

An Optimized Solid Phase Extraction Procedure for EPA Method 8081 and 8082 Analytes in Water

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INTRODUCTION

EPA methods 8081, organochlorine pesticides (OCPs) by GC, and 8082, polychlorinated biphenyls by GC (PCBs), are hazardous waste test methods (SW-846) regulated under the Resource Conservation and Recovery Act (RCRA). These methods are performance based which allow analysts to modify the,sampling and analytical approaches to meet the measurement requirements. In other words, these methods convey "what" needs to be accomplished but not prescriptively "how". For water samples in, methods 8081 and 8082, liquid-liquid extraction and solid phase extraction (SPE) are allowed to extract the target analytes from various aqueous samples. EPA method 3535A outlines several solid phase extraction methods for different EPA methods including 8081 and 8082, which use C18 SPE disk for sample extraction, and elution with methylene chloride (DCM), a toxic chlorinated solvent which needs solvent exchange to n-hexane prior to GC-ECD analysis. Method 3535A is also a performance-based SW-846 method that allows for method modifications. In this poster, we have explored a new elution solvent approach in order to eliminate DCM from the extraction protocol.

SPE Procedure

STEP 1: Sample Preparation

a) Fill sample bottles with 1 liter of water and spike with 5 mL of concentrated sulfuric acid to adjust the pH to less than 2
b) Add 100 µl of standard to the samples

STEP 3: Sample Loading

a) Close all valves and add samples bottles to the bottle holders
b) Slowly run the samples through the cartridges at a rate no faster than 30 mL/minute
c) Once the entire sample is run through, allow the cartridges to dry under full vacuum for 15 minutes

d) While drying, shake/tap the cartridges to remove excess watere) Once fully dry, analyte elution can begin

a) Insert collection vials into the UCT SPeVAP

b) Dry samples down to 1 mL at 40°C with a gentle nitrogen stream
Note: Do not let the samples evaporate to dryness. This will result in analyte recovery loss.
c) Transfer the sample to an autosampler vial to analyze

a) Setup the Hydraflow with UCT ECUNIC18 cartridges
b) Add 10 mL of acetone to the cartridges, allow them to soak for 2 minutes, then draw to organic waste
c) Repeat (b) with 10 mL of hexane and allow the hexane to completely dry out
d) Repeat (b) with 10 mL of methanol
e) Before all the methanol is pulled through the cartridges, close the valves, add 30 mL of DI water, soak for 2 minutes, then pull to aqueous waste down to 20 mL

STEP 2: Cartridge Conditioning

STEP 4: Analyte Flution

a) Rinse the sample bottles with 5 mL of acetone and elute
b) Eluate should be collected slowly at a drop-wise rate
c) Repeat (a and b) 3 more times with 10 mL of hexane
d) The final elution volume should be 5 mL of acetone and 30 mL of hexane, 35 mL total
e) Pack Pyrex funnels with glass wool and add 15 grams of sodium sulfate
f) Rinse the sodium sulfate with 10 mL of hexane and collect as waste
g) Insert the funnels into new 70 mL collection vials
h) Pour the sample over the sodium sulfate and break up any clumping with a glass stir rod
i) Rinse the collection vial with 10 mL of hexane and pour over the sodium sulfate
j) Repeat (i) 2 more times, breaking up clumping between each rinse, for a total solvent volume of 65 mL

NOTES: *: Use new sample bottles, glass vials and graduated tubes as aldrin may degrade on the active sites of the old/used glassware. **: Continue evaporating to 1 mL if higher sensitivity is desired.

