

Inter-calibration Exercise on Organics

- KORDI -

Sang Hee Hong

Laboratory Facility



Lab. Equipment

1. Analytical instruments

- GC (3): HP 5890 with μ -ECD(1), and FPD(1), HP 6890 with FPD(1)
- GC/MS (5): HP 5972 (2), HP 5975N (1), Shimadzu QP 2010, Shimadzu QP 5000
- GC-C/IRMS (1):
- GCXGC (1): Thermo
- HPLC/MS/MS (1): Agilent 1200 with API3200
- HPLC (3): HP 1050 series with UV and fluorescence(1), prep HPLC (2)
- Fluorescence spectrophotometer (1), Plate reader (1)

2. Technical equipment

- accelerated solvent extractor (1), shaker (2), vacuum rotary evaporator (3), centrifuge (3), vortex mixer (5), nitrogen blower (3), furnace (3), oven (4), vacuum oven (1), deep freezer (1), distillers (1), balance (4), tumbler (1), high volume water sampler (1), high volume air sampler (4)

3. Other laboratory apparatus

- 99.999% pure gas supplies with stainless steel gas line and gas purifiers



Apparatus and Materials

- Glassware

Cleaning using Micro detergent → washing with tap water → combustion in a muffle furnace at 400°C for at least 4hr → Solvent rinse before use

- Reagents and Consumable Materials

- Aluminum Oxide (alumina): Aldrich; 80-200 mesh, or equivalent; combusted at 440°C for 4 hours and stored at 130°C

- Chromatographic Silica: Davisil; silica gel, grade 12, 100-200 mesh or equivalent; activated at 170°C for at least 24 hours and stored at 170°C

- Sodium Sulfate: Fisher; activated at 450°C for at least 5 hours and stored at 120°C

- Solvents: Checking the purity by GC/ECD and GC/MS prior to use

 - Methylene chloride; Burdick and Jackson; GC² grade (higher than pesticide grade).

 - Methanol; Burdick and Jackson; GC² grade (higher than pesticide grade).

 - Hexane; Burdick and Jackson; GC² grade (higher than pesticide grade).

 - Toluene; Burdick and Jackson; GC² grade (higher than pesticide grade).

List of CRMs in Lab.

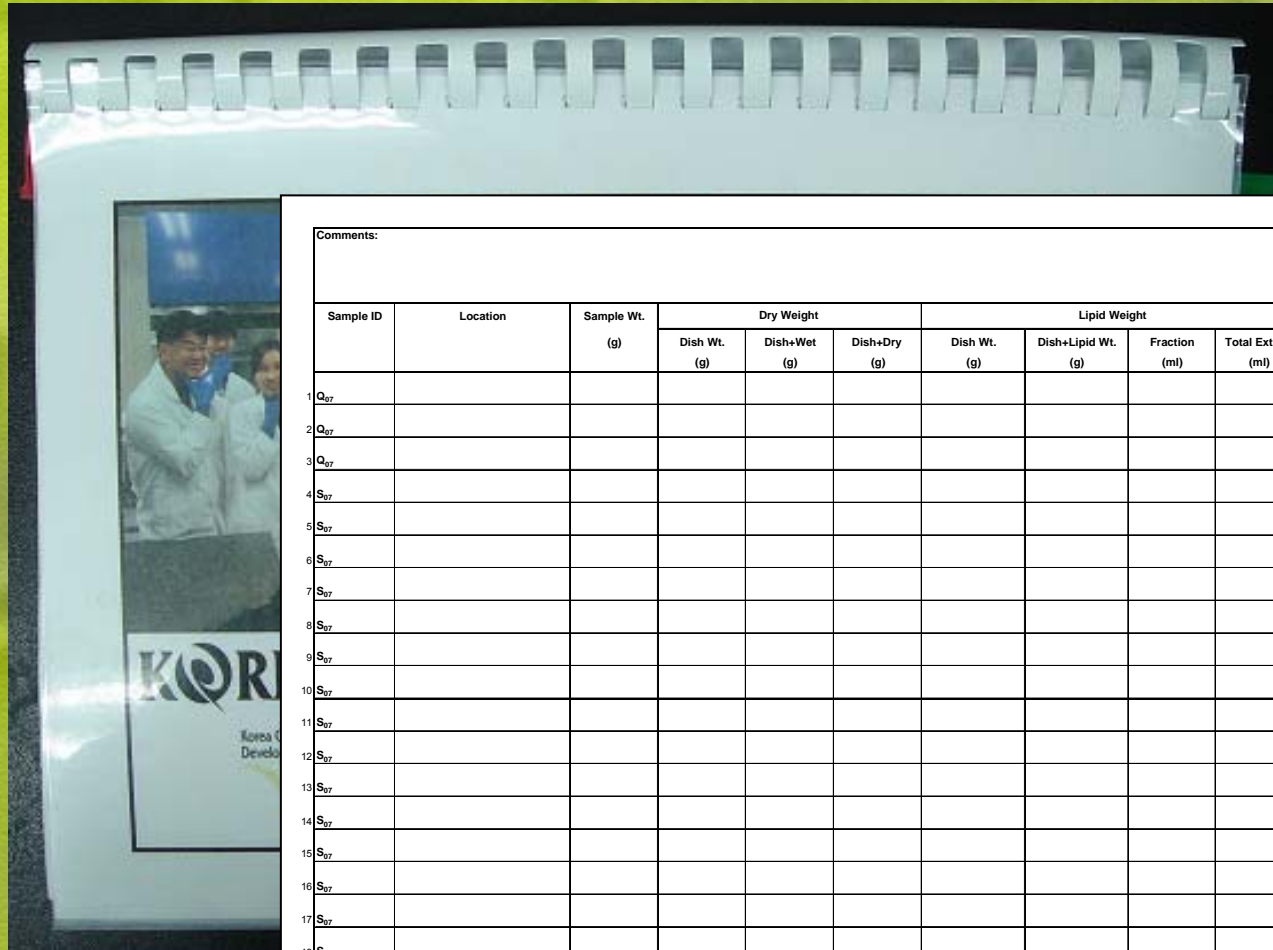
NIST (National Institute of Standards and Technology)	Content
1941	marine sediment
1941b	marine sediment
2977	Mussel Tissue
2978	Mussel Tissue
1946	Fish Tissue

Environment Canada	Content
EC-1	Sediment
EC-2	Sediment
EC-4	Sediment
EC-5	Sediment
SES-1	Marine sediment

IAEA	Content
IAEA-417	Marine sediment
IAEA-432	Mussel Tissue
IAEA-142	Mussel Tissue
IAEA-140	Sea plant
IAEA-435	Tuna
IAEA-406	Fish homogenate
MA-A-3/oc	Shrimp
MA-B-3/oc	Fish homogenate

Others	Content
IMPC-21	Sewage sludge

Laboratory Log Book



Comments:										Matrix	Sample Preparation:	
										Sediment: <input type="checkbox"/>	Date	Analyst
										Tissue: <input type="checkbox"/>	Init:	
										Other: <input type="checkbox"/>		
Sample ID	Location	Sample Wt. (g)	Dry Weight			Lipid Weight				Comments	Surrogate Spiking:	
			Dish Wt. (g)	Dish+Wet (g)	Dish+Dry (g)	Dish Wt. (g)	Dish+Lipid Wt. (g)	Fraction (ml)	Total Extract (ml)		Surrogate ID:	Amount:
1 Q ₂₇											Surrogate ID:	Amount:
2 Q ₂₇											Surrogate ID:	Amount:
3 Q ₂₇											Date	Analysts
4 S ₂₇											Init:	Witness Init:
5 S ₂₇											Extraction:	Analyst
6 S ₂₇											Date	Init:
7 S ₂₇											Concentration for S/AI:	Analyst
8 S ₂₇											Date	Init:
9 S ₂₇											Silica gel/Alumina Clean up:	Analyst
10 S ₂₇											Date	Init:
11 S ₂₇											Concentration for HPLC:	Analyst
12 S ₂₇											Date	Init:
13 S ₂₇											HPLC Clean up:	Analyst
14 S ₂₇											Date	Init:
15 S ₂₇											Conc. for Instr. Analysis:	Analyst
16 S ₂₇											Date	Init:
17 S ₂₇											Int. Std. Spiking:	Surrogate ID:
18 S ₂₇											Amount:	Surrogate ID:
19 S ₂₇											Amount:	Date
20 S ₂₇											Analysts	Init:
21 S ₂₇											Witness Init:	Instrumental Analysis:
										GC-ECD		
										GC-MSD		

Data File _____

Sample storage system



Test samples and CRMs

- Average weight of sample extracted



Sample ID	Average weight (g)
MA-YSLME/ORG/1	3.05 ± 0.02
IAEA 435	2.08 ± 0.01
SD-YSLME/ORG/1	1.03 ± 0.03
IAEA 417	0.57 ± 0.02

- Moisture content



Sample ID	Moisture content (%)
MA-YSLME/ORG/1	3.6 ± 0.15
IAEA 435	4.7 ± 0
SD-YSLME/ORG/1	0.73 ± 0.12
IAEA 417	0.49 ± 0.05

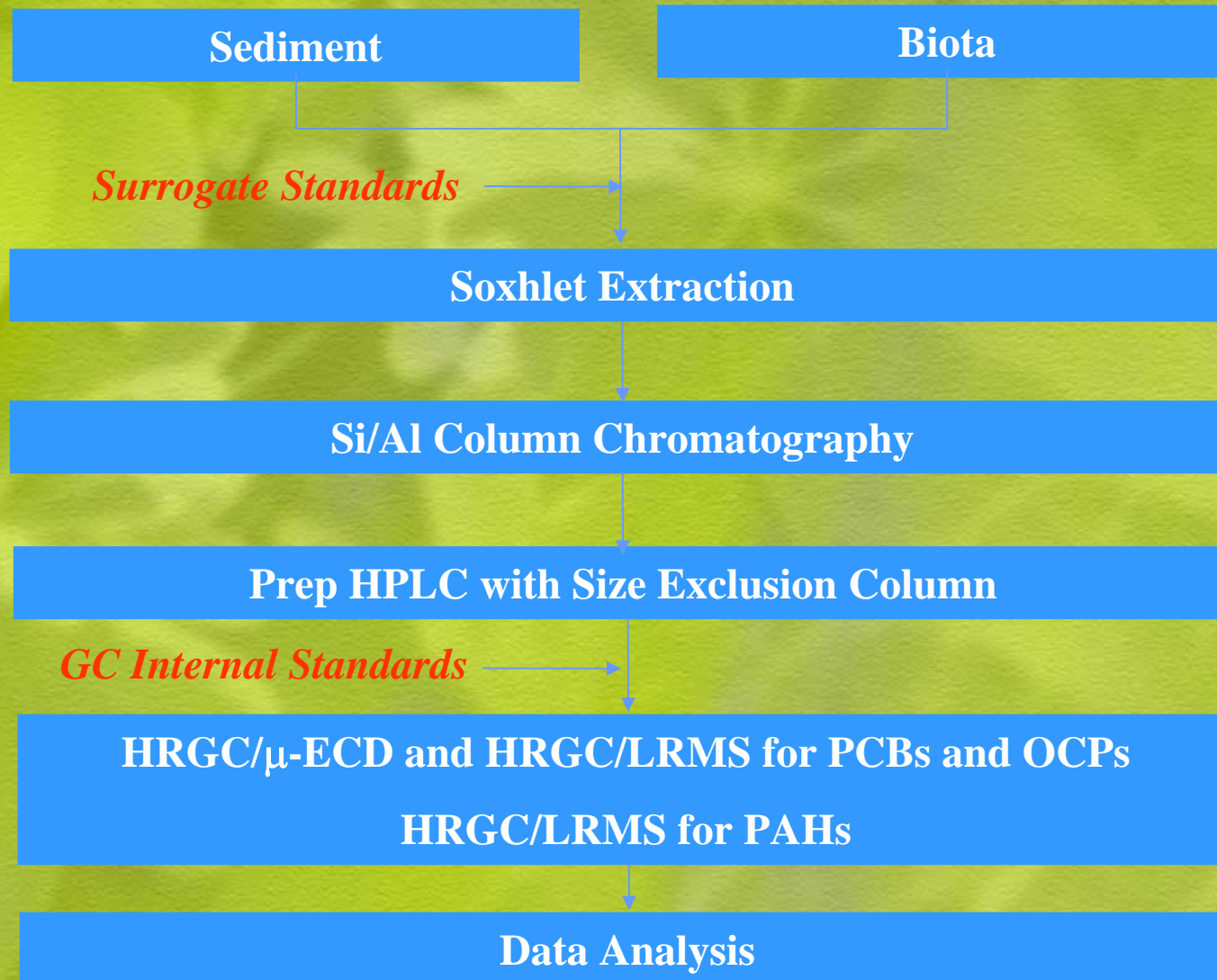
Sample batch in this study

- Blank sample (#2)
- MA-YSLME/ORG/1 (#5)
- SD-YSLME/ORG/1 (#5)
- IAEA 435 (#3)
- IAEA 417 (#3)
- Matrix spike sample (#2) – 500 ng for PAHs,
20 ng for PCBs and OCPs

The image shows a page from a laboratory log book titled 'LABORATORY LOG BOOK'. The page contains a table with columns for 'Sample ID', 'Location', 'Sample', 'Dry Weight', and 'Total Extract'. The table is filled with handwritten entries, including sample IDs like '141', '142', '143', '144', '145', '146', '147', '148', '149', '150', '151', '152', '153', '154', '155', '156', '157', '158', '159', '160'. The entries include sample descriptions such as 'Blank sample', 'Matrix spike', and 'IAEA 435'. The table also has columns for 'Date', 'Time', 'Initials', and 'Comments'. The page is signed 'M. J. H. H.' and dated '21.09.04'.

