

Determination of Organochlorine Pesticides Residue in Tap Water

by Sepaths-6 SPE/GC-ECD

LabTech, Inc.

Organochlorine pesticides is a broad-spectrum, high efficient, inexpensive pesticides, which were widely used worldwide. However, the strong stability makes organochlorine pesticides accumulate largely through the food chain, which eventually influences human beings health. Because of its strong stability, long half-life, the organochlorinepesticides residue exceeded maximum residue limit (MRL) happens frequently. This study develops a method to determine organochlorine pesticides residue in tap water by using solid phase extraction(SPE) coupled with gas chromatography-electron capture detection(GC-ECD). This method shows great recovery and easy operation.

1. Experimental

1.1 Instrumentation and Materials

Sepaths-6[®] Automated SPE System (LabTech);

GC-ECD;

Sepath-C Nitrogen Evaporators (LabTech)

Methanol (SP. Fisher, US.);

Ethyl Acetate (AR. Beijing Chemical Reagents Corporation, Beijing, China. Distilled before use.)

n-Hexane (AR. Beijing Chemical Reagents Corporation, Beijing, China. Distilled before use.)

SPE disk (LabTech)

1.2 Method Summary

Table 1. Extract method of carbaryl by Sepaths-6[®] Automated SPE system.

Step	Solvent	Soak Time	Volume	Dry Time
Prewet 1	Hexane	3 min	10 mL	0 min
Prewet 2	Methanol	2 min	5 mL	0 min
Prewet 3	Reagent Water	0 min	5 mL	0 min
Process Sample 1 L				

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Wash 1	Reagent Water	--	10 mL	2 min
Rinse 1	Ethyl Acetate	2 min	7 mL	1 min
Rinse 2	Ethyl Acetate	2 min	7 mL	1 min
Rinse 3	Hexane	2 min	8 mL	1 min
Rinse 4	Hexane	2 min	8 mL	2 min

The collected extracts from Sepaths-6 ® Automated SPE system were placed under the Sepath-C Nitrogen Evaporators, evaporated by N₂ till 1.0 mL at 40 °C prior to analysis.

1.3 Apparatus

1.3.1 GC-ECD Method

Column: TM-Pesticide 1 30m×0.53mm×1.0um

Oven Temperature: The initial temperature was 180°C for 4 minutes, which was increased to

220°C at 5°C/min. Then this temperature was held for 6 min.

