

## **QUICK METHOD FOR DETERMINATION OF ORGANOCHLORINE PESTICIDES IN DRINKING WATERS. APPLICATION AT ACCIDENTAL CONTAMINATION**

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**Abstract.** Due to the more frequent incidental contamination with pesticides in recent years a simple and inexpensive method for determination of organochlorine pesticides in drinking water has been developed. It is a modified version of classic GC/ECD method based on a single hexane extraction without concentration and clean-up steps. The detection limits for different organochlorine pesticides are from 0.02 to 0.1  $\mu\text{g/l}$ ; variation coefficients – from 3 to 16 %, specific mean recovery – from 65 to 90%. The method is used for determination of pesticides in drinking water in three cases of incidental contamination in the country. The highest quantities detected are: trifluralin: 1700  $\mu\text{g/l}$  and DDT (total) – 0.32  $\mu\text{g/l}$ .

*Keywords:* organochlorine pesticides determination, drinking waters, G-ECD method.

### **AIMS AND BACKGROUND**

The WHO and the EU recommendations set a limit on total pesticide content in drinking water of up to 0.5  $\mu\text{g/l}$  and on the content of a particular pesticide of up to 0.1  $\mu\text{g/l}$ . Although the majority of organochlorine pesticides (OCP) is banned for application in Bulgaria, still there are residual amounts of them detected in the environmental compartments due to their high cumulative ability and persistence.

During the past years there are more frequent accidental contaminations with OCP as a result of unregulated storage and disposal of residues of banned pesticides or criminal activities. The necessity of a quick reaction in such cases made us search for a quick, simple and economical method for determination of OCP in drinking water.

### **EXPERIMENTAL**

This method is based on single extraction with hexane of OCP from the water directly in the sampling container and gas chromatographic (GC) determination with electron capture detector (ECD).

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One litre of water was collected in a glass bottle. The sample did not need conservation. It was stored in a refrigerator and the determination was performed within 24 h.

Before the extraction some 200 ml of the water were poured out. The level of the liquid in the bottle was marked in order to enable the further check of the exact amount of the sample collected for analysis. 10 ml of hexane were added to the 800 ml of the water sample. The extraction was performed for about 5 min in the bottle in which the sample entered the laboratory. Injection of 1 to 5  $\mu$ l of hexane extract (upper layer) into the gas chromatographer (GC). If necessary the hexane layer can be moved to the narrow part of the bottle by addition of distilled water. Gas chromatographic determination – GC with ECD.

The analytical parameters of the method were determined with a gas chromatographer Perkin Elmer – AutoSystem XL with ECD and capillary column RTX 1701, L 30 m, id 0.25mm, ft 0.25  $\mu$ m, at the following GC conditions:

$T_{inj} = 200\text{ }^{\circ}\text{C}$ ,  $T_{det} = 375\text{ }^{\circ}\text{C}$ , speed of Ha make-up gas 30 ml/min, speed of the mobile phase (He) 0.43 ml/min, flow separation 1:50. The temperature program of the column was: from 100  $^{\circ}\text{C}$  with 5 $^{\circ}$ /min to 275 $^{\circ}\text{C}$ , 275 $^{\circ}\text{C}$  for 5 min.

Figure 1 presents a chromatogram of OCP standard solution at these conditions.

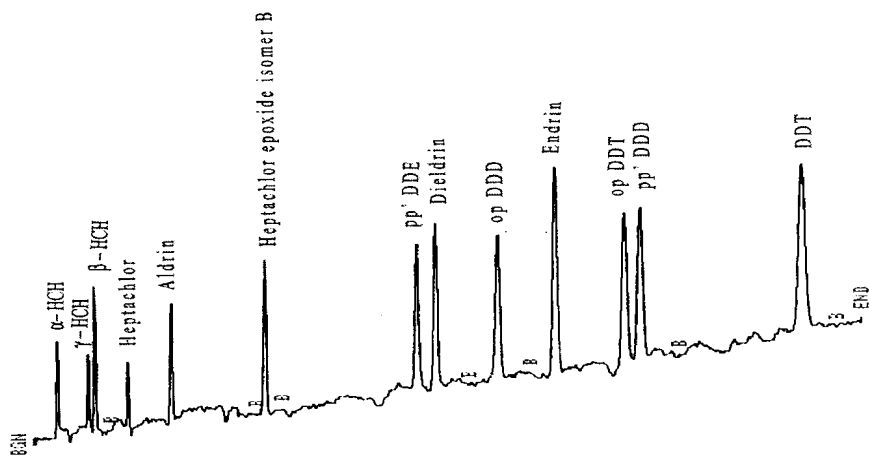


Fig. 1. Chromatogram of OCP standard solution (0.01 mg/ml) at these conditions

The determination of the analytical parameters was performed using 5 samples of drinking water with standard additive (Table 1). The linear range of the detector for the different OCPs is in the range 10–20.