Sample ID	Location	Sample	Dry Weight	Total Extract	Comments
141	Blank sample				
142	Matrix spike				
143	IAEA 435				
144	MA-YSLME/ORG/1				
145	SD-YSLME/ORG/1				
146	IAEA 417				
147	IAEA 417				
148	IAEA 417				
149	IAEA 417				
150	IAEA 417				
151	IAEA 417				
152	IAEA 417				
153	IAEA 417				
154	IAEA 417				
155	IAEA 417				
156	IAEA 417				
157	IAEA 417				
158	IAEA 417				
159	IAEA 417				
160	IAEA 417				

Diagram of Analytical Procedure



Extraction



Dehydrated sample with sodium sulfate

Si/Al column of chromatography



Silica gel
Alumina

GC/ECD & GC/MS



HPLC



Internal Standards

CH Internal standard

Dibromooctafluorobiphenyl (DBOFB)

PCB103

PCB198

AH Internal standard

Naphthalene-d8

Acenaphthene-d10

Phenanthrene-d12

Chrysene-d10

Perylene-d12

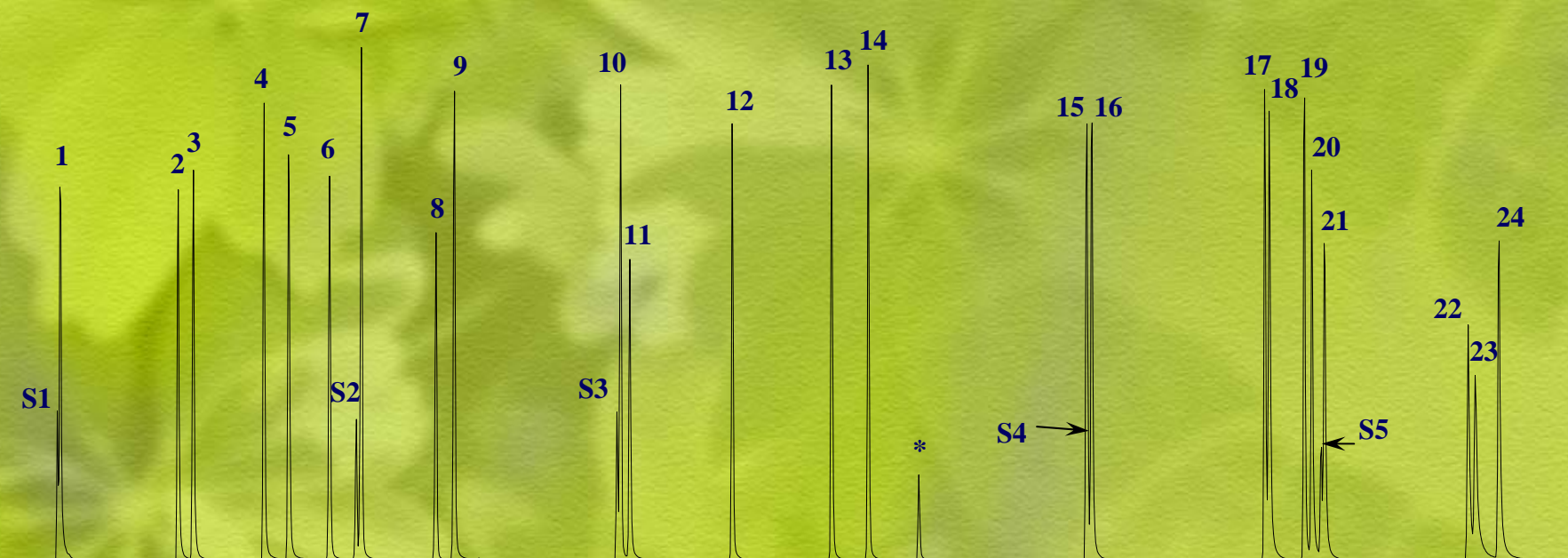
CH GC Internal standard

Tetrachloro-m-xylene (TCMX)

AH GC Internal standard

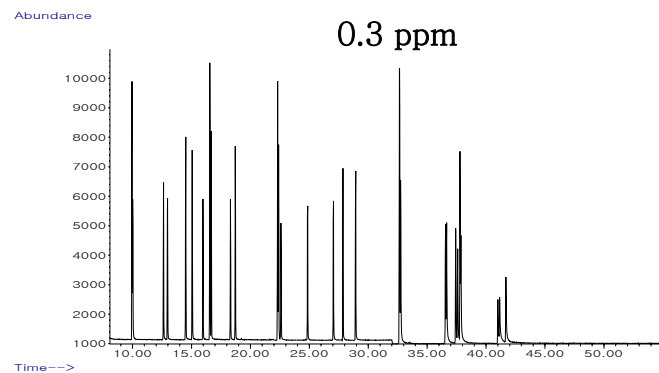
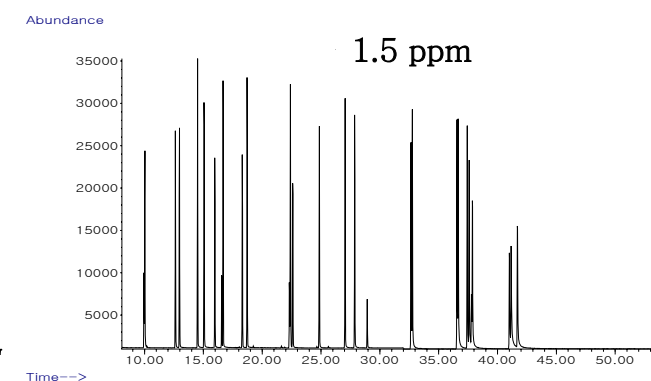
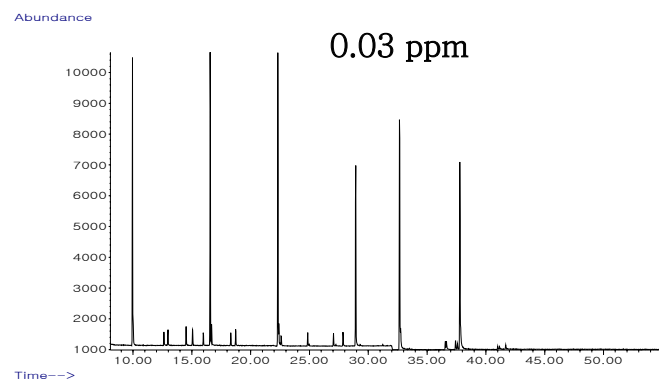
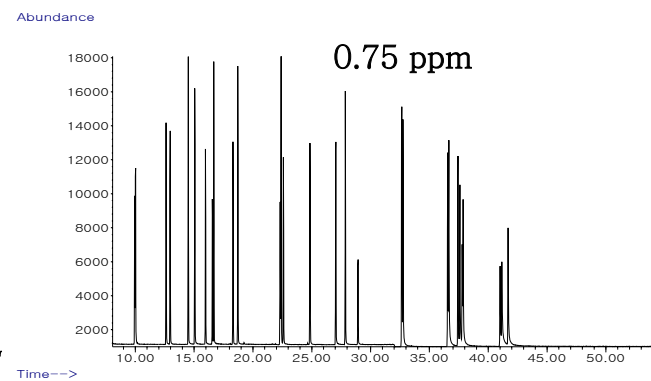
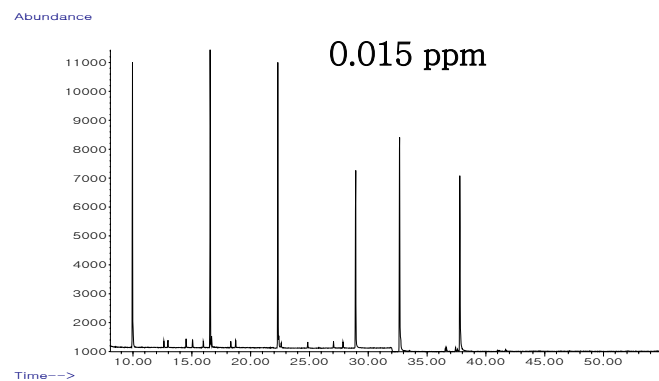
Terphenyl-d14

Calibration standard : PAH mixture

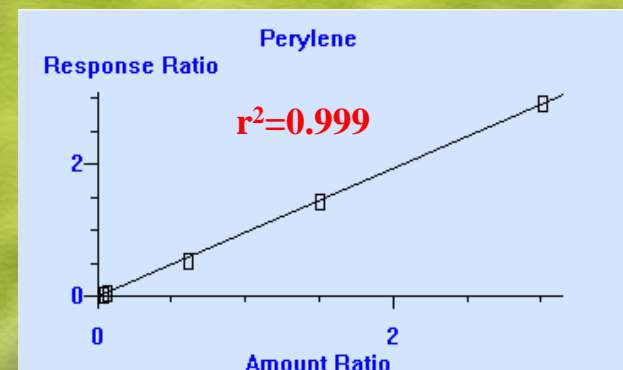
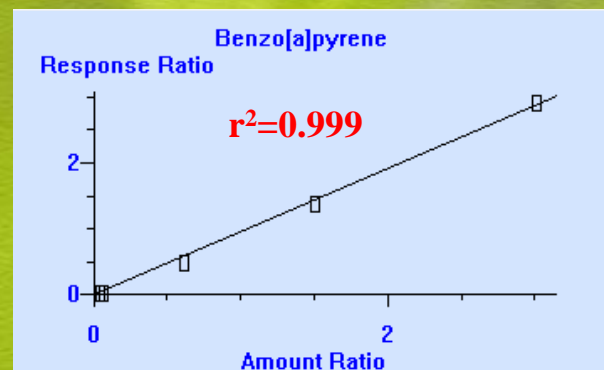
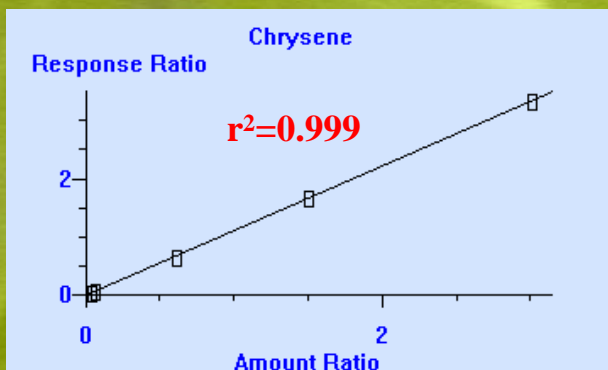
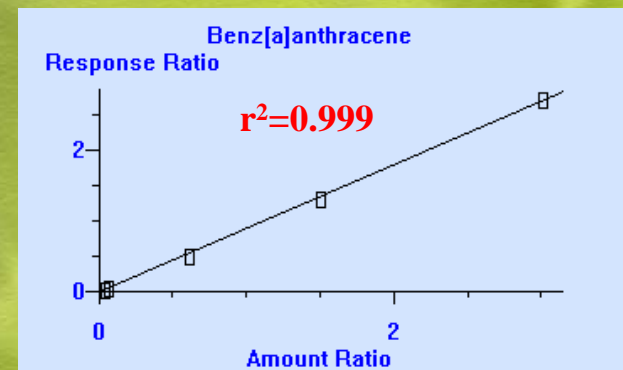
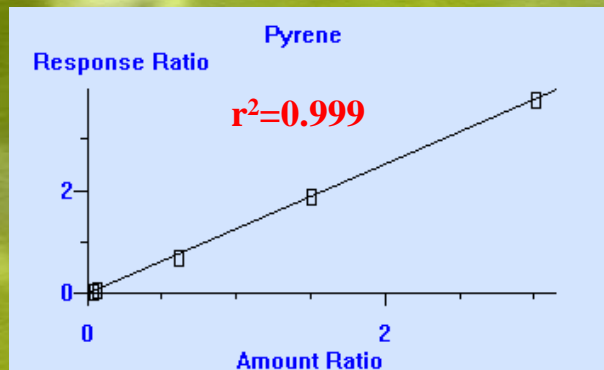
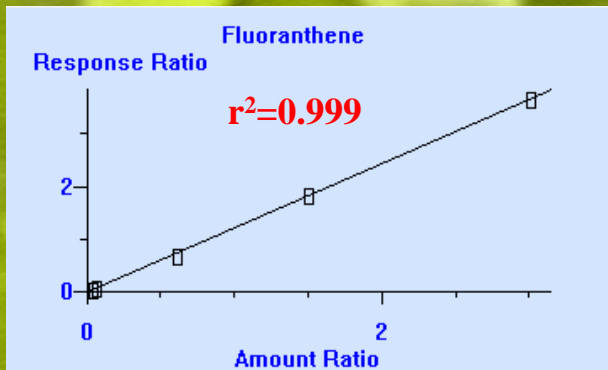
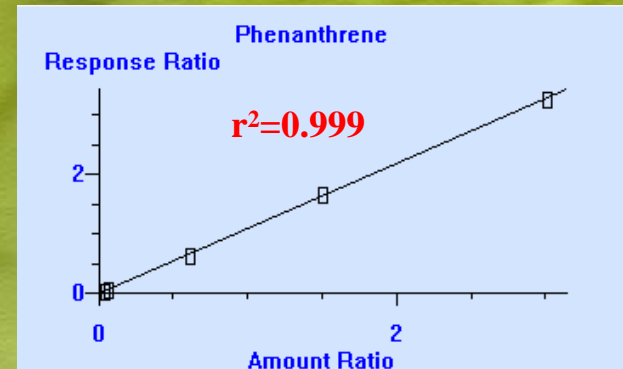
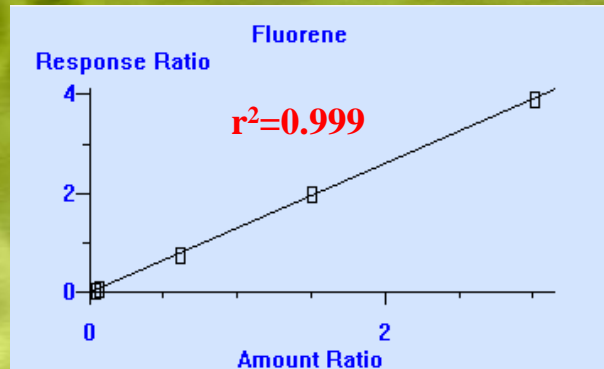
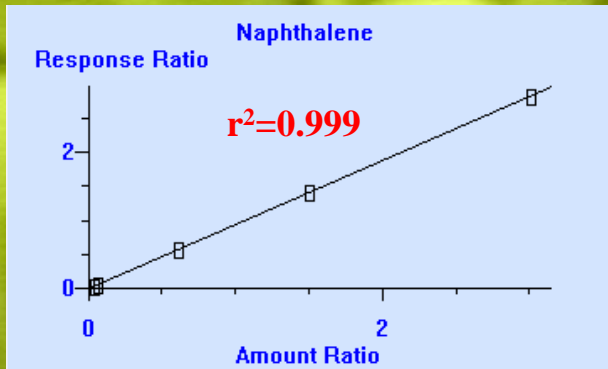


