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Contamination of Potato Tubers and Carrots in Greece with Lindane Residues

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Lindane is the common name for gamma-hexachlorocyclohexane, an insecticide formulated as a dust, in the form of aerosoles, wettable powders, emulsions, granules and in tablets for fumigations. It has been used for the protection against insects of wheat, cotton, corn and beet cultivations and in seed treatment (U.S. E.P.A 1988). It is a toxic organochlorine compound that can be hazardous to humans, animals and the environment if incorrectly or carelessly handled.

Lindane is degraded slowly in the environment. Its field half life is usually well above 100 days (Wauchope et al 1992) depending on many factors e.g. the specific site, the type of soil and the climate. For this reason lindane use in Greece was restricted to seed treatment and soil application, 8-months before sowing of potato and 12-months before sowing of carrot cultivations. This restriction however did not have practical effects and information existed during the last years for improper use of lindane in potato and carrot cultivations. In the present study, potato and carrot samples originated from various intensively cultivated regions of Greece were analyzed during the period 1988-1992 for determining lindane residues.

MATERIALS AND METHODS

Potato and carrot samples were taken from fields of six different intensively cultivated regions of Greece : Achaea, Attiki, Eleia, Euboea, Messinia and Voeotia, during the period 1988-1992. Sampling was performed close to harvest, and according to FAO/WHO recommendations (1986). One sample, at least 2 kg, of 12 potato tubers or carrots was taken from different sites of the field. A second sample was taken from fields with area greater than 1 Ha. All samples were forwarded to the laboratory within 2 days of collection, and homogenized within 1

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day. The homogenized material was refrigerated in glass jars until analysis. Control samples were collected from fields known not to have been treated with lindane.

The general method of A. Ambrus et al. (1981) was followed for the extraction. According to the method 150 mL of acetone are added to 50 g of homogenized sample and the mixture is blended at high speed. The extract is filtered through a Buchner funnel, rinsed with 50 mL of acetone, and transferred into a 1-L separatory funnel. 450 mL of 4% Na_2SO_4 solution are added in the funnel and lindane is extracted with 100-, 50- and 50- mL portions of CH_2Cl_2. The extracts are filtered through 30 g of anhydrous Na_2SO_4 which is then rinsed with 20 mL of CH_2Cl_2. The volume of the filtrate is reduced using a vacuum rotary evaporator at 30°C to ca 2 mL. 10 mL of acetone are added, and the solution is evaporated to ca 2mL. This step is repeated twice. The solution is then evaporated to dryness and 10 mL of acetone are added. The sample extract after filtration through Na_2SO_4 and without any cleanup is transferred to a 10-mL volumetric flask, acetone is added up to volume and the sample is ready for gas chromatographic analysis.

A Varian aerograph model 3700 gas chromatograph equipped with a Ni-63 electron capture detector, and a 2 m X 2 mm i.d. glass column containing 1 : 1 mixture of 10% OV-101 and 15% OV-210 on Chromosorb WHP (80-100 mesh), was used. The injection port temperature was 220°C, the detector 300°C and the column temperature was programmed from 160° (10 min.) to 200°C (10 min.) at a rate of 5°C/min. Nitrogen carrier-gas flow rate was 60 mL/min. 1μL of the sample extract was injected and quantification of lindane was performed by measuring the peak height.

RESULTS AND DISCUSSION

The method of analysis used was simple and fast, as the cleanup stage was deleted. The response of the electron capture detector for lindane was linear in the range 0.005-0.05 ng with correlation coefficient r=0.994. Quantitation of lindane in samples was made by comparing the detector response for the sample to that measured for the calibration standard, within the linear range.

The efficiency of the method was evaluated by spiking control samples with lindane at various concentration levels. Figure 1 shows a gas chromatogram of a fortified potato sample. The results of the recovery study are presented in Table 1. Average recoveries were from 98 to 108% and coefficients of variation from 5.8 to 14.4%, values within the accepted range (Greve, 1984). The method's limit of detection is evaluated as the product of the standard deviation at the lowest validation level with the Student t-value (U.S., E.P.A. 1984). The t-value