Injection Temperature: 220°C

Detection Temperature: 280°C

Sample Volume: 2µL

Split Ratio: Splitless

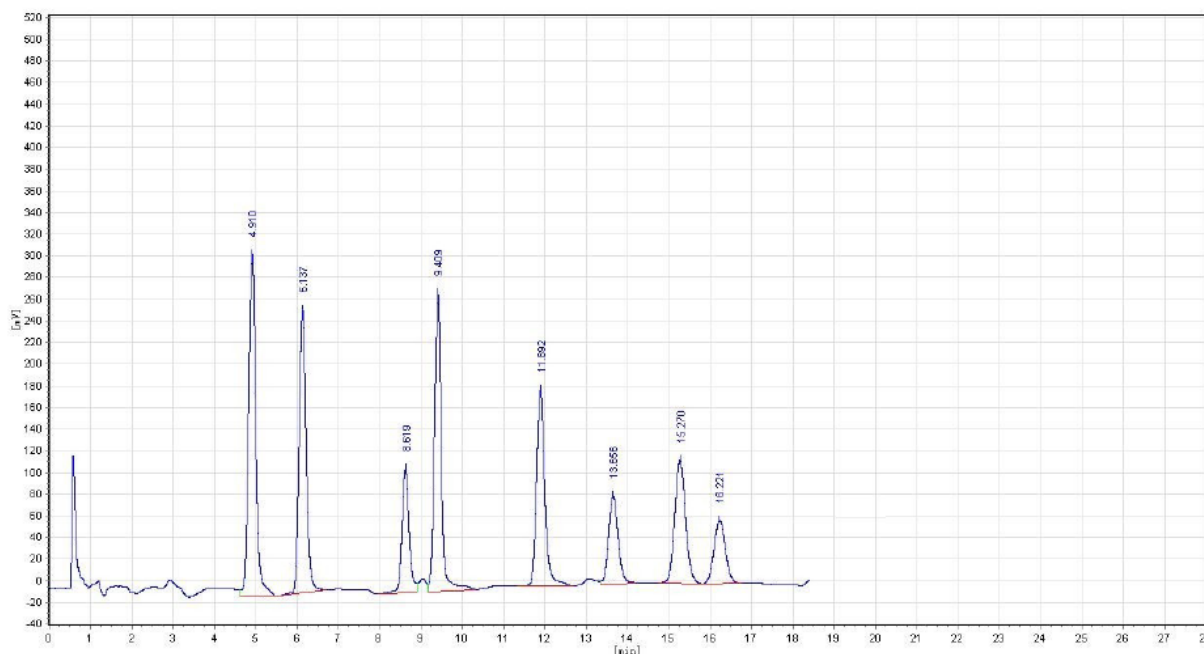


Figure 1. Chromatogram of 8 kinds of Organochlorine Pesticides on GC-ECD.

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2. Results and Discussion

2.1 Calibration Curve and Limit of Detection

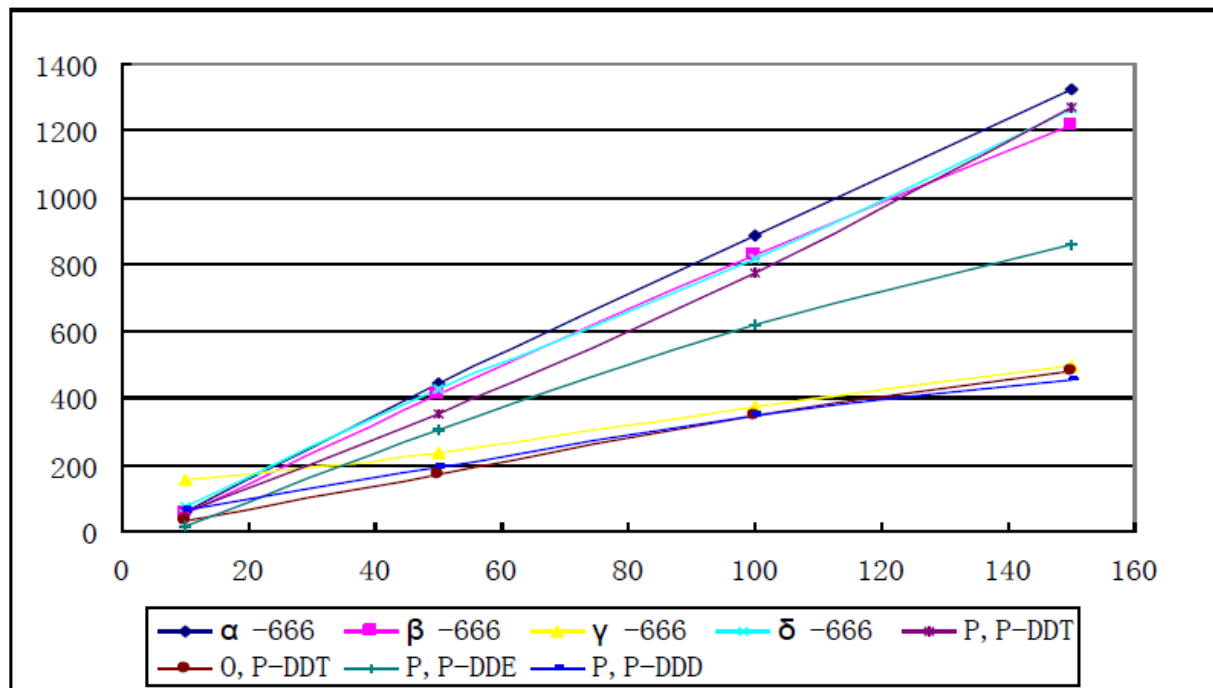


Figure 2. Calibration curves of 8 kinds of Organochlorine Pesticides.

From these plots, the linear range is 10 ppb-150 ppb. The equations are listed below, where x is sample concentration, y is peak area.