- | | | | | |
|----------------------------|-------------------------------|--------------------------|----------------------------|----------------------------|
| 1. Naphthalene | 7. Acenaphthene | 13. Fluoranthene | 19. Benzo[e]pyrene | S1. Naphthalene- d_8 |
| 2. 2-Methylnaphthalene | 8. 2,3,5-Trimethylnaphthalene | 14. Pyrene | 20. Benzo[a]pyrene | S2. Acenaphthene- d_{10} |
| 3. 1-Methylnaphthalene | 9. Fluorene | 15. Benz[a]anthracene | 21. Perylene | S3. Phenanthrene- d_{10} |
| 4. Biphenyl | 10. Phenanthrene | 16. Chrysene | 22. Indeno(1,2,3-cd)pyrene | S4. Chrysene- d_{12} |
| 5. 2,6-Dimethylnaphthalene | 11. Anthracene | 17. Benzo[b]fluoranthene | 23. Dibenz[a,h]anthracene | S5. Perylene- d_{12} |
| 6. Acenaphthylene | 12. 1-Methylphenanthrene | 18. Benzo[k]fluoranthene | 24. Benzo[ghi]perylene | * Terphenyl- d_{14} |

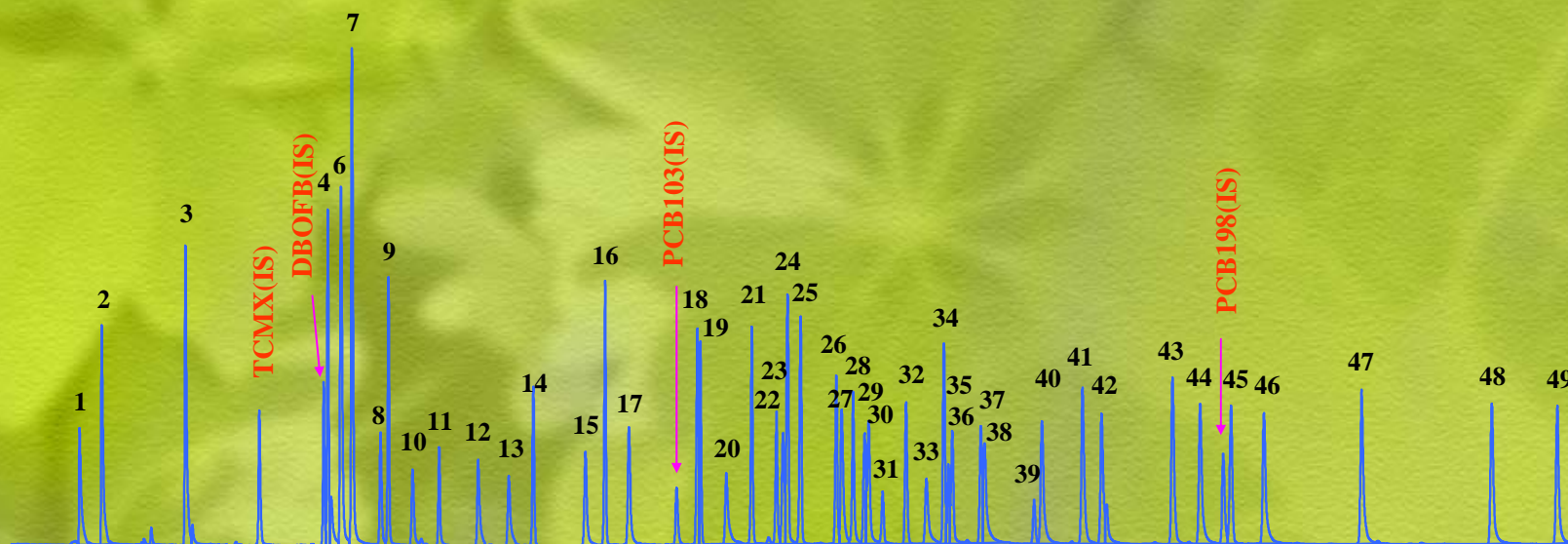
Chromatogram of PAH Calibration standard



PAHs Calibration Curve



Calibration standard I : OCPs and PCBs



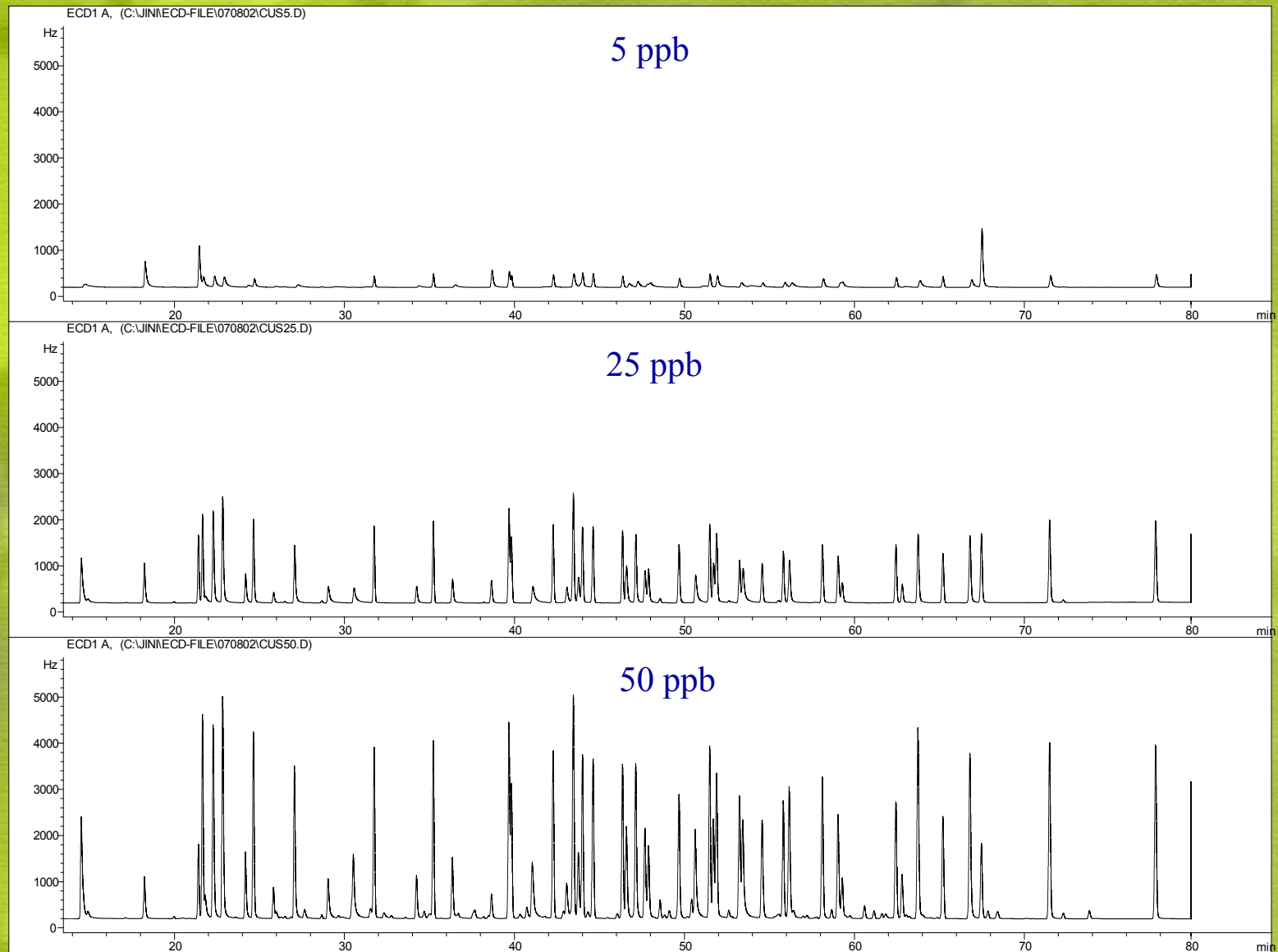
1. 1,2,4,5-Tetrachlorobenzene
2. 1,2,3,4-Tetrachlorobenzene
3. Pentachlorobenzene
4. α -HCH
5. PCB8
6. Hexachlorobenzene
7. Pentachloroanisole
8. β -HCH
9. γ -HCH
10. PCB18
11. δ -HCH
12. PCB29
13. PCB28
14. Heptachlor
15. PCB52

16. Aldrin
17. PCB44
18. Heptachlor epoxide
19. Oxychlordane
20. PCB66
21. γ -Chlordane
22. *o,p'*-DDE
23. PCB101
24. α -Chlordane
25. *trans*-Nonachlor
26. Dieldrin
27. PCB87
28. *p,p'*-DDE
29. PCB110
30. *o,p'*-DDD

31. Endrin
32. Endosulfan II
33. PCB118
34. *cis*-Nonachlor
35. *p,p'*-DDD
36. *o,p'*-DDT
37. PCB153
38. PCB105
39. *p,p'*-DDT
40. PCB138
41. PCB187
42. PCB128
43. PCB200
44. PCB180
45. Mirex

46. PCB170
47. PCB195
48. PCB206
49. PCB209

Chromatogram of PCBs and OCPs Calibration standard in this study

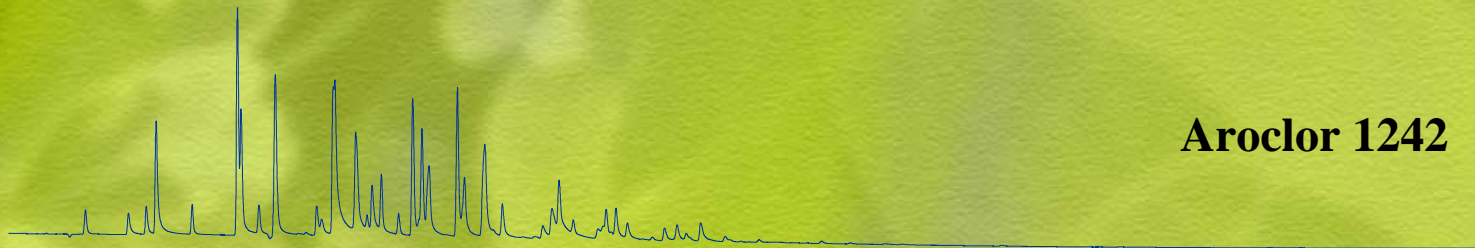


Calibration standard II : PCB mixture

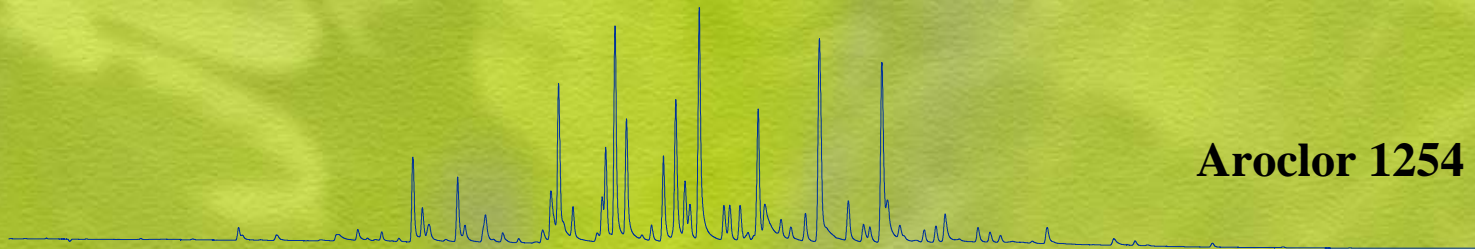
Aroclor 1016 : 1242 : 1254 : 1260 = 1 : 1 : 1 : 1



