

Determination of Carbaryl in Tap Water by Sepaths-6 SPE/HPLC

LabTech, Inc.

Carbaryl (1-naphthyl methylcarbamate) is a widely used insecticide in the recent 20 years. The pesticide residue from the environment damages blood system, nerve system, and reproductive system of human beings. In several countries such as Britain, Australia and Germany, carbaryl has been added into prohibited pesticides list. For the concentration of carbaryl in drinking water, both Chinese government and United State Environmental Protection Agency (US EPA) *have strict limit*. High-performance liquid chromatography (HPLC), the conventional method of carbaryl detection, is time-consuming and laborious. With Sepath[®] Automated solid-phase extraction (SPE) system and SPE disks, the efficiency has been greatly increased, saving considerably time and labor. This study develops one application of carbaryl detection in water by pretreating the tap water sample via SPE, and analyzing by HPLC. This method shows a great recovery with facilitated operation.

1. Experimental

1.1 Instrumentation and Materials

Sepaths-6[®] Automated SPE System (LabTech. Ltd, Boston, US.)

Methanol (AR. Beijing Chemical Reagents Corporation, Beijing, China)

Rapidisk C18 47 mm SPE disk (LabTech. Ltd, Beijing, China)

HPLC LC600 System coupled with UV detector (LabTech. Ltd, Beijing, China)

Flow rate: 1 mL / min

Mobile phase: Methanol / Water (v / v) 65 / 35

Column: Ultimate C18 (4.6 mm × 250 mm, 5 μm)

Column Temperature: 35 °C

Sample Volume: 20 μL

Wavelength: 280 nm

1.2 Method Summary

- 1) 1000 mL water samples were used.
- 2) A 40 mL 0.8 ppm carbaryl standard solution was added into sample bottle.
- 3) Put the sample bottle onto the Sepaths-6[®] Automated SPE Module with the Rapidisk C18 SPE disk.
- 4) 40 mL collecting bottle was placed at the solution outlet to collect *extract*.
- 5) Loaded method listed in table 1 and got started.
- 6) After the method completed, took off the collecting bottle.
- 7) Added Methanol till volume reached 40 mL.
- 8) Analyzed by HPLC.

1.3 Extract Method

LabTech, Inc.

Address: 114 South Street, Hopkinton,
MA 01748, U.S.A

Tel: (508) 435-5500

Fax: (508) 435-5595

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

Step	Solvent	Soak Time	Volume	Dry Time
Prewet 1	Methanol	1 min	5 mL	30 sec
Prewet 2	Methanol	1 min	5 mL	30 sec
Prewet 3	Reagent Water	5 second	5 mL	2 sec
Prewet 4	Reagent Water	5 second	5 mL	2 sec
Process Sample				
Air Dry 2:00 min				
Rinse 1	Methanol	2 min	5 mL	20 sec
Rinse 2	Methanol	30 sec	5 mL	20 sec
Rinse 3	Methanol	30 sec	5 mL	20 sec
Rinse 4	Methanol	30 sec	5 mL	1 min

In the above experiments, it takes 35 min to run six 1-Liter samples from 6 channels, respectively. Compared with single-channel SPE or manual SPE instruments, the efficiency of our Sepaths-6 ® Automated SPE system has been greatly enhanced.