α -666	$y = 9.0287x - 21.465$	$R^2 = 0.9996$
β -666	$y = 8.3155x - 15.119$	$R^2 = 0.9993$
γ -666	$y = 2.4953x + 120.71$	$R^2 = 0.9965$
δ -666	$y = 8.4382x - 8.5691$	$R^2 = 0.9994$
P,P-DDT	$y = 8.655x - 57.722$	$R^2 = 0.9958$
O,P-DDT	$y = 3.2334x + 7.0878$	$R^2 = 0.9960$
P,P-DDE	$y = 6.0085x - 14.612$	$R^2 = 0.9922$
P,P-DDD	$y = 2.8035x + 46.326$	$R^2 = 0.9903$

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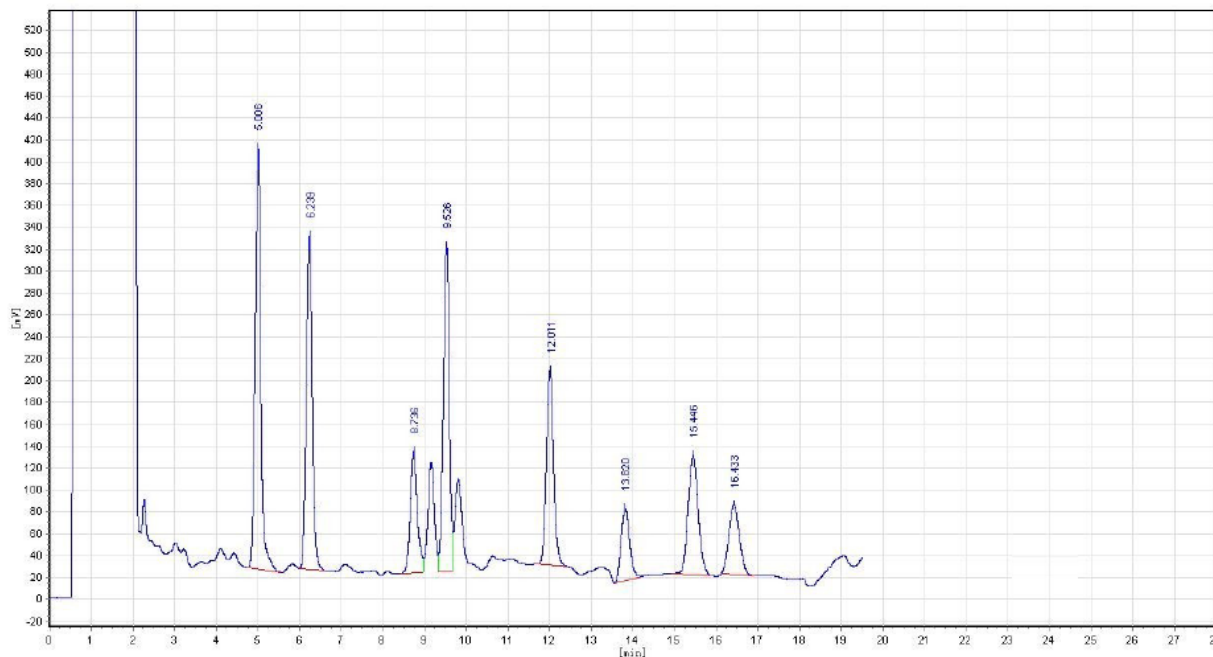


Figure 3. Chromatogram of 8 kinds of Organochlorine Pesticides standard in tap water on GC-ECD.

The accuracy of our method was evaluated by additive recycle experiments. The results of the organic chlorine pesticide were displayed in Table 1.

Table 1. The Recovery of Organochlorine Pesticides in Tap Water.