REFERENCES

on GC-ECD or GC-MS

- [1] https://www.epa.gov/sites/production/files/2015-12/documents/8081b.pdf
- [2] https://www.epa.gov/sites/production/files/2015-07/documents/8082a.pdf
- [3] https://waste.zendesk.com/hc/en-us/articles/217452058-How-does-EPA-s-Performance-Based-MeasurementSystem-PBMS-approach-affect-SW-846-
- [4] https://www.epa.gov/sites/production/files/2015-06/documents/epa-3535a.pdf

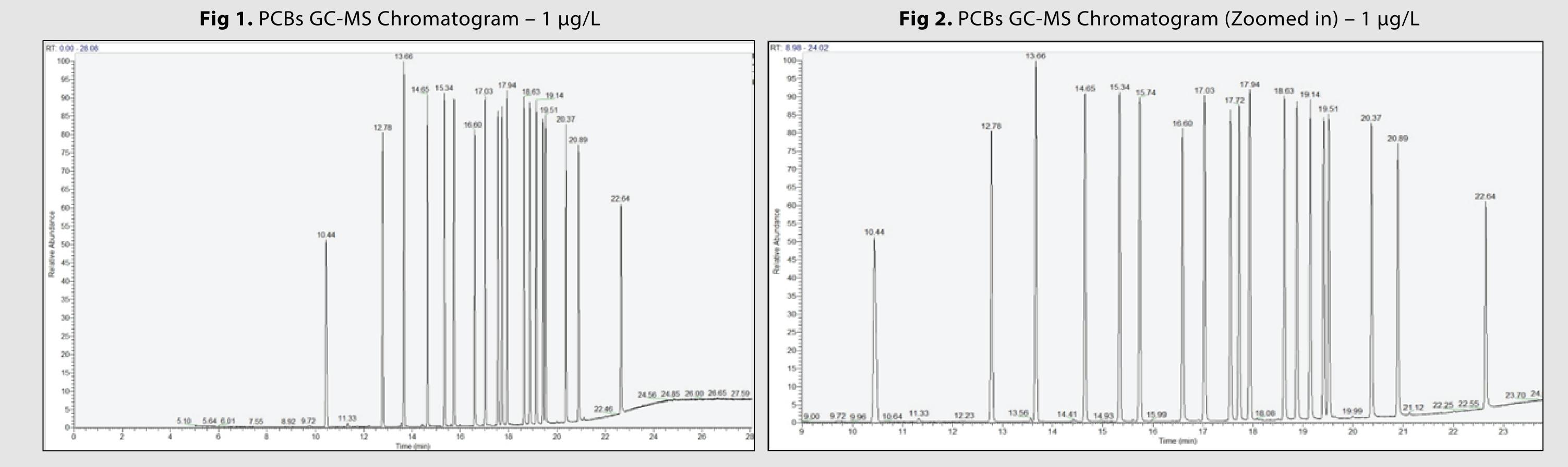
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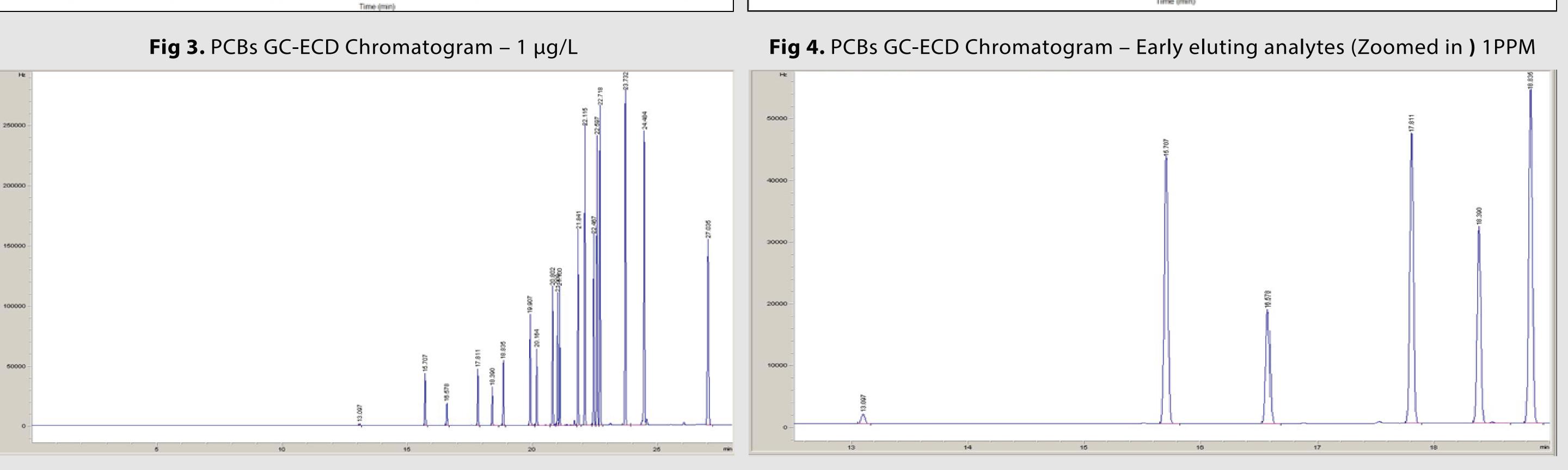