2. Results

2.1 Chromatogram of Standard, Control, and Spiked Sample Solutions

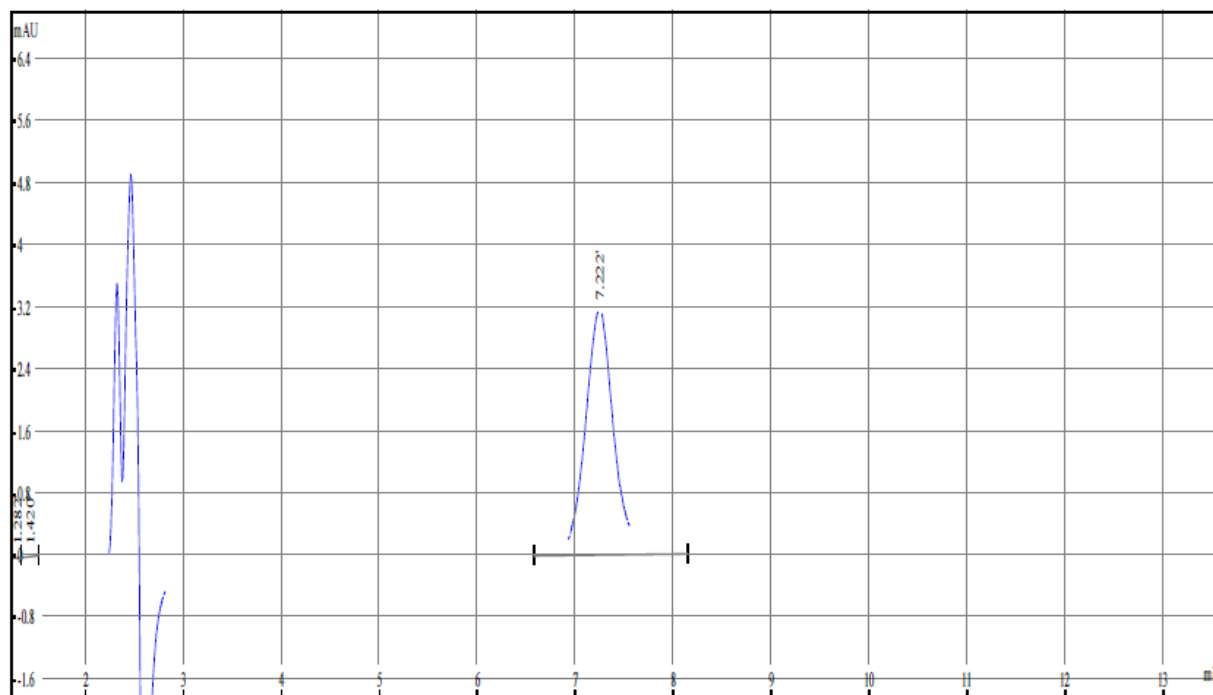


Figure 1. HPLC chromatogram of carbaryl standard.

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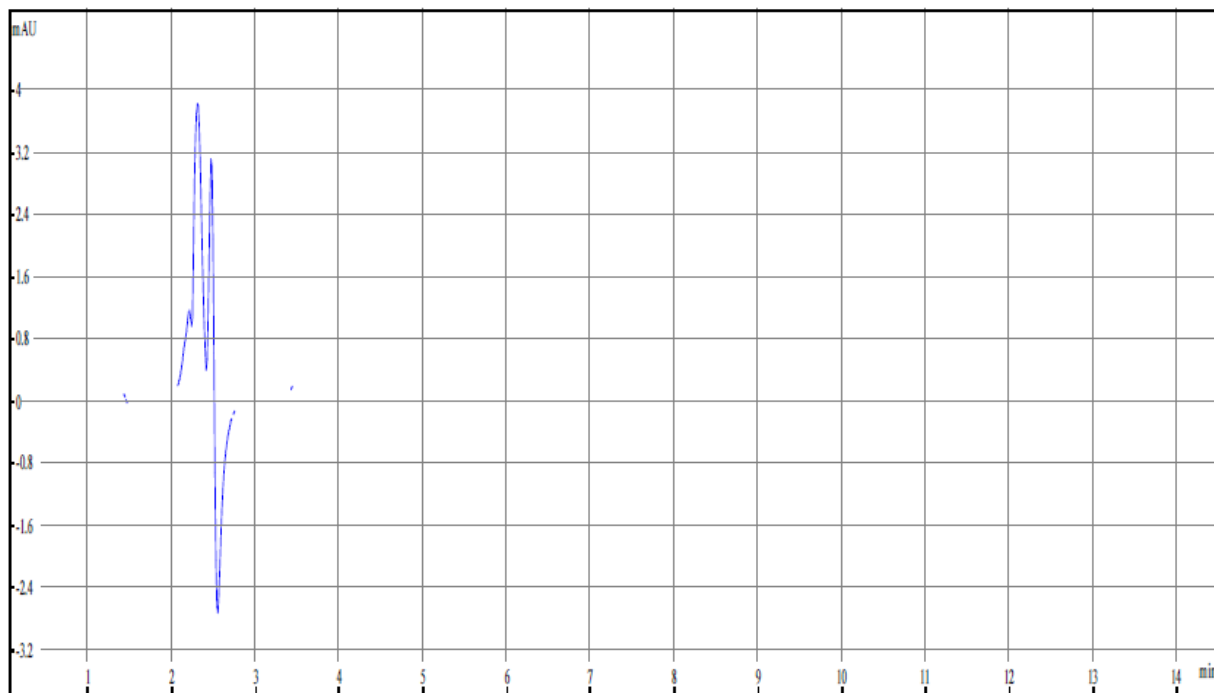


Figure 2. HPLC chromatogram of tap water after SPE.