		Standard (mg/g)	Measured (mg/g)	Recovery (%)
1	α -666	200	213.98	106.99
			203.14	101.57
			193.12	96.56
			188.99	94.50
			197.33	98.67
2	β -666	200	217.55	108.78
			209.90	104.95
			185.01	92.51
			203.17	101.58
			197.28	98.64
3	γ -666	200	204.17	102.08
			212.70	106.35
			175.88	87.94
			268.29	134.14

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			192.87	96.44
4	δ -666	200	225.73	112.86
			198.83	99.42
			207.51	103.76
			226.37	113.19
			191.61	95.81
5	P,P-DDT	200	201.37	100.69
			223.85	111.92
			197.03	98.51
			192.93	96.46
			194.91	97.45
6	O,P-DDT	200	175.30	87.65
			210.62	105.31
			208.16	104.08
			206.97	103.49
			215.77	107.88
7	P,P-DDE	200	215.04	107.52
			185.83	92.91
			179.48	89.74
			209.34	104.67
			203.70	101.85
8	P,P-DDD	200	194.17	97.09
			209.35	104.67
			233.77	116.89
			214.22	107.11
			194.15	97.07

The water samples were spiked with one kind of organochlorine pesticide, extracted via SPE, then analysis by GC-ECD three times continuously. The results of peak area on table 2 below shows the recovery and RSD of 8 kinds of organochlorine pesticides.

Table 2. The RSD of Organochlorine Pesticides in Tap Water.

Item	α -666	β -666	γ -666	δ -666	P,P-DDT	O,P-DDT	P,P-DDE	P,P-DDD
1	3785.448	3009.495	1349.540	2960.282	2129.499	923.274	1871.482	1126.737
2	3844.901	3158.502	1435.250	2942.996	2383.406	972.279	1739.195	1179.389
3	3922.923	3050.534	1313.259	3103.537	2647.132	1084.909	1715.599	1355.31
4	3727.606	3102.758	1475.507	3042.421	2215.406	959.252	1853.438	1279.078
5	3140.690	2531.451	1189.939	2293.325	2145.536	889.795	1705.439	1062.449
RSD (%)	8.48	8.48	8.26	11.43	9.40	7.65	4.46	9.77

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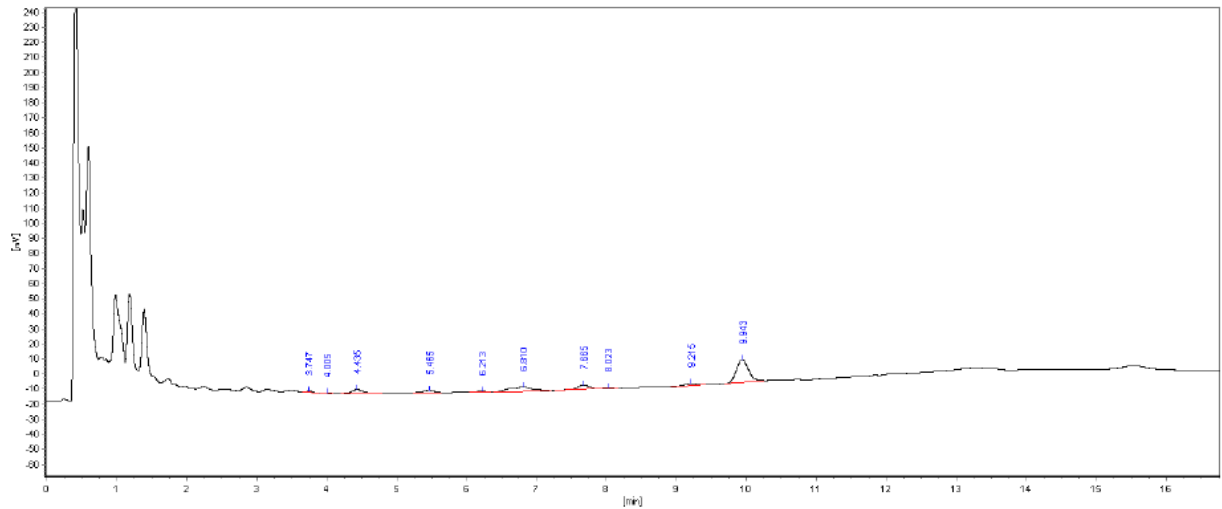


Figure 3. Chromatogram of 8 kinds of organochlorine pesticides spiked in tap water on GC-ECD after SPE.

3. Conclusion

This study develops a method organochlorine pesticides detection by Sepaths-6[®] automated SPE system extracting, followed by GC-ECD analysis. The mean recoveries of 8 kinds organochlorine pesticides are 80.19 % - 91.98 %, and the RSD are less than 7.7 %). The results illustrate that this method is reliable and precise.

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