03 cdl)	ND(.325 cdl)	ND(.829 cdl)	ND(.369 cdl)	ND(1.26 cdl)	ND(.368 cdl)
123678HXCDD		ND(.472 cdl)	9.5	ND(.348 cdl)	3.04	ND(.325 cdl)	1.61	ND(.369 cdl)	10.3	ND(.368 cdl)
123789HXCDD		ND(.472 cdl)	3.81	ND(.348 cdl)	ND(1.19 mpc)	ND(.325 cdl)	ND(.949 mpc)	ND(.369 cdl)	ND(4 mpc)	ND(.368 cdl)
1234678HPCDF		ND(.478 cdl)	ND(.736 cdl)	ND(.428 cdl)	ND(5.17 mpc)	ND(.444 cdl)	ND(1.63 mpc)	ND(.395 cdl)	ND(.877 cdl)	ND(.558 cdl)
1234789HPCDF		ND(.478 cdl)	ND(.736 cdl)	ND(.428 cdl)	ND(1.07 cdl)	ND(.444 cdl)	ND(.671 cdl)	ND(.395 cdl)	ND(.877 cdl)	ND(.558 cdl)
1234678HPCDD		0.416(c)	9.8(c)	0.373(c)	7.83	ND(.312 cdl)	11.2(c)	0.619(c)	25.9(c)	ND(.527 mpc)
123467890CDF		ND(.108 cdl)(c)	ND(.272 cdl)(c)	0.483(c)	ND(1.37 cdl)	ND(.624 cdl)	ND(.873 cdl)	ND(.158 cdl)(c)	ND(.653 cdl)(c)	ND(.147 cdl)(c)
123467890CDD		0.868(c)	7.11(c)	1.14(c)	6.22	ND(.624 cdl)	9.04	2.24(c)	21.1(c)	0.855(c)
80-TPCB		172	1660(d)	39.2	2890(d)	40.3	1600(d)	130	2980(d)	149
79-TPCB		ND(.296 cdl)	22.6	ND(.221 cdl)	24.1	ND(.34 cdl)	13.8	ND(.289 cdl)	30.4	ND(.372 cdl)
78-TPCB		ND(.296 cdl)	ND(.607 cdl)	ND(.221 cdl)	ND(.633 cdl)	ND(.34 cdl)	ND(.689 cdl)	ND(.289 cdl)	ND(.835 cdl)	ND(.372 cdl)
81-TPCB		0.521	43.4	ND(.221 cdl)	18.4	ND(.34 cdl)	9.12	0.339	51.9	0.377
77-TPCB		13.4	837	3.07	477	3.14	232	7.7	1020	8.13
127-PePCB		ND(.286 cdl)	ND(.622 cdl)	ND(.244 cdl)	ND(.658 cdl)	ND(.276 cdl)	ND(.657 cdl)	ND(.252 cdl)	ND(.917 cdl)	ND(.32 cdl)
126-PePCB		1.86	177	0.468	112	0.339	59	1.27	247	1.21
169-HxPCB		0.228	26.4	0.301	17	ND(.238 cdl)	14.3	ND(.228 cdl)	25.1	ND(.262 cdl)
PCDD/F I-TE		2.18	22.73	0.75	8.86	0.75	5.85	0.76	16.50	1.16
CoPCB I-TE		0.19	18.38	0.05	11.61	0.04	6.16	0.13	25.46	0.13
Total I-TE		2.38	41.11	0.81	20.46	0.78	12.01	0.90	41.96	1.29