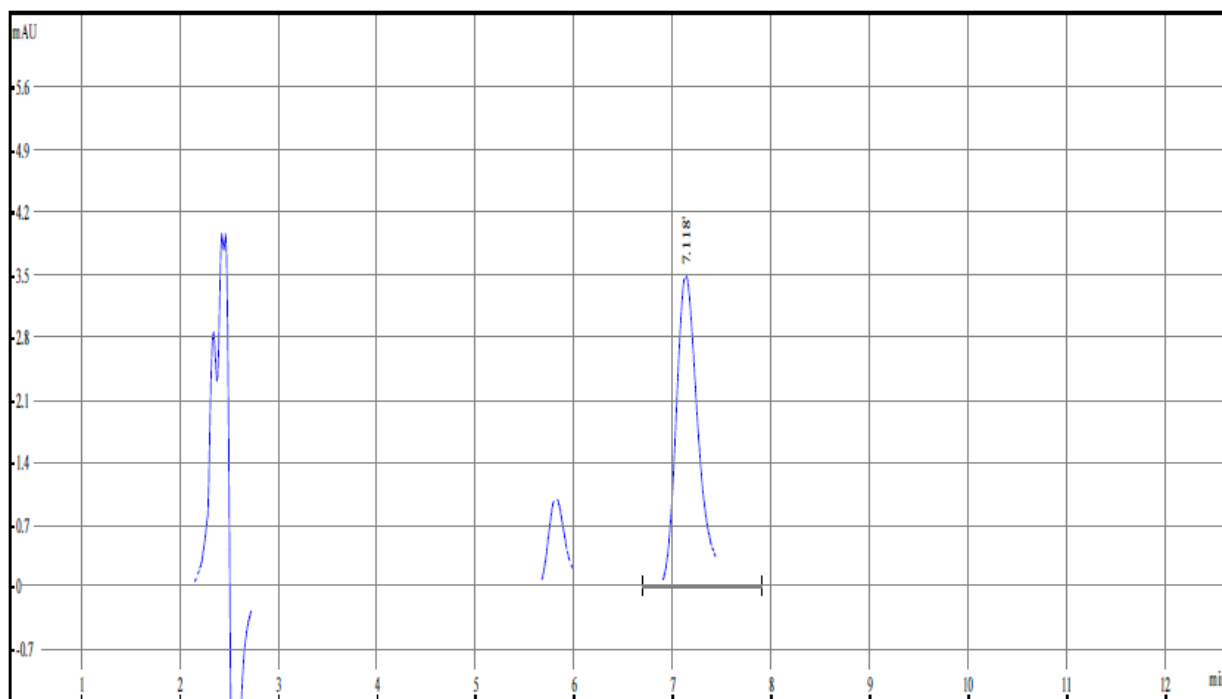


Figure 3. HPLC chromatogram of tap water spiked with carbaryl.

2.2 Recovery of Carbaryl Spiked Water Samples

To determine the precision of our system, 20 spiked tap water samples were analyzed by HPLC. 1-Liter tap water samples were spiked at a concentration of 0.8 mg/L with carbaryl standard. After extracted by Sepaths-6[®] Automated SPE system, the extracts were diluted with methanol to 40 mL. Recovery was calculated by comparing the ratio(s) of analyte peak areas.

Test Number	Standard Spiked (mg/L)	Analyzed Results (mg/L)	Recovery (%)	Avg. Recovery (%)	RSD
1	0.8	0.6983	87.29	93.50	6.04
2		0.7271	90.89		
3		0.7321	91.51		
4		0.7244	90.55		
5		0.6902	86.27		
6		0.7598	94.97		
7		0.6833	85.41		
8		0.6975	87.19		
9		0.7762	97.03		
10		0.7781	97.26		
11		0.7802	97.52		
12		0.7866	98.33		
13		0.7349	91.86		
14		0.7310	91.37		
15		0.7310	89.12		
16		0.8217	102.71		
17		0.8300	103.75		
18		0.7998	99.98		

3. Conclusion

This study develops a method of carbaryl extraction via Sepaths-6[®] automated SPE system coupled with HPLC-UV analysis. This method has good recovery (mean recovery 93.5 %) and the great repeatability (RSD 6.04 %). The results illustrate the reliability of this method and the instrument, which is high efficient, fast and simple to operate.

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