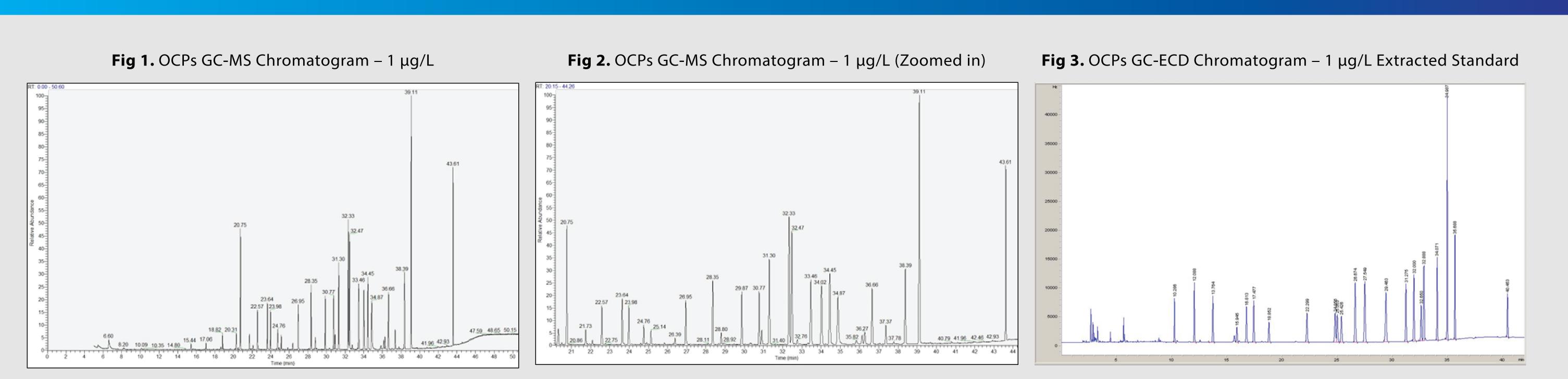
OCPs: GC-ECD INSTRUMENT PARAMETERS s: GC-ECD INSTRUMENT PARAMETERS Front Inlet Initial Temp Pressure Split Ratio Split Flow 10.7 mL/min Detector (μECD) Makeup Flow Temperature Makeup Gas Type Makeup Flow Temperature program Makeup Gas Type Nitrogen Rate (°C/min) Final Temperature (°C) Hold Time (min) **Temperature program** Rate (°C/min) Final Temperature (°C) Hold Time (min)