Isomer	Sample ID	PRST-2Tomalley	SCO-1Meat	SCO-1Tomalley	SCO-2Meat	SCO-2Tomalley	VER-1Meat	VER-1Tomalley	VER-2Meat	VER-2Tomalley
2378TCDF		39.1	0.246	23	0.221	20.6	0.368	37.6	0.746	ND(38.4 mpc)
2378TCDD		3.08	ND(.0803 cdl)	1.43	ND(.0801 cdl)	1.2	ND(.0826 cdl)	2.24	0.104	2.45
12378PECDF		ND(19.9 mpc)	ND(.405 cdl)	ND(7.98 mpc)	ND(.376 cdl)	ND(5.88 mpc)	ND(.495 cdl)	ND(16.1 mpc)	ND(.395 cdl)	ND(13.7 mpc)
23478PECDF		17.9	ND(.405 cdl)	8.18	ND(.376 cdl)	6.37	ND(.495 cdl)	12.8	ND(.395 cdl)	11.7
12378PECDD		6.95	ND(.404 cdl)	4.17	ND(.326 cdl)	3.18	ND(.49 cdl)	8.23	ND(.334 cdl)	7.81
123478HXCDF		ND(1.12 mpc)	ND(.416 cdl)	ND(1.27 mpc)	ND(.372 cdl)	ND(1.06 cdl)	ND(.488 cdl)	ND(1.56 mpc)	ND(.412 cdl)	ND(1.3 mpc)
123678HXCDF		ND(26.1 mpc)	ND(.427 mpc)	ND(14.1 mpc)	ND(.372 cdl)	ND(10.1 mpc)	ND(.526 mpc)	ND(59.9 mpc)	ND(.96 mpc)	ND(51.9 mpc)
234678HXCDF		ND(2.49 mpc)	ND(.416 cdl)	ND(1.42 mpc)	ND(.372 cdl)	ND(1.61 mpc)	ND(.488 cdl)	ND(2.95 mpc)	ND(.412 cdl)	ND(2.74 mpc)
123789HXCDF		ND(1.76 mpc)	ND(.416 cdl)	ND(.944 cdl)	ND(.372 cdl)	ND(1.06 cdl)	ND(.488 cdl)	ND(1.23 mpc)	ND(.412 cdl)	ND(.759 mpc)
123478HXCDD		3.37	ND(.38 cdl)	1.67	ND(.375 cdl)	1.16	ND(.427 cdl)	ND(.971 cdl)	ND(.391 cdl)	ND(.966 cdl)
123678HXCDD		11.8	ND(.38 cdl)	5.65	ND(.375 cdl)	3.7	ND(.427 cdl)	25.8	0.527	28
123789HXCDD		3.31	ND(.38 cdl)	ND(2.19 mpc)	ND(.375 cdl)	1.12	ND(.427 cdl)	7.4	ND(.391 cdl)	7.27
1234678HPCDF		ND(36.3 mpc)	ND(.628 cdl)	ND(42.2 mpc)	ND(.519 cdl)	ND(9.71 mpc)	0.701	ND(120 mpc)	ND(1.19 mpc)	ND(55.5 mpc)
1234789HPCDF		ND(3.49)	ND(.628 cdl)	ND(.936 cdl)	ND(.519 cdl)	ND(1.01 cdl)	ND(.559 cdl)	ND(.75 cdl)	ND(.483 cdl)	ND(1.07)
1234678HPCDD		24.8	ND(.242 cdl)	6.36	ND(.275 cdl)	4.69	0.659	31.2	1.01	37.1(c)
12346789OCDF		ND(2.08 cdl)	ND(.48 cdl)	ND(1.38 cdl)	ND(.568 cdl)	ND(1.39 cdl)	ND(.643 cdl)	ND(.836 cdl)(c)	ND(.758 cdl)	ND(.816 cdl)(c)
12346789OCDD		29.9	ND(.48 cdl)	3.16	ND(.568 cdl)	5.96	1.78	19.2	2.64	34.1(c)
80-TPCB		2490(d)	80	1700(d)	82.1	1830(d)	149	1590(d)	127	1850(d)
79-TPCB		49.7	ND(.352 cdl)	32.6	ND(.312 cdl)	29.6	ND(.359 cdl)	20.5	ND(.294 cdl)	19.4
78-TPCB		ND(.762 cdl)	ND(.352 cdl)	ND(.685 cdl)	ND(.312 cdl)	ND(.7 cdl)	ND(.359 cdl)	ND(.654 cdl)	ND(.294 cdl)	ND(.569 cdl)
81-TPCB		68.4	ND(.352 cdl)	21	ND(.312 cdl)	19.4	0.393	28.7	0.401	20.9
77-TPCB		1060	5.83	531	5.27	531	7.61	584	9.39	459
127-PePCB		ND(.701 cdl)	ND(.307 cdl)	ND(.679 cdl)	ND(.278 cdl)	ND(.677 cdl)	ND(.312 cdl)	ND(.618 cdl)	ND(.273 cdl)	ND(.609 cdl)
126-PePCB		317	1.22	166	0.9	128	1.05	142	1.46	113
169-HxPCB		39.9	ND(.282 cdl)	25.7	ND(.209 cdl)	20.3	ND(.32 cdl)	19.1	0.205	17.4
PCDD/F I-TE		26.08	0.83	13.53	0.75	10.47	0.99	28.84	0.94	27.00
CoPCB I-TE		32.63	0.13	17.12	0.09	13.07	0.11	14.68	0.15	11.70
Total I-TE		58.71	0.95	30.65	0.84	23.54	1.10	43.52	1.10	38.70

Table 2. MS/MSD Recovery & Precision

Isomer	Lobster Meat		Lobster Tomalley	
	Average Recovery	%RPD(a)	Average Recovery	%RPD(a)
2378TCDF	109	5.5	78	10.3
2378TCDD	108	12.1	91	6.6
12378PECDF	100	9.0	125	4.0
23478PECDF	111	4.5	109	3.7
12378PECDD	103	4.9	98	9.2
123478HXCDF	105	7.6	111	1.8
123678HXCDF	113	1.8	139	2.9
234678HXCDF	101	7.9	104	2.9
123789HXCDF	109	12.0	103	14.6
123478HXCDD	113	11.6	97	5.2
123678HXCDD	101	2.0	91	11.0
123789HXCDD	91	11.0	84	4.8
1234678HPCDF	117	4.3	124	7.3
1234789HPCDF	95	16.8	81	4.9
1234678HPCDD	101	5.9	100	0.0
12346789OCDF	95	5.3	101	7.0
12346789OCDD	94	7.5	99	1.0
80-TPCB	104	1.9	-(b)	-
79-TPCB	98	2.0	92	4.3
78-TPCB	101	1.0	92	3.3
81-TPCB	103	3.9	97	4.1
77-TPCB	100	1.0	99	4.0
127-PePCB	97	2.1	-(c)	-
126-PePCB	101	3.0	98	2.0
169-HxPCB	103	1.0	93	2.2

(a)-Percent relative difference = (high-low)/average X 100.
(b)-Unable to calculate recovery due to peak saturation for this isomer.
(c)-Unable to calculate recovery due to an interference co-eluting with this peak.