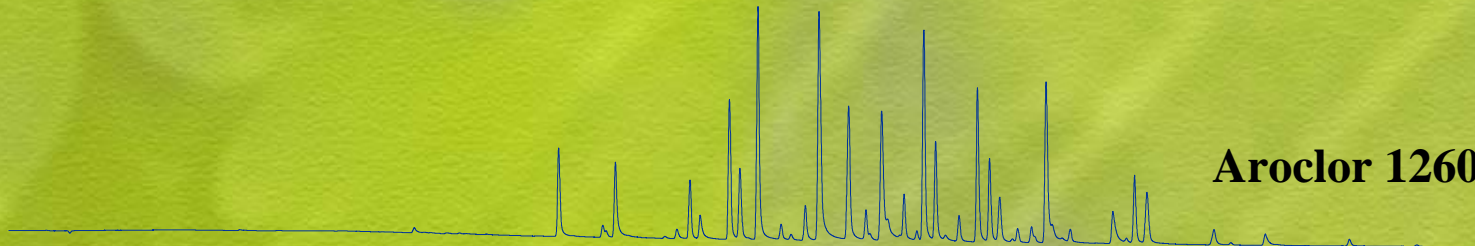
Aroclor 1016



Aroclor 1242

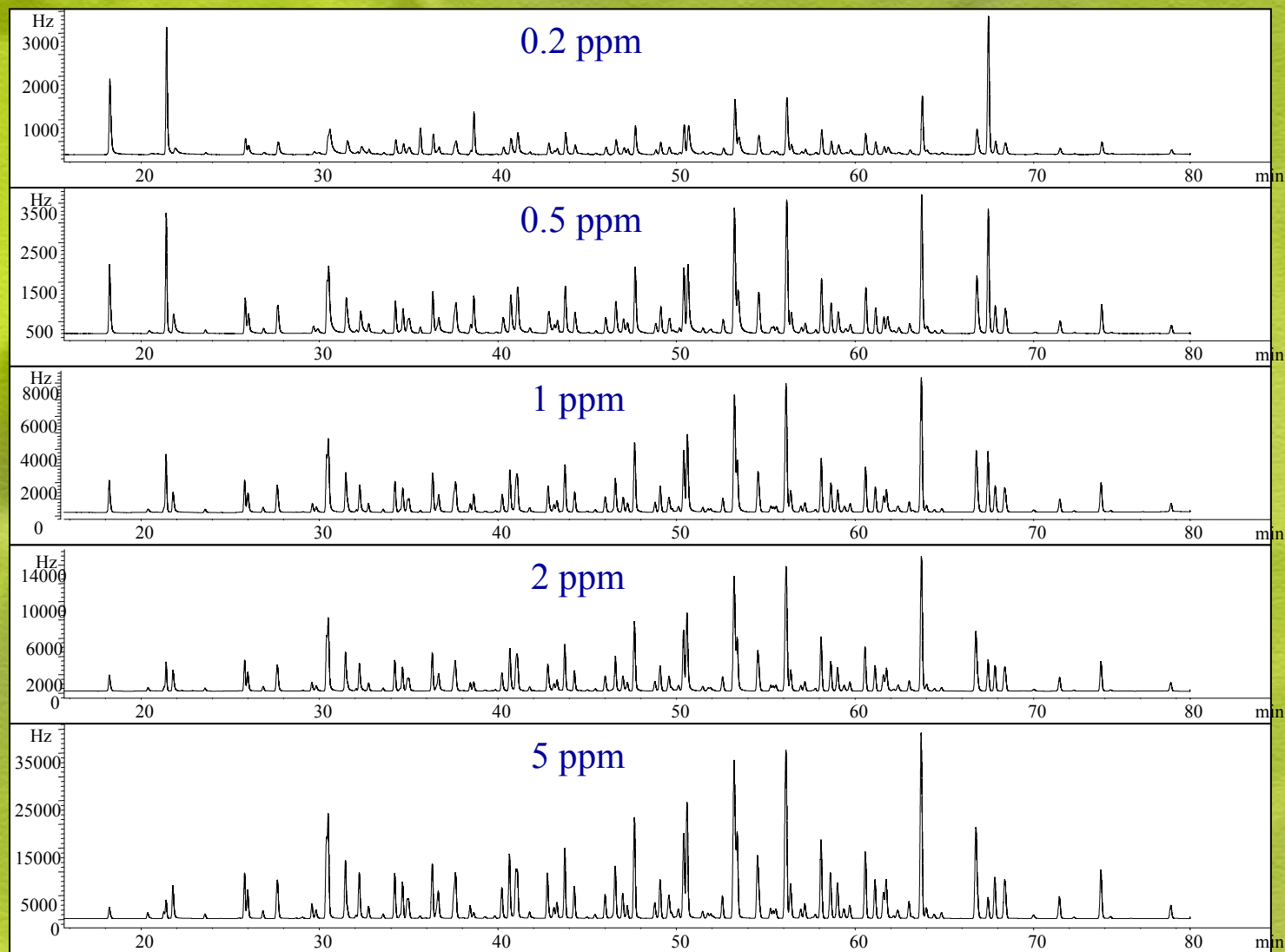


Aroclor 1254



Aroclor 1260

Chromatogram of PCBs Calibration standard in this study



CHs Calibration Curve

$$Y=A \times X^B$$

	A	B	R2
Pentachlorobenzene	0.54	0.92	1.00
a-HCH	0.39	0.95	1.00
PCB8	2.33	1.04	1.00
HCB	0.32	1.01	1.00
Pentachloroanisole	0.29	0.98	1.00
b-HCH	1.02	0.90	1.00
r-HCH(Lindane)	0.45	0.88	1.00
PCB18	2.13	0.78	1.00
d-HCH	0.62	0.77	1.00
PCB29	1.31	0.90	1.00
PCB28	1.21	0.56	1.00
Heptachlor	0.46	0.92	1.00
PCB52	1.46	0.90	1.00
Aldrin	0.40	0.97	1.00
PCB44	1.06	0.93	1.00
Heptachlor epoxide	0.33	0.99	1.00
Oxychlorane	0.44	1.04	1.00
PCB66	1.04	0.68	1.00
r-chlordane	0.41	0.97	1.00
DDMU	1.46	0.86	1.00
o,p'-DDE	0.59	0.91	1.00
Endosulfan I	0.45	1.11	1.00
PCB101	1.09	0.75	1.00
a-chlordane	0.33	1.10	1.00
trans-nonachlor	0.40	0.99	1.00
Dieldrin	0.43	0.96	1.00
PCB87	0.67	0.92	1.00
p,p'-DDE	0.46	0.88	1.00
PCB110	0.89	0.74	1.00
o,p'-DDD	0.72	1.05	1.00
Endrin	5.20	0.97	1.00
Endosulfan II	0.48	0.97	1.00
PCB118	0.87	0.66	1.00
cis-Nonachlor	0.49	0.82	1.00
p,p'-DDD	0.62	1.02	1.00
o,p'-DDT	0.30	1.17	1.00
PCB153	0.61	1.00	1.00
PCB105	0.74	0.69	1.00
p,p'-DDT	0.61	0.89	1.00
PCB138	0.55	0.97	1.00
PCB187	0.44	0.98	1.00
PCB128	0.68	0.80	0.99
PCB200	0.45	1.05	1.00
PCB180	0.40	0.87	1.00
Mirex	0.48	1.08	1.00
PCB170	0.39	0.92	1.00
PCB195	0.32	0.99	1.00
PCB206	0.31	1.03	1.00
PCB209	0.35	1.09	1.00

	A	B	R2
PCB 7/9	3.15	1.01	1.00
PCB 6	5.46	0.97	1.00
PCB 8/5	3.95	1.05	1.00
PCB 19	3.04	1.14	1.00
PCB 18	2.50	1.11	1.00
PCB 17/15	2.69	1.11	1.00
PCB 24/27	1.34	1.05	1.00
PCB 16/32	0.98	1.11	1.00
PCB 29	1.35	0.99	1.00
PCB 26	1.86	1.00	1.00
PCB 25	1.55	1.07	1.00
PCB 31	1.60	0.92	1.00
PCB 28	0.79	1.12	1.00
PCB 20/33/53	1.44	1.04	1.00
PCB 51	1.91	1.14	1.00
PCB 22	1.37	0.98	1.00
PCB 45	1.71	1.18	1.00
PCB 46	2.37	1.13	1.00
PCB 69	7.17	1.03	1.00
PCB 52	2.22	1.10	1.00
PCB 49	1.84	1.08	1.00
PCB 47/48/75	0.92	1.10	1.00
PCB 35	2.31	0.84	1.00
PCB 44	1.03	1.10	1.00
PCB 37/59/42	0.59	0.99	1.00
PCB 41/64	0.86	1.06	1.00
PCB 96	1.09	1.11	0.99
PCB 40	1.26	1.00	1.00
PCB 100/67	3.63	1.02	1.00
PCB 63	1.38	1.08	1.00
PCB 74	0.91	1.04	1.00
PCB 70	0.88	1.05	1.00
PCB 66	0.67	1.02	1.00
PCB95	1.53	1.14	1.00
PCB 91	1.37	1.17	1.00
PCB 60/56	0.70	0.99	1.00
PCB 92	1.63	1.12	1.00
PCB 84	1.27	1.15	1.00
PCB 101/90	1.50	1.08	1.00
PCB 99	1.02	1.05	1.00
PCB 119	0.76	0.98	0.99
PCB 83	1.28	1.16	1.00
PCB 97	0.99	1.10	1.00
PCB 87/115/	0.71	1.04	1.00
PCB 85	0.67	1.03	1.00
PCB 136	1.78	1.17	1.00
PCB 110/77	0.63	1.04	1.00
PCB 82	0.67	1.02	1.00
PCB 151	0.83	1.07	1.00

	A	B	R2
PCB 135	1.14	1.05	1.00
PCB 107	0.61	0.92	1.00
PCB 149/123	0.81	1.07	1.00
PCB 118	0.44	1.04	1.00
PCB 134	1.04	1.05	1.00
PCB 131/122/114	0.68	1.03	1.00
PCB 146	0.73	1.04	1.00
PCB 153	0.68	1.07	1.00
PCB 105	0.38	1.04	1.00
PCB 141/179	0.50	1.03	1.00
PCB 137	0.25	0.97	1.00
PCB 176/130	2.04	1.15	1.00
PCB 160/138	0.30	1.06	1.00
PCB 158	0.42	1.08	1.00
PCB 129	1.04	1.01	1.00
PCB 178	1.40	1.09	1.00
PCB 175	0.61	1.22	1.00
PCB 187	0.35	1.04	1.00
PCB 183	0.29	1.02	1.00
PCB 128	0.60	1.04	1.00
PCB 167	0.32	0.96	1.00
PCB 185	0.66	1.07	1.00
PCB 174	0.40	1.05	1.00
PCB 177	0.07	1.05	1.00
PCB 202/171/156	1.62	1.05	1.00
PCB173	0.09	0.95	1.00
PCB 157/201/200	1.17	1.05	1.00
PCB 172	0.35	1.01	1.00
PCB 197	0.63	1.08	0.99
PCB 180	0.24	1.03	1.00
PCB 193	0.38	1.20	1.00
PCB 191	0.44	1.11	1.00
PCB 170/190	0.32	1.02	1.00
PCB 199	0.21	1.05	1.00
PCB 203/196	0.23	1.02	1.00
PCB 189	0.21	0.96	1.00
PCB 208/195	0.26	1.02	1.00
PCB 207	0.27	1.04	1.00
PCB 194	0.19	1.00	1.00
PCB 205	0.35	1.00	1.00
PCB 206	0.35	1.02	1.00
PCB 209	0.40	1.06	1.00

Gas chromatographic conditions: PAHs

Apparatus type :Gas Chromatography

Injector type : Split/splitless

Temperature :300 °C

Volume injected :2... μ L

Split ratio :100:1

Splitter closing time :1 min

Carrier gas : He

Flow :1.0 cm³ min⁻¹

Column type :DB-5.MS

Stationary phase:5% hexyl 95% polysiloxane

Length:30 m

Internal diameter:0.25 mm

Film thickness :0.25 μ m

Detector type : Mass spectrometer

Temperature : 280°C

Acquisition Delay 10.0 min

Selected Ion Monitoring 1 cycle/sec

Oven temperature program:

initial temp.:60 °C for 2 min

1st rate: 6 °C.min⁻¹ to 300 °C

isothermal:300 °C for 13 min

Recorder/Integrator type :Agilent Chemstation



Gas chromatographic conditions: PCBs and OCPs

Apparatus type : Gas Chromatography

Injector type : Split/splitless

Temperature : 275 °C

Volume injected : 2 µL

Split ratio : 100:1

Splitter closing time : 0.5 min

Carrier gas : He

Flow : 1.2 cm³ min⁻¹

Column type : DB-5

Stationary phase: 5% hexyl 95% polysiloxane

Length: 30 m

Internal diameter: 0.25 mm

Film thickness : 0.25 µm

Detector type : µ-Electron Capture Detector

Temperature : 300°C

Make-up gas : Argon:Methane (95:5)

make-up gas flow: 40 cm.min⁻¹

Oven temperature program:

initial temp.: 100 °C	for ... 1 min
1 st rate: 5 °C.min ⁻¹	to ... 140 °C
isothermal: 140 °C	for ... 1 min
2 nd rate: 1.5 °C.min ⁻¹	to ... 250 °C.
isothermal: 250 °C	for ... 1 min...
3 rd rate: 10 °C.min ⁻¹	to ... 300 °C
isothermal: 300 °C	for 10 min

Recorder/Integrator type : Agilent Chemstation



Selected ion monitoring programs for PAHs, PCBs and OCPs

	Retention Time	Target m/z	Confirmation m/z	Compounds
Group 1	5~ 22	136		Naphthalene-d8
		128		Naphthalene
		142	141	2-methyl naphthalene
		142	141	1-methyl naphthalene
		154	152	Biphenyl
		156	154	2,6-dimethyl naphthalene
		164	162	Acenaphthene-d10
		152	151	Acenaphthylene
		154	153,152	Acenaphthene
		170		2,3,5-trimethyl naphthalene
		166		Fluorene
Group 2	22~32	188		Phenanthrene-d10
		178	176	Phenanthrene
		178	176	Anthracene
		192	191	1-methyl phenanthrene
		202	101	Fluoranthene
		202	101	Pyrene
		244		Terphenyl-d14
Group 3	32~55	240		Chrysene-d12
		228	226	Benzo[a]anthracene
		228	226	Chrysene
		252	250	Benzo[b]fluoranthene
		252	250	Benzo[k]fluoranthene
		252	250	Benzo[e]pyrene
		252	250	Benzo[a]pyrene
		264	260	Perylene-d12
		252	250	Perylene
		276	138	Indeno[1,2,3-cd]pyrene
		278	139	Dinbenz[a,h]anthracene
		276	138	Benzo[ghi]perylene

	Retention Time	Target m/z	Confirmation m/z		Compounds
Group 1	5 ~ 18	207	244		TCMX
		456	296		DBOFB
		222	224	152	CB 8
Group 2	18~ 31	256	258	221	CB18
		256	258	186	CB29
		256	258	186	CB 28
		292	290	220	CB 52
		292	290	220	CB 44
		326	324	254	CB 103
Group 3	31 ~ 53.5	292	290	220	CB 66
		326	324	254	CB 101
		326	324	254	CB 87
		326	324	254	CB 110
		326	324	254	CB 118
		360	362	290	CB 153
		326	324	254	CB 105
		360	362	290	CB 138
Group 4	53.5 ~ 80	394	396	324	CB 187
		360	362	290	CB 128
		430	428	358	CB 200
		394	396	324	CB 180
		394	396	324	CB 170
		430	428	358	CB 198
		430	428	358	CB 195
		464	466	392	CB 206
		498	500	428	CB 209

	Retention Time	Target m/z	Confirmation m/z		Compounds
Group 1	5~ 15	216	214	220	1,2,4,5-TCBZ
		216	214	220	1,2,3,4-TCBZ
		250	252	215	Pentachlorobenzene
Group 2	15 ~ 31	207	244		TCMX
		456	296		DBOFB
		219	217	181	α-HCH
		284	286	249	HCB
		280	265	237	Pentachloroanisole
		219	217	181	β-HCH
		219	217	181	γ-HCH
		219	217	181	δ-HCH
		272	374	274	Heptachlor
		263	265	66	Aldrin
Group 3	31 ~ 33	115	387	237	Oxychlorane
		353	355	81	Heptachlor epoxide
		375	373	237	γ-chlordane
		246	248	317	o,p'-DDE
		212	282	247	DDMU
		339	241	195	endosulfan I
		375	373	237	α-chlordane
		409	407	411	trans-nonachlor
		326	324	254	PCB 103
		Group 4	33 ~ 53.5	263	79
246	248			318	p,p'-DDE
235	237			165	o,p'-DDD
263	265			81	Endrin
339	195			241	Endosulfan II
409	407			411	cis-nonachlor
235	237			165	p,p'-DDD
235	237			165	o,p'-DDT
235	237			165	p,p'-DDT
Group 5	53.5 ~ 80			272	274
		430	428	358	PCB 198

Confirmation and Calibration methods for target compounds

Method used to confirm compound's identity:

1. Check each target peak's retention time with authentic standard and its shape
2. For the compounds showing high concentration, we monitor two or three selected ion for each target compound and check the ratio of quantitation and confirmation ion.