The methods provide a possibility for increasing the sensitivity up to 10 times by separating an aliquot from the extract and its concentration by ventilating with nitrogen.

**Table 1.** Analytical parameters of the method using hexane as extracting solvent

Compounds	<i>n</i>	C (µg/dm <sup>3</sup> )	$\bar{X}$ (µg/dm <sup>3</sup> )	Sx (µg/dm <sup>3</sup> )	V (%)	A (%)
Treflan	5	0.050	0.036	0.004	9.7	72
Lindane	5	0.050	0.033	0.001	2.7	65
Aldrin	5	0.050	0.045	0.003	7.2	90
Alachlor	5	1.000	0.850	0.224	26.3	85
Dieldrin	5	0.050	0.041	0.010	23.2	82
pp'-DDE	5	0.050	0.034	0.007	20.5	67
op-DDT	5	0.050	0.031	0.010	33.0	62
pp'-DDT	5	0.050	0.032	0.013	41	63

*n* – number of samples; C – concentration of standard water solution;  $\bar{X}$  – mean value; Sx – mean standard deviation; V – variation ratio; A – recovery.

This method was applied at determination of OCP in water at some accidental pesticide contaminations. The results of those analyses are presented in Table 2 and Fig. 2.

**Table 2.** OCP amounts detected in water at four accidental contaminations

Site	Identified pesticide	Sample No	Detected amounts (µg/l)
1	2	3	4
Local water source, village Tuhoviste, Satovcha district	Lindane	N1	0.14
		N2	<0.1
		N3	<0.1
Locall wells, village Sokolare, district Byala Slatina	Treflan	N1	1700
		N2	<0.1
		N3	<0.1
		N4	<0.1
Koinare village N1- central drinking water source	op-DDT	N1	0.06
		N2	0.08
		N3	0.46
N2, N3 – surface waters, Dola river	pp'-DDT	N1	0.19
		N2	0.17
		N3	0.83
	pp'-DDE	N1	0.06
		N2	0.04
		N3	0.30
	DDT <sub>tot</sub>	N1	0.32
		N2	0.30
		N3	1.63

to be continued

1	2	3	4
Zvanartsi village	op-DDT	N1	0.13
N1 – local well,		N2	0.046
		N3	0.144
Town of Kubrat	pp-DDT	N1	0.093
N2, N3-surface		N2	0.374
open water bodies		N3	0.273
	pp-DDE	N1	0.020
		N2	0.151
		N3	0.064
	DDT <sub>tot</sub>	N1	0.126
		N2	0.570
		N3	0.480

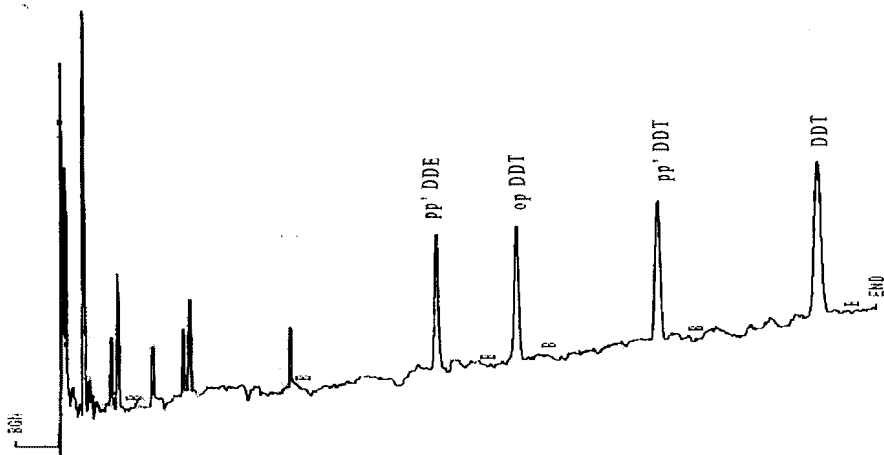


Fig. 2. Chromatogram of accidental pesticide contaminations in Koinare village

## CONCLUSIONS

The application of this method enables the elimination of purification and concentration of combined extracts that could cause loss of the determined compound or a greater variation ratio. In all cases the routine methods require exclusive precision during the course of the analysis using large amounts of organic solvents and, in some cases, special glassware. To the contrary, this method provides a very simplified procedure, hardly requires any laboratory glassware and uses minimal amounts of hexane (10 ml per sample).

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