PCBs CHROMATOGRAMS





OCPs CHROMATOGRAMS



RESULTS

	Analytes	GC-EDC-RT	GC-MS-RT	Recovery %	RSD %
PCBs:	2-Chlorobiphenyl	13.09	10.44	104.3	6
	2,3-Dichlorobiphenyl	15.71	12.78	87.2	10
	2,2',5-Trichlorobiphenyl	16.59	13.66	118.2	9
	2,4',5-Trichlorobiphenyl	17.82	14.65	126.3	7
	2,2',5,5'-Tetrachlorobiphenyl	18.40	15.34	112.1	6
	2,2',3,5'-Tetrachlorobiphenyl	18.84	15.74	108.3	6
	2,3',4,4'-Tetrachlorobiphenyl	19.92	16.60	94.6	4
	2,2',4,5,5'-Pentachlorobiphenyl	20.17	17.03	100.1	6
	2,2',3,4,5'-Pentachlorobiphenyl	20.81	17.55	97.6	6
	2,3,3',4',6-Pentachlorobiphenyl	21.11	17.72	98.4	6
	2,2',3,5,5',6-Hexachlorobiphenyl	21.01	17.94	100.3	6
	2,2',4,4',5,5'-Hexachlorobiphenyl	21.85	18.63	90.2	4
	2,2',3,4,5,5'-Hexachlorobiphenyl	22.12	18.87	95.5	6
	2,2',3,4,4',5'-Hexachlorobiphenyl	22.48	19.14	95.3	5
	2,2',3,4',5,5',6-Heptachlorobiphenyl	22.61	19.40	95.1	5
	2,2',3,4,4',5',6-Heptachlorobiphenyl	22.73	19.51	107.0	6
	2,2',3,4,4',5,5'-Heptachlorobiphenyl	23.74	20.37	95.7	6
	2,2',3,3',4,4',5-Heptachlorobiphenyl	24.49	20.89	93.7	7
	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	27.05	22.64	93.2	5

Analytes	GC-EDC-RT	GC-MS-RT	Recovery %	RSD %
2,4,5,6-tetrachloro-m-xylene	10.28	20.75	83.1	12
α-BHC	12.08	25.10	106.0	10
ү-ВНС	13.76	22.58	113.9	10
β-ВНС	15.94	23.59	108.7	9
δ-BHC	16.81	23.97	108.4	11
Heptachlor	17.47	26.95	108.5	11
Aldrin	18.85	28.36	123.8	21
Heptachlor epoxide	22.29	29.88	106.7	11
cis-Chlordane	24.85	30.79	107.3	10
trans-Chlordane	25.05	31.31	106.2	10
4,4'-DDE	25.42	32.38	100.9	10
Endosulfan I	26.67	31.42	102.3	11
Dieldrin	27.55	32.48	110.3	11
Endrin	29.46	33.48	97.3	29
4,4'-DDD	31.27	34.44	104.5	12
Endosulfan II	32.00	34.02	109.5	11
4,4'-DDT	32.65	36.66	103.3	10
Endrin aldehyde	32.89	34.86	102.6	12
Methoxychlor	34.07	39.10	99.0	10
Endosulfan sulfate	34.99	36.26	102.2	11
Endrin ketone	35.69	38.38	96.8	3
Decachlorobiphenyl	40.45	43.60	83.6	15

CONCLUSION

OCPs:

This Poster describes an optimized SPE procedure using ECUNIC18 SPE cartridges to extract PCBs and OCPs target analytes in water samples, and eluting with acetone and n-hexane mixture instead of DCM. After evaporation, the analytes are enriched in n-hexane thus no solvent exchange is needed for GC-ECD detection. An optimized SPE procedure has been demonstrated for the extraction of EPA method 8081 and 8082 analytes in water. The modified method uses non-chlorinated solvents (acetone and n-hexane) to elute the target analytes and surrogates that are retained on the C18 sorbent. Using Sodium sulfate and then rinsing it a few times has substantially improved Aldrin recoveries. Excellent recoveries > 85 % for most of the analytes in the method and RSD% was < 10%. This is a very good method for a high-volume production lab that would like to streamline and SPE extraction. The method was carried out using UCT's new extraction manifold, the Hydraflow. Many advantages were gained from employing this manifold, one of which is the high vacuum pressure which allows for drying the cartridges. This step is very critical in obtaining acceptable recoveries. Any moisture left in the cartridges can be the root cause behind lower recoveries.