Calculation of results:

$$Y = A \times X^B$$

$$\text{Relative Concentration} = A * \text{Relative Area}^B$$

$$(\text{Analyte Amount} / \text{Volumn}) / (\text{Surrogate STD Amount} / \text{Volumn}) = A * \text{Relative Area}^B$$

$$\text{Analyte Amount} = A * \text{Relative Area}^B * \text{Surrogate STD Amount}$$

$$(\text{Analyte Amount} / \text{Sample Amount}) = A * \text{Relative Area}^B * (\text{Surrogate STD Amount} / \text{Sample Amount})$$

$$[\text{Analyte}] = A * \text{Relative Area}^B * (\text{Surrogate STD Amount} / \text{Sample Amount})$$

$$[\text{Analyte}] = A * \text{Relative Area}^B * (\text{Surrogate STD Amount} / \text{Sample Amount}) * (100 / \text{Dry Wt.})$$

$$[\text{Analyte}] = \text{ng g}^{-1} \text{ dry Wt.}$$

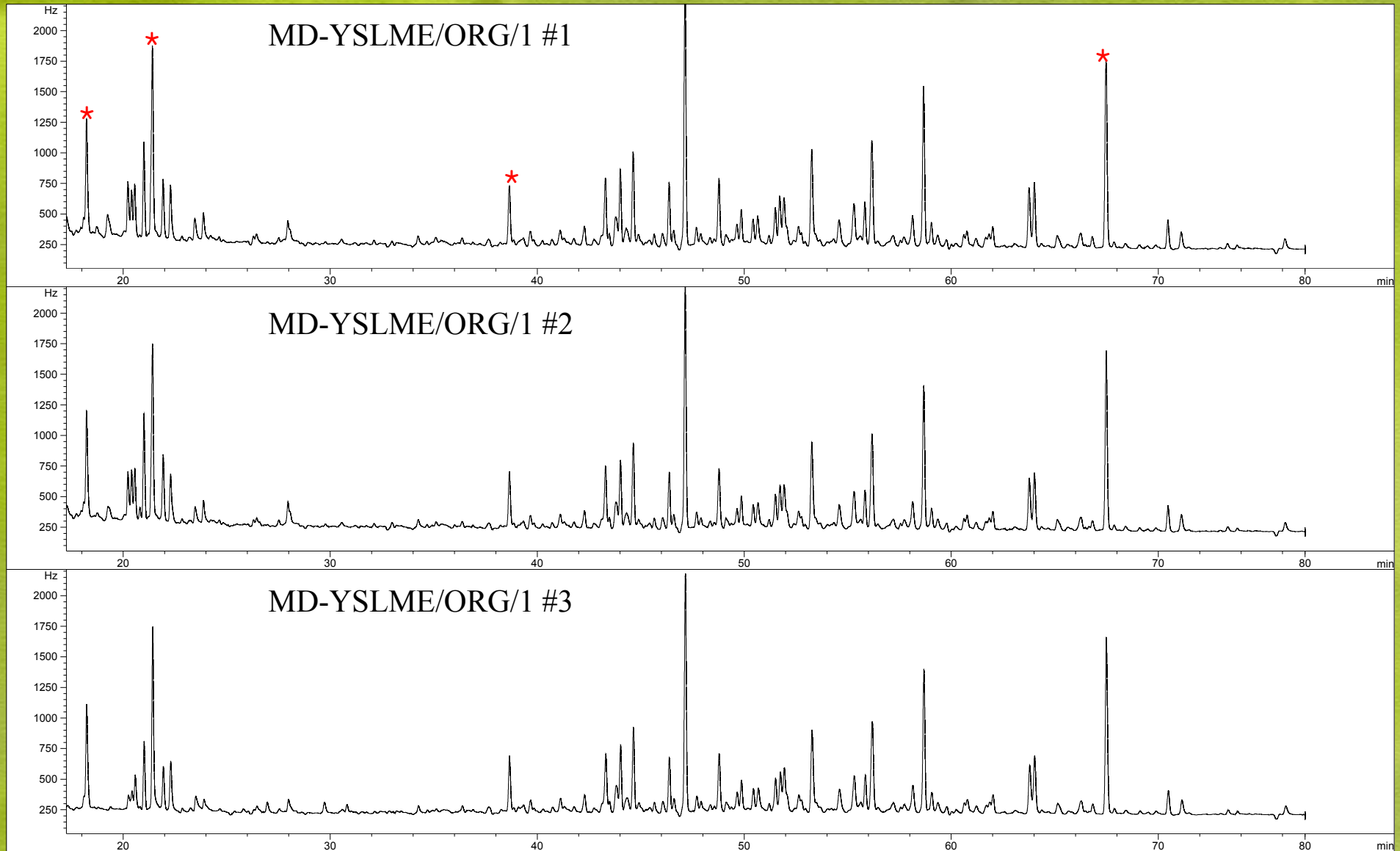
$$A = e^{(\sum \ln \text{Relative Conc.} - B * \sum \ln \text{Relative Area}) / n}$$

$$B = [n * (\sum \ln \text{Relative Area} * \ln \text{Relative Conc.}) - \sum \ln \text{Relative Area} * \sum \ln \text{Relative Conc}] / [n * \sum \ln \text{Relative Area}^2 - (\sum \ln \text{Relative Area})^2]$$

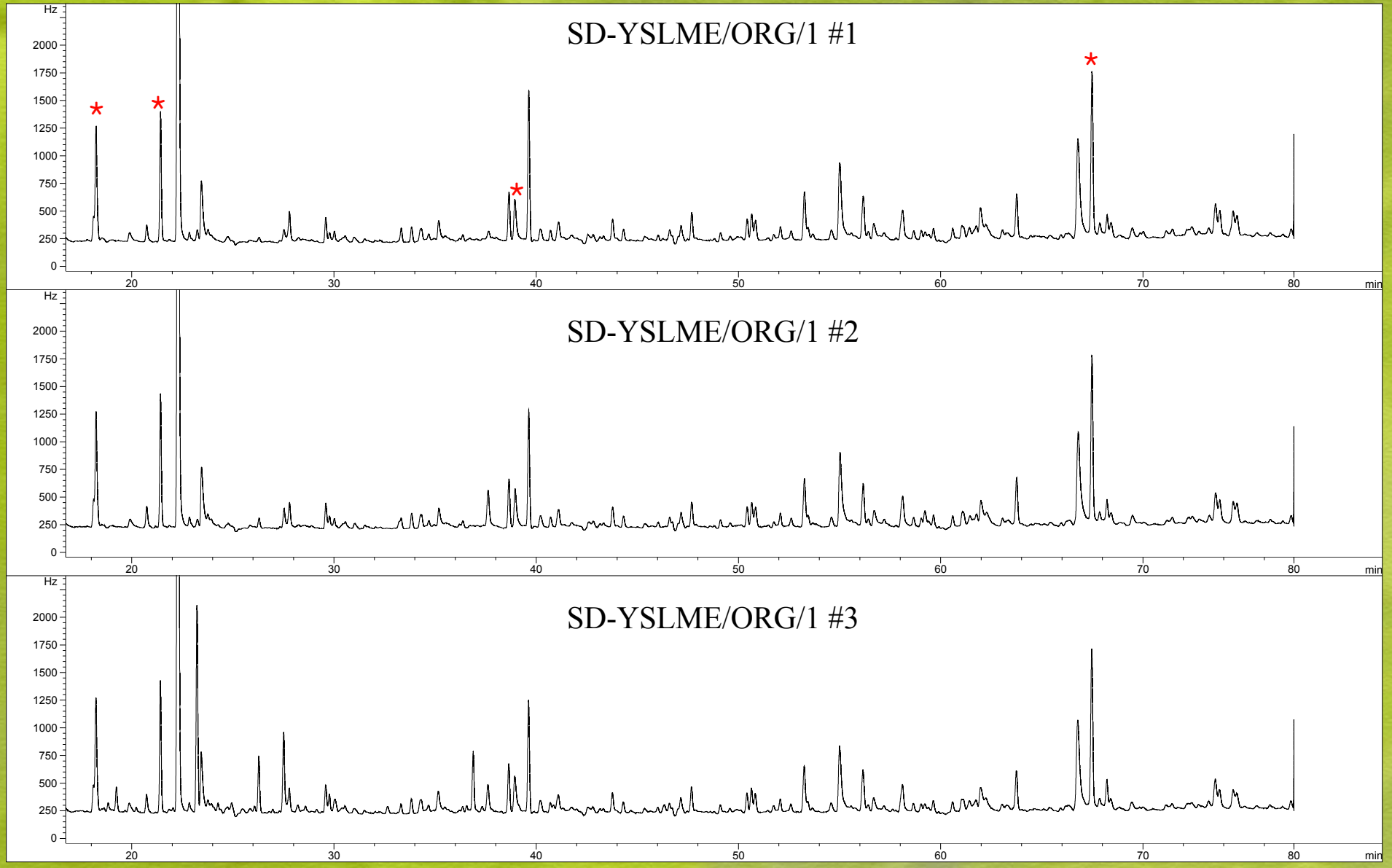
$$R^2 = [B^2 * \{\sum \ln \text{Relative Area}^2 - [(\sum \ln \text{Relative Area})^2 / n]\}] / [\sum \ln \text{Relative Conc.}^2 - \{(\sum \ln \text{Relative Conc.})^2 / n\}]$$

Chromatogram of test samples

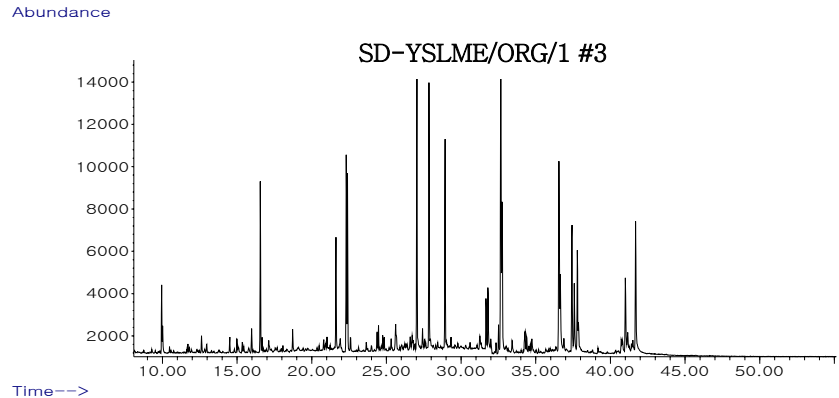
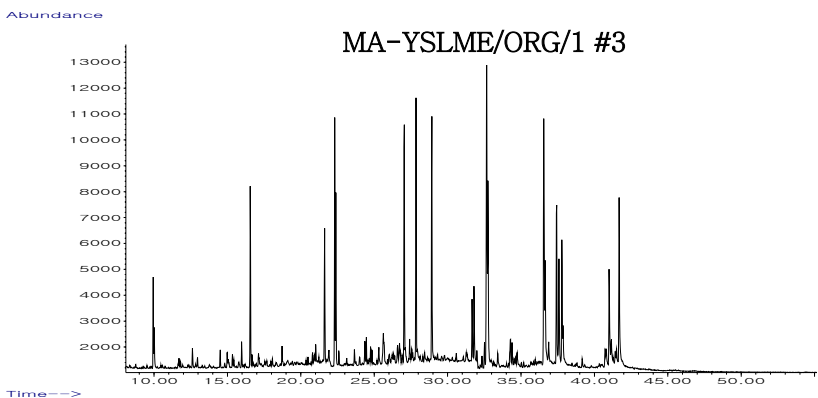
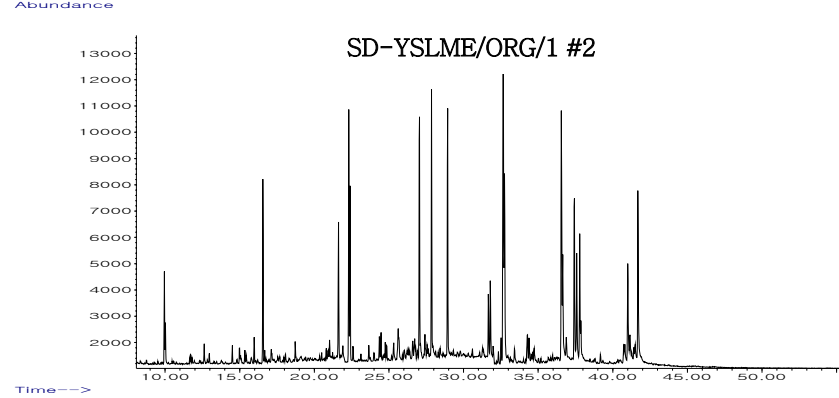
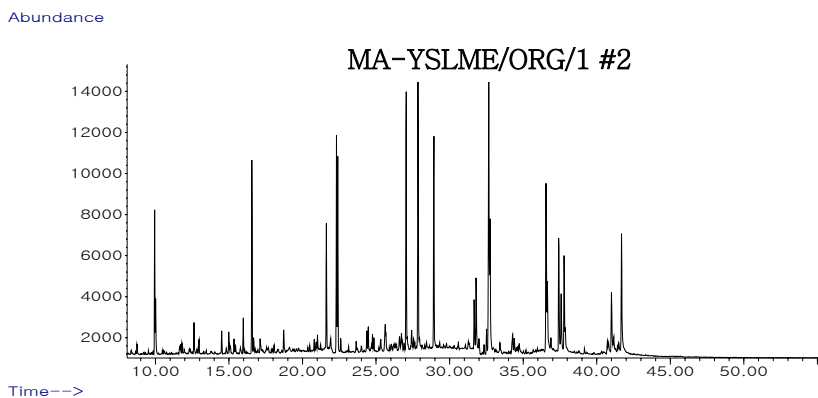
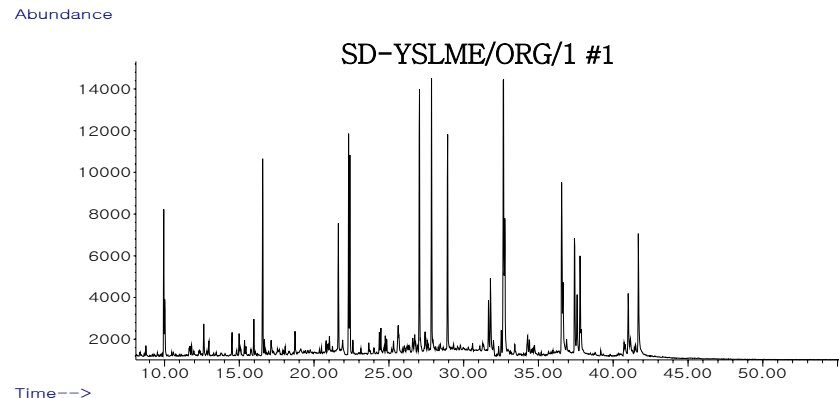
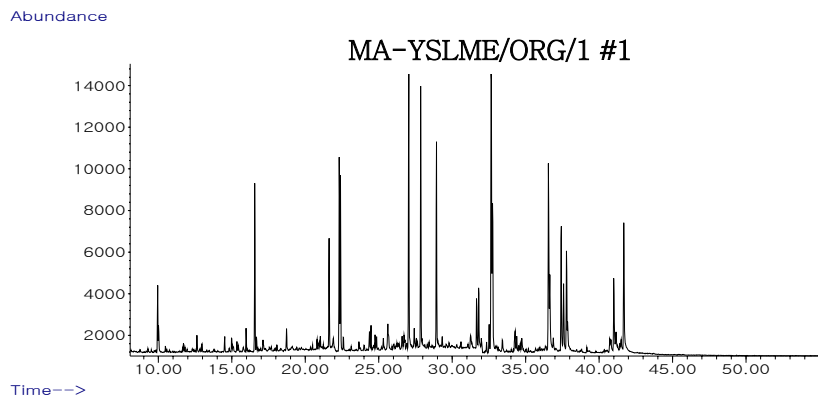
Chromatogram of mussel test sample for OCPs and PCBs



Chromatogram of sediment test sample for OCPs and PCBs



Chromatogram of test samples for PAHs



Quality Control Procedure in this study

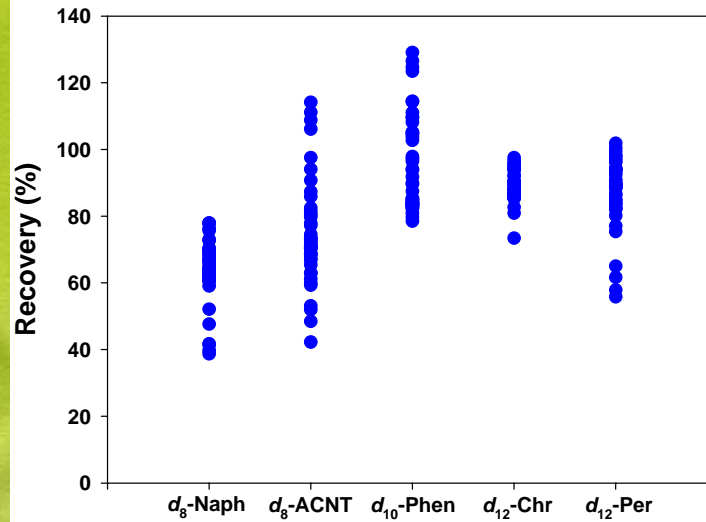
- 1. Procedural Blank Analysis**
- 2. Duplicate Sample Analysis**
- 3. Spike Sample Analysis**
- 4. Certified Reference Material Analysis**

Recovery of internal standards

PAHs

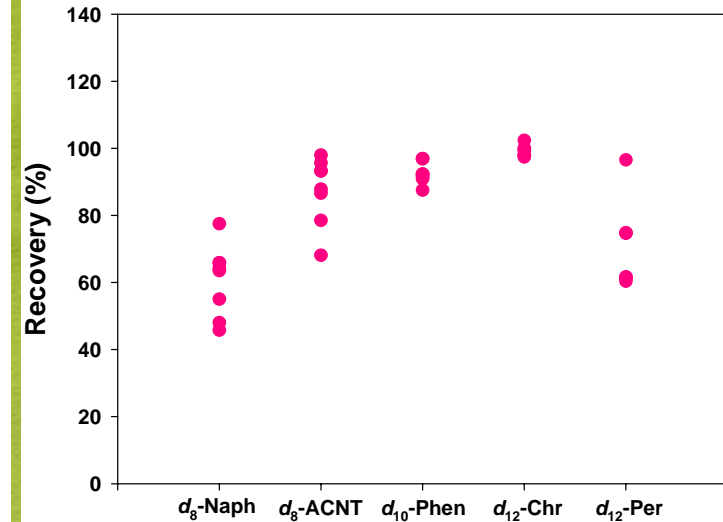
Previous study

Surrogate recovery (%) (n=106)



This study

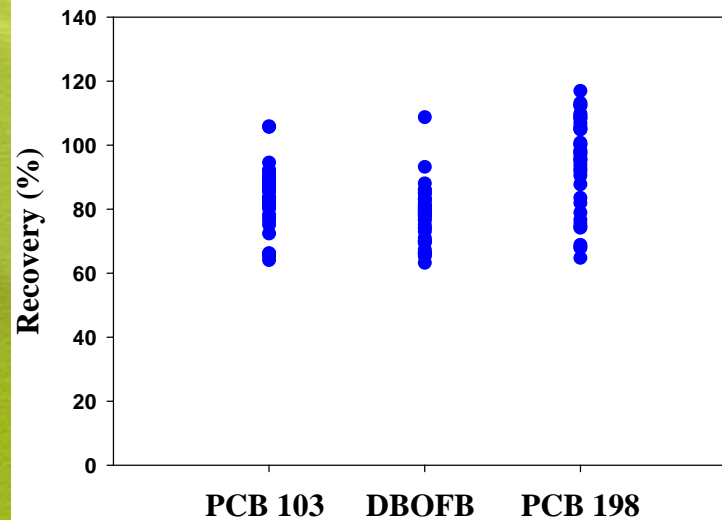
Surrogate recovery (%)



PCBs
&OCPs

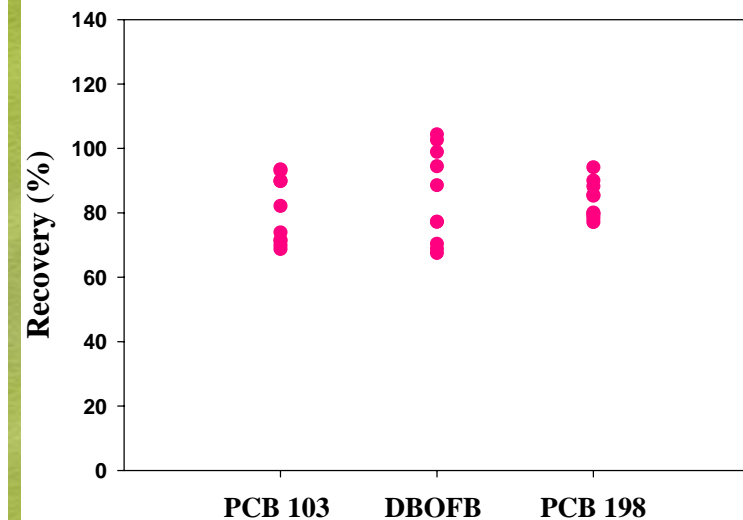
Previous study

Surrogate recovery (%) (n=41)



This study

Surrogate recovery (%)



Recovery of target compounds

Recovery of target compounds in spiked samples in this study

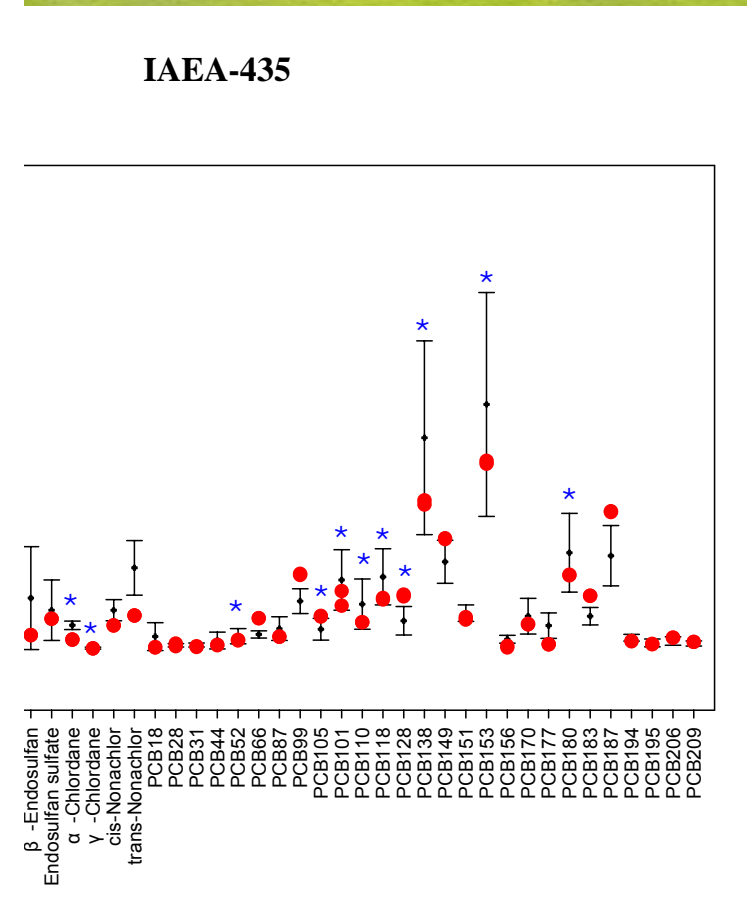
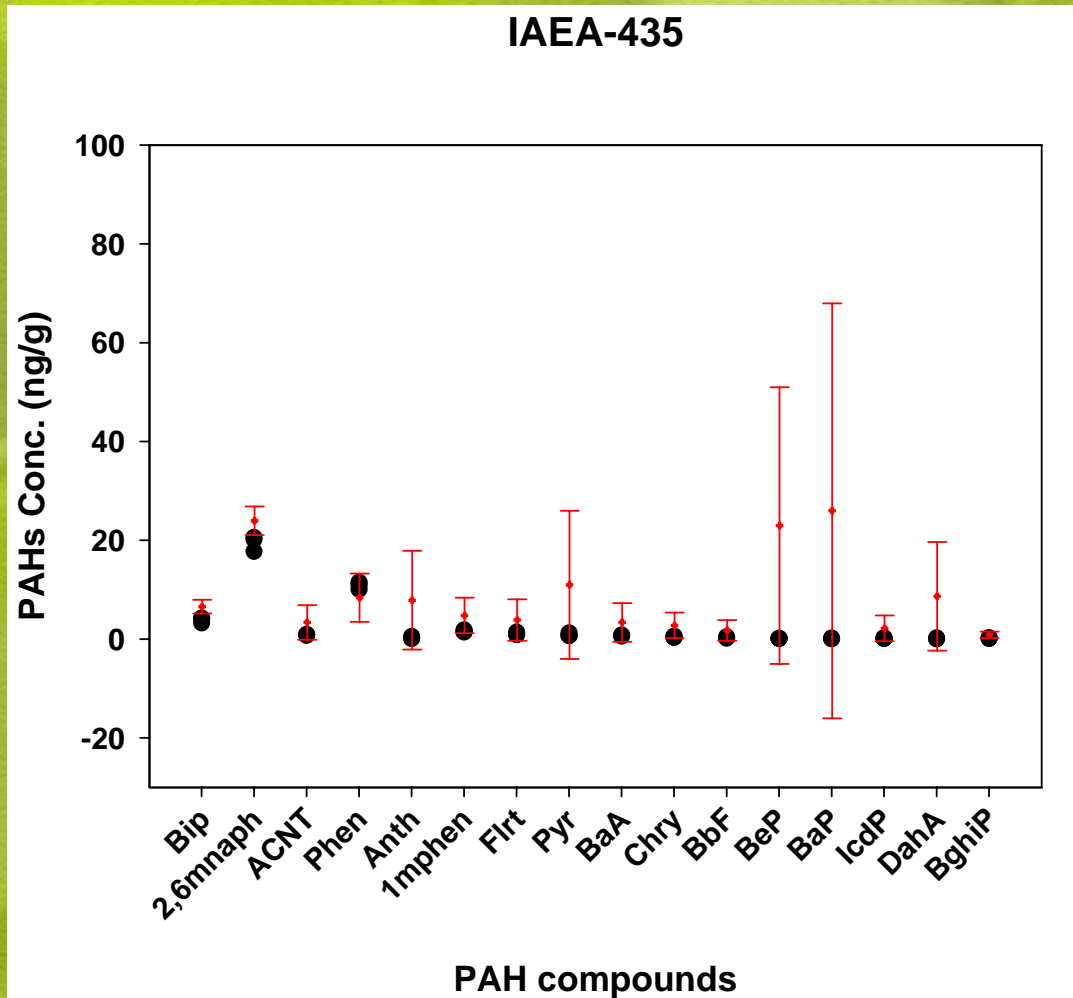
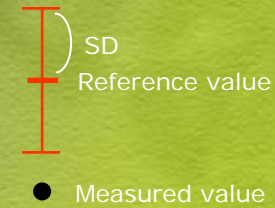
Compounds	Average Recovery (% _{n=2})
Naphthalene	83±1.2
Phenanthrene	81± 0.9
Fluoranthene	90± 2.2
Pyrene	87± 0.5
Benz[a]anthracene	94± 2.6
Chrysene/Triphenylene	74±1.9
Benzo[a]pyrene	77±12
Benzo[ghi]perylene	94±4.4
Acenaphthylene	77±2.5
Acenaphthene	78±0.4
Fluorene	87±3.3
Anthracene	100±6.0
Benzo[b]fluoranthene	89±1.3
Benzo[k]fluoranthene	76±3.7
Indeno[1,2,3-cd]pyrene	115±2.6
Dibenzo[a,h]anthracene	134±4.5

Compounds	Average recovery (% _{n=2})
Pentachlorobenzene	49±3
HCB	79 ± 7
Pentachloroanisole	69± 0
α-HCH	68 ±3
β-HCH	76 ±2
γ-HCH	72±1
δ-HCH	85±1
α-chlordane	95 ±2
β-chlordane	67±1
Oxychlordane	66 ±2
Heptachlor	74±3
Heptachlor epoxide	65±1
cis-nonachlor	86 ± 2
trans-nonachlor	95 ± 1
Aldrin	72±1
Dieldrin	95± 0
mirex	85±1
endosulfan II	76±0
o,p'-DDE	74± 2
p,p'-DDE	104 ±3
o,p'-DDD	75 ± 3
p,p'-DDD	109± 1
o,p'-DDT	82 ±5
p,p'-DDT	92 ± 1

Compounds	Average recovery (% _{n=2})
PCB8	71 ± 10
PCB18	65± 8
PCB28	81 ± 3
PCB29	80 ±2
PCB44	69 ±5
PCB52	82 ±7
PCB66	98 ±0
PCB87	81 ± 1
PCB101	99 ±2
PCB105	85 ±2
PCB110	85 ±2
PCB118	83 ± 0
PCB128	94 ± 1
PCB138	110 ±0
PCB153	115 ±4
PCB170	77 ±2
PCB180	89 ±2
PCB187	84 ±2
PCB195	78 ±2
PCB200	75 ± 1
PCB206	79 ±2
PCB209	79± 1

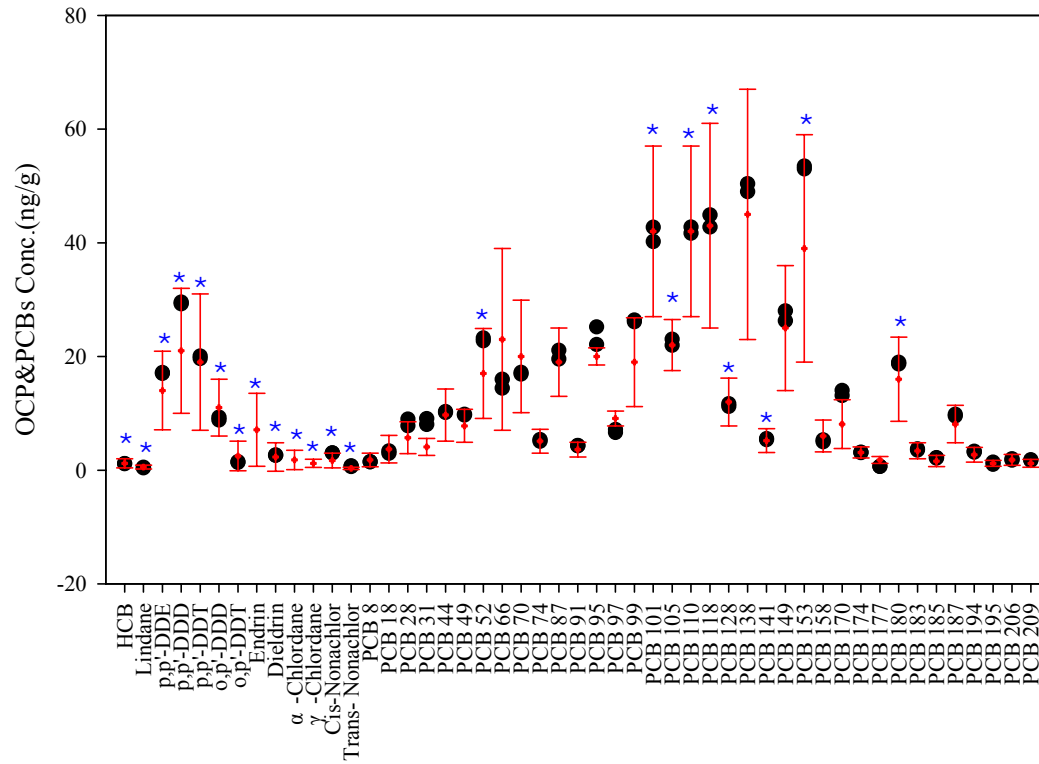
CRM Analysis Result

IAEA 435 (Tuna fish flesh homogenate)

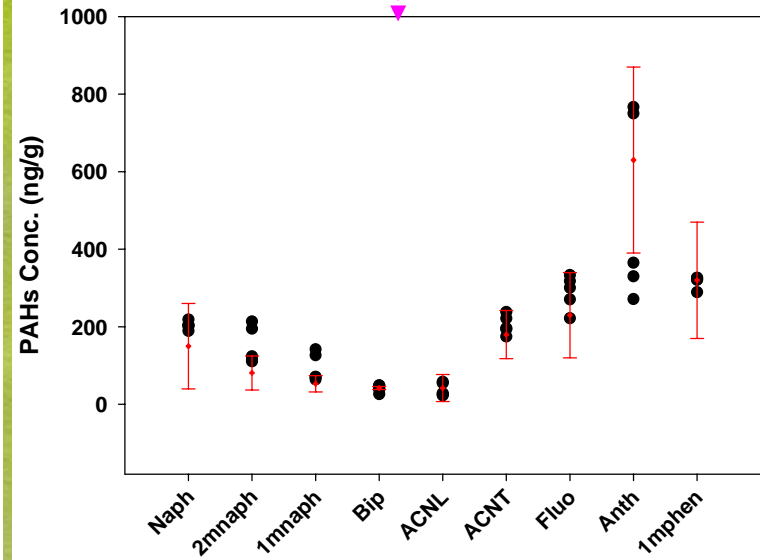
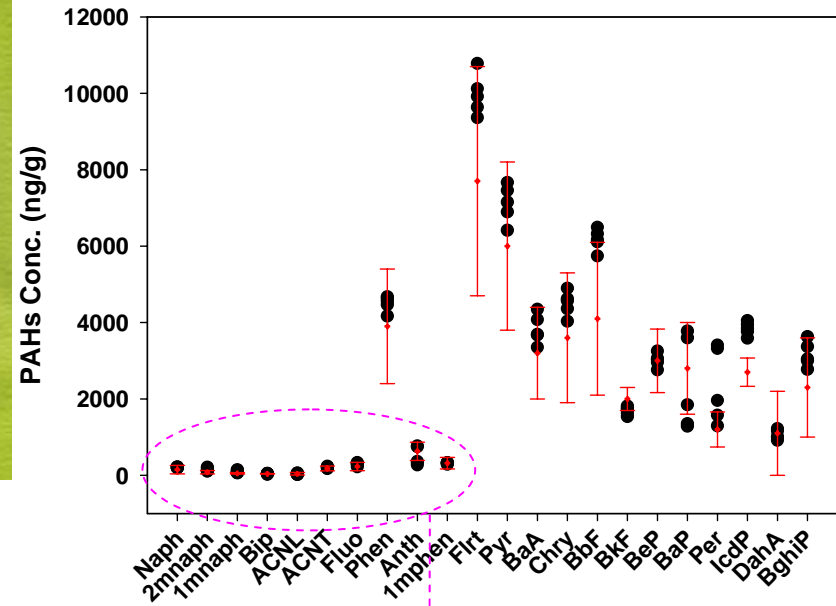


IAEA 417 (Marine sediment)

IAEA-417



IAEA-417



Results: SD-YSLME/ORG/1

compounds	Units	Concentration (dry weight)			Mean value	Standard deviation	RSD (%)
		1 st value	2 nd value	3 rd value			
<i>Polycyclic Aromatic Hydrocarbons:</i>							
Naphthalene	ng/g dw	122.3	122.6	121.5	122.1	0.57	0.5
Phenanthrene	ng/g dw	143.4	148.7	163.8	152	10.59	7.0
1-Methylphenanthrene	ng/g dw	18.6	18.7	19.4	18.9	0.44	2.3
Fluoranthene	ng/g dw	364.6	366.6	364.7	365.3	1.13	0.3
Pyrene	ng/g dw	315.7	332.2	327.4	325.1	8.49	2.6
Benz[a]anthracene	ng/g dw	99.8	97.7	105.4	101	3.98	3.9
Chrysene/Triphenylene	ng/g dw	170.7	184.6	189.9	181.7	9.92	5.5
Benzo[e]pyrene	ng/g dw	163.4	159.2	158.8	160.5	2.55	1.6
Benzo[a]pyrene	ng/g dw	71.7	87.3	87.2	82.1	8.98	10.9
Benzo[ghi]perylene	ng/g dw	214.9	227.1	229.2	223.7	7.72	3.5
<i>Polycyclic Aromatic Hydrocarbons:</i>							
2-Methylnaphthalene	ng/g dw	55.3	56.8	66.2	59.4	5.91	9.9
1-Methylnaphthalene	ng/g dw	33.2	34.7	39.7	35.9	3.4	9.5
Biphenyl	ng/g dw	23.7	20.8	24	22.8	1.77	7.8
2,6-Dimethylnaphthalene	ng/g dw	15.4	14.5	15.9	15.3	0.71	4.6
Acenaphthylene	ng/g dw	47.6	49.3	48.6	48.5	0.85	1.8
Acenaphthene	ng/g dw	13.5	14.9	15.2	14.5	0.91	6.3
2,3,5-Trimethylnaphthalene	ng/g dw	10.8	12.2	10.9	11.3	0.78	6.9
Fluorene	ng/g dw	29.7	31.5	33.9	31.7	2.11	6.7
Anthracene	ng/g dw	22.8	23.2	20.5	22.2	1.46	6.6
Benzo[b]fluoranthene	ng/g dw	280.4	284.6	276	280.3	4.3	1.5
Benzo[k]fluoranthene	ng/g dw	80.7	88	82.6	83.8	3.79	4.5
Perylene	ng/g dw	78.4	76.5	69.9	74.9	4.46	6.0
Indeno[1,2,3-cd]pyrene	ng/g dw	216.8	217.5	203.9	212.7	7.66	3.6
Dibenzo[a,h]anthracene	ng/g dw	30.5	39.8	39.4	36.6	5.26	14.4

Results: SD-YSLME/ORG/1

compounds	Units	Concentration (dry weight)			Mean value	Standard deviation	RSD (%)
		1 st value	2 nd value	3 rd value			
HCB	ng g ⁻¹ dw	68	62.9	64.6	65.16	2.57	3.9
γ HCH (lindane)	ng g ⁻¹ dw	0.37	0.4	0.37	0.38	0.02	5.3
p,p' DDE	ng g ⁻¹ dw	1.8	1.81	1.74	1.78	0.04	2.2
p,p' DDD	ng g ⁻¹ dw	0.6	0.6	0.7	0.66	0.06	9.1
p,p' DDT	ng g ⁻¹ dw	<0.3	<0.3	<0.3			
Aldrin	ng g ⁻¹ dw	-	-	-	-	-	
Dieldrin	ng g ⁻¹ dw	0.15	0.12	0.12	0.13	0.02	15.4
Endrin	ng g ⁻¹ dw	<0.5	<0.5	<0.5			
Heptachlor	ng g ⁻¹ dw	<0.2	<0.2	<0.2			
Heptachlor epoxide	ng g ⁻¹ dw	<0.2	<0.2	<0.2			
α endosulfan	ng g ⁻¹ dw	0.08	0.07	0.08	0.08	0.01	12.5
β endosulfan	ng g ⁻¹ dw	0.63	0.65	0.63	0.63	0.01	1.6
α chlordane	ng g ⁻¹ dw	0.16	0.18	0.17	0.17	0.01	5.9
γ chlordane	ng g ⁻¹ dw	<0.2	<0.2	<0.2			
Pentachlorobenzene	ng g ⁻¹ dw	4.8	4.7	4.9	4.77	0.11	2.3
cis-nonachlor	ng g ⁻¹ dw	0.51	0.52	0.51	0.52	0.01	1.9
trans-nonachlor	ng g ⁻¹ dw	0.13	0.1	0.1	0.11	0.02	18.2
INDIVIDUAL PCB CONGENERS:							
PCB 18	ng g ⁻¹ dw	2.09	2.19	2.28	2.18	0.09	4.1
PCB 22	ng g ⁻¹ dw	0.73	0.8	0.73	0.75	0.04	5.3
PCB 26	ng g ⁻¹ dw	8.88	8.6	8.76	8.75	0.14	1.6
PCB 28	ng g ⁻¹ dw	4.7	4.85	4.48	4.68	0.18	3.8
PCB 31	ng g ⁻¹ dw	1.67	1.5	1.51	1.56	0.09	5.8
PCB 44	ng g ⁻¹ dw	1.01	1.03	1.03	1.02	0.01	1.0
PCB 49	ng g ⁻¹ dw	2.24	2.06	2.18	2.16	0.09	4.2
PCB 52	ng g ⁻¹ dw	1.81	1.68	1.8	1.76	0.07	4.0
PCB 66	ng g ⁻¹ dw	1.34	1.38	1.28	1.33	0.05	3.8
PCB 70	ng g ⁻¹ dw	1.94	1.91	1.92	1.92	0.02	1.0
PCB 74	ng g ⁻¹ dw	1.57	1.45	1.46	1.49	0.07	4.7
PCB 84	ng g ⁻¹ dw	0.83	0.79	0.8	0.8	0.02	2.5
PCB 85	ng g ⁻¹ dw	0.36	0.31	0.37	0.35	0.03	8.6
PCB 87/115	ng g ⁻¹ dw	1.26	1.32	1.22	1.27	0.05	3.9
PCB 91	ng g ⁻¹ dw	1.08	1	0.92	1	0.08	8.0

compounds	Units	Concentration (dry weight)			Mean value	Standard deviation	RSD (%)
		1 st value	2 nd value	3 rd value			
INDIVIDUAL PCB CONGENERS:							
PCB 92	ng g ⁻¹ dw	1.2	1.08	1.17	1.15	0.07	6.1
PCB 97	ng g ⁻¹ dw	1.17	1.09	1.1	1.12	0.04	3.6
PCB 99	ng g ⁻¹ dw	2.21	2.13	2.15	2.16	0.04	1.9
PCB 101/90	ng g ⁻¹ dw	5.29	5.18	5.29	5.25	0.06	1.1
PCB 105	ng g ⁻¹ dw	2.65	2.7	2.68	2.68	0.02	0.7
PCB 107	ng g ⁻¹ dw	0.44	0.33	0.39	0.39	0.05	12.8
PCB 110/77	ng g ⁻¹ dw	5.09	5.13	5.17	5.13	0.04	0.8
PCB 118	ng g ⁻¹ dw	2.31	2.38	2.34	2.34	0.03	1.3
PCB 119	ng g ⁻¹ dw	0.16	0.17	0.16	0.16	0.01	6.3
PCB 128	ng g ⁻¹ dw	0.76	0.82	0.79	0.79	0.03	3.8
PCB 134	ng g ⁻¹ dw	0.38	0.45	0.42	0.42	0.03	7.1
PCB 135	ng g ⁻¹ dw	1.16	1.03	1.03	1.07	0.08	7.5
PCB 131/122/114	ng g ⁻¹ dw	2.8	3.16	3.11	3.02	0.19	6.3
PCB 160/138	ng g ⁻¹ dw	4.81	4.97	4.75	4.84	0.11	2.3
PCB 141/179	ng g ⁻¹ dw	1.1	1.08	1.11	1.1	0.01	0.9
PCB 146	ng g ⁻¹ dw	1.3	1.3	1.34	1.31	0.02	1.5
PCB 149/123	ng g ⁻¹ dw	3.04	3.13	3.16	3.11	0.06	1.9
PCB 151	ng g ⁻¹ dw	1.51	1.55	1.5	1.52	0.03	2.0
PCB 153	ng g ⁻¹ dw	5.76	5.79	5.84	5.8	0.04	0.7
PCB 158	ng g ⁻¹ dw	0.75	0.71	0.78	0.75	0.04	5.3
PCB 167	ng g ⁻¹ dw	0.4	0.45	0.38	0.41	0.04	9.8
PCB170	ng g ⁻¹ dw	2.3	2.6	2.4	2.43	0.15	6.2
PCB 172	ng g ⁻¹ dw	0.69	0.69	0.68	0.69	0.01	1.4
PCB 174	ng g ⁻¹ dw	0.79	0.83	0.84	0.82	0.03	3.7
PCB 176/130	ng g ⁻¹ dw	1.3	1.13	1.36	1.26	0.12	9.5
PCB 178	ng g ⁻¹ dw	1.23	1.05	1.25	1.17	0.11	9.4
PCB 180	ng g ⁻¹ dw	3.48	3.3	3.61	3.46	0.16	4.6
PCB 183	ng g ⁻¹ dw	0.5	0.58	0.56	0.54	0.04	7.4
PCB 187	ng g ⁻¹ dw	2.53	2.32	2.57	2.47	0.13	5.3
PCB 189	ng g ⁻¹ dw	0.21	0.22	0.23	0.22	0.01	4.5
PCB 191	ng g ⁻¹ dw	0.13	0.12	0.12	0.12	0.01	8.3
PCB 194	ng g ⁻¹ dw	0.99	0.91	1.04	0.98	0.07	7.1
PCB 203/196	ng g ⁻¹ dw	0.6	0.59	0.63	0.61	0.02	3.3
PCB 206	ng g ⁻¹ dw	0.48	0.48	0.47	0.48	0.01	2.1

Results: MA-YSLME/ORG/1

compounds	Units	Concentration (dry weight)			Mean value	Standard deviation	RSD (%)
		1 st value	2 nd value	3 rd value			
<i>Polycyclic Aromatic Hydrocarbons:</i>							
Naphthalene	ng/g dw	25.21	23.89	23.64	24.25	0.84	3.5
Phenanthrene	ng/g dw	7.32	7.1	7.58	7.33	0.24	3.3
1-Methylphenanthrene	ng/g dw	0.82	0.76	0.76	0.78	0.03	3.8
Fluoranthene	ng/g dw	2.08	1.98	2.11	2.06	0.07	3.4
Pyrene	ng/g dw	1.22	1.26	1.26	1.25	0.02	1.6
Benz[a]anthracene	ng/g dw	0.79	0.76	0.71	0.75	0.04	5.3
Chrysene/Triphenylene	ng/g dw	0.96	0.94	0.95	0.95	0.01	1.1
Benzo[e]pyrene	ng/g dw	0.23	0.25	0.24	0.24	0.01	4.2
Benzo[a]pyrene	ng/g dw	0.09	0.12	0.09	0.1	0.02	20.0
Benzo[ghi]perylene	ng/g dw	0.2	0.18	0.2	0.19	0.01	5.3
<i>Alkylated Polycyclic Aromatic Hydrocarbons:</i>							
2-Methylnaphthalene	ng/g dw	25.21	23.89	23.64	24.25	0.84	3.5
1-Methylnaphthalene	ng/g dw	10.91	10.78	10.26	10.65	0.34	3.2
Biphenyl	ng/g dw	3.17	3	2.98	3.05	0.1	3.3
2,6-Dimethylnaphthalene	ng/g dw	5.23	5.23	5.31	5.26	0.05	1.0
Acenaphthylene	ng/g dw	1.15	1.1	0.99	1.08	0.08	7.4
Acenaphthene	ng/g dw	0.49	0.49	0.49	0.49	0	0.0
2,3,5-Trimethylnaphthalene	ng/g dw	4.96	4.71	4.78	4.82	0.13	2.7
Fluorene	ng/g dw	1.76	1.75	1.68	1.73	0.04	2.3
Anthracene	ng/g dw	0.61	0.43	0.43	0.49	0.1	20.4
Benzo[b]fluoranthene	ng/g dw	0.49	0.49	0.5	0.49	0.01	2.0
Indeno[1,2,3-cd]pyrene	ng/g dw	0.08	0.09	0.1	0.09	0.01	11.1

Results: MA-YSLME/ORG/1

compounds	Units	Concentration (dry weight)			Mean value	Standard deviation	RSD (%)
		1 st value	2 nd value	3 rd value			
HCB	ng g ⁻¹ dw	1.82	1.61	1.76	1.73	0.1	5.8
γ HCH (lindane)	ng g ⁻¹ dw	0.16	0.17	0.21	0.18	0.02	11.1
p,p' DDE	ng g ⁻¹ dw	10.56	10.46	10.67	10.56	0.1	0.9
p,p' DDD	ng g ⁻¹ dw	3.29	3.25	3.28	3.27	0.02	0.6
p,p' DDT	ng g ⁻¹ dw	3.18	3.14	3.2	3.17	0.03	0.9
Aldrin	ng g ⁻¹ dw	-	-	-	-	-	-
Dieldrin	ng g ⁻¹ dw	2.79	2.86	2.85	2.83	0.04	1.4
Endrin	ng g ⁻¹ dw	3.31	3.39	3.44	3.38	0.07	2.1
Heptachlor	ng g ⁻¹ dw	0.1	0.11	0.1	0.11	0.01	9.1
Heptachlor epoxide	ng g ⁻¹ dw	0.6	0.64	0.59	0.61	0.02	3.3
β endosulfan	ng g ⁻¹ dw	1.06	1.12	1.06	1.08	0.03	2.8
α chlordane	ng g ⁻¹ dw	2.47	2.49	2.48	2.48	0.01	0.4
γ chlordane	ng g ⁻¹ dw	1.08	0.96	0.95	1	0.07	7.0
cis-nonachlor	ng g ⁻¹ dw	2.14	2.19	2.19	2.18	0.03	1.4
trans-nonachlor	ng g ⁻¹ dw	3.74	3.71	3.71	3.72	0.02	0.5
o,p'-DDE	ng g ⁻¹ dw	0.83	0.75	0.78	0.79	0.04	5.1
o,p'-DDD	ng g ⁻¹ dw	0.87	0.84	0.89	0.87	0.02	2.3
o,p'-DDT	ng g ⁻¹ dw	1.71	1.71	1.82	1.75	0.06	3.4
INDIVIDUAL PCB CONGENERS:							
PCB 18	ng g ⁻¹ dw	0.65	0.57	0.54	0.59	0.06	10.2
PCB 28	ng g ⁻¹ dw	1.68	1.86	1.74	1.76	0.09	5.1
PCB 41/64	ng g ⁻¹ dw	0.33	0.34	0.31	0.33	0.01	3.0
PCB 44	ng g ⁻¹ dw	0.61	0.61	0.61	1.06	0.004	0.4
PCB 52	ng g ⁻¹ dw	1.26	1.34	1.4	1.33	0.07	5.3
PCB 63	ng g ⁻¹ dw	0.66	0.76	0.75	0.72	0.06	8.3
PCB 66	ng g ⁻¹ dw	0.82	0.85	0.84	0.84	0.01	1.2
PCB 82	ng g ⁻¹ dw	4.79	4.81	4.68	4.76	0.07	1.5

compounds	Units	Concentration (dry weight)			Mean value	Standard deviation	RSD (%)
		1 st value	2 nd value	3 rd value			
PCB 84	ng g ⁻¹ dw	10.13	10.06	9.96	10.05	0.08	0.8
PCB 87/115	ng g ⁻¹ dw	1.45	1.46	1.4	1.44	0.03	2.1
PCB 91	ng g ⁻¹ dw	0.63	0.6	0.7	0.64	0.05	7.8
PCB 92	ng g ⁻¹ dw	1.49	1.25	1.35	1.36	0.12	8.8
PCB95	ng g ⁻¹ dw	2.61	2.71	2.6	2.64	0.06	2.3
PCB 101/90	ng g ⁻¹ dw	5.04	4.76	4.75	4.85	0.16	3.3
PCB 105	ng g ⁻¹ dw	1.27	1.27	1.1	1.21	0.1	8.3
PCB 110/77	ng g ⁻¹ dw	1.88	1.85	1.88	1.87	0.02	1.1
PCB 118	ng g ⁻¹ dw	2.64	2.58	2.57	2.59	0.04	1.5
PCB 119	ng g ⁻¹ dw	0.17	0.2	0.17	0.18	0.02	11.1
PCB 128	ng g ⁻¹ dw	1.18	1.18	1.25	1.2	0.04	3.3
PCB 134	ng g ⁻¹ dw	4.2	4.19	4.13	4.17	0.04	1.0
PCB 131/122/114	ng g ⁻¹ dw	9.2	9.21	8.81	9.07	0.23	2.5
PCB 137	ng g ⁻¹ dw	1.38	1.4	1.38	1.39	0.01	0.7
PCB 160/138	ng g ⁻¹ dw	7.15	7.01	6.91	7.02	0.12	1.7
PCB 141/179	ng g ⁻¹ dw	1.54	1.6	1.6	1.58	0.04	2.5
PCB 146	ng g ⁻¹ dw	1.34	1.24	1.32	1.3	0.05	3.8
PCB 149/123	ng g ⁻¹ dw	1.82	1.9	1.82	1.84	0.05	2.7
PCB 151	ng g ⁻¹ dw	0.28	0.28	0.27	0.28	0.01	3.6
PCB 153	ng g ⁻¹ dw	6.6	6.67	6.71	6.66	0.06	0.9
PCB 167	ng g ⁻¹ dw	0.53	0.54	0.53	0.53	0.003	0.6
PCB 170/190	ng g ⁻¹ dw	0.58	0.6	0.56	0.58	0.02	3.4
PCB 172	ng g ⁻¹ dw	0.16	0.14	0.14	0.15	0.01	6.7
PCB 174	ng g ⁻¹ dw	0.43	0.43	0.44	0.44	0.01	2.3
PCB 180	ng g ⁻¹ dw	2.22	2.26	2.34	2.27	0.06	2.6
PCB 183	ng g ⁻¹ dw	4.88	4.88	4.9	4.89	0.01	0.2
PCB 187	ng g ⁻¹ dw	1.65	1.65	1.65	1.65	0.004	0.2
PCB 189	ng g ⁻¹ dw	0.03	0.03	0.03	0.03	0.004	13.3
PCB 193	ng g ⁻¹ dw	2.92	2.8	2.87	2.86	0.06	2.1
PCB 199	ng g ⁻¹ dw	0.11	0.1	0.11	0.11	0.01	9.1
PCB 203/196	ng g ⁻¹ dw	0.16	0.16	0.16	0.16	0.002	1.3
PCB 209	ng g ⁻¹ dw	0.16	0.15	0.17	0.16	0.01	0.01



*Thank you for
your attention!*