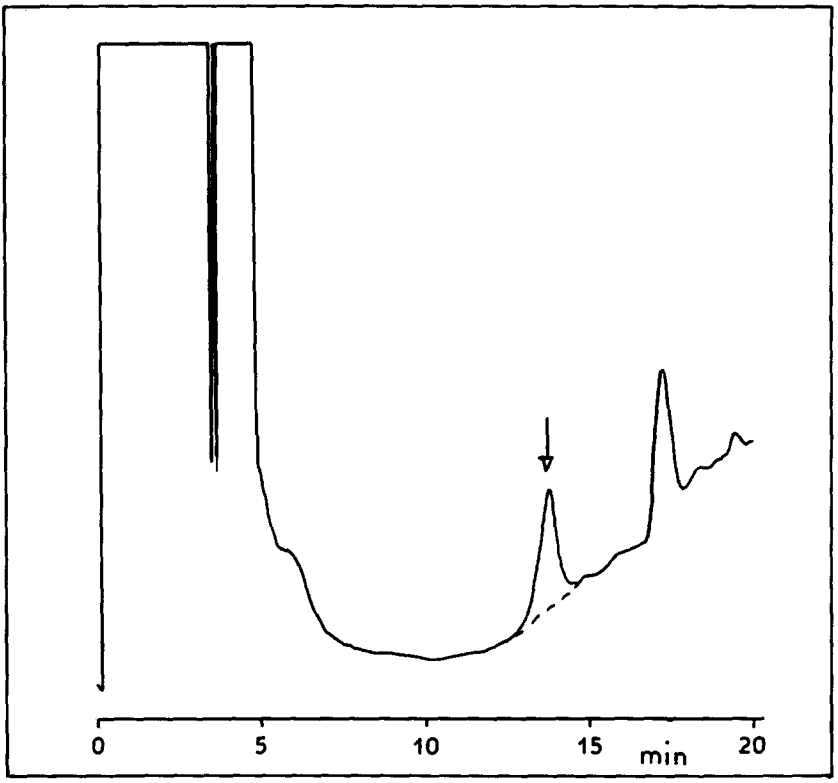

Figure 1. Gas chromatogram (1µL) of control potato sample fortified with 5 µg/kg lindane. The dotted line is the method blank and lindane is indicated with the arrow.

Table 1. Mean recoveries (%) and coefficients of variation (C.V.) for lindane in potatoes, at various fortification levels.

Concentration (µg/kg)	Recovery (%)	C.V. (%)*
1	103	5.8
2.5	108	14.4
5	101	9.2
7.5	102	8.1
10	98	8.0
15	104	7.3

* Three samples for each fortification level

is 6.96 at 99% confidence level for 2 degrees of freedom and the detection limit was found to be 0.4 µg/kg.

83 potato and 25 carrot samples were analyzed for lindane according to the described method. Each sample was analyzed in duplicate and the average result was recorded. The results are presented in Table 2. As seen from this table, lindane residues in potatoes ranged from

Table 2. Lindane residues (μg/kg) in potatoes and carrots from various regions of Greece. P1 to P5 = potatoes from Achaea, Eleia, Euboea, Messinia and Voeotia respectively, C1 and C2 = carrots from Attiki and Voeotia respectively. ND = not detected.

Origin	1988				1990				1991	1992			
P1	ND	10	15	75	ND	ND	ND	27	ND	ND	ND	3	
	ND	10	30	250	ND	ND	ND		ND	ND	1	3	
	ND	10	40		ND	ND	4		1	ND	1	4	
	ND	10	70		ND	ND	10		3	ND	3	7	
P2	ND	15								ND	ND	ND	4
	ND	75								ND	ND	ND	11
	6									ND	ND	1	15
										ND	ND	3	
P3	ND	ND	ND	ND	1								
P4	ND	ND	ND	ND									
P5	ND	ND	ND	9						ND	1		
	ND	ND	ND							ND	4		
C1	ND	ND	ND	5									
	ND	ND	1	9									
C2	ND	ND	9	46						ND			
	ND	1	9	90						1			
	ND	5	10							1			
	ND	8	10										

not detected (< 0.4 μg/kg) to 250 μg/kg and in carrots from not detected to 90 μg/kg. All these values are well below the national maximun residue limit (MRL) for lindane, that is 1 mg/kg. However, residues in three potato samples of 1988 exceeded the MRL set by the Codex Alimentarius Commission (FAO/WHO,1986) that is 0.05 mg/kg. Lindane residues in all carrot samples were also below the MRL set by the Codex Alimentarius Commission (0.2 mg/kg).

From the 83 analysed potato samples, lindane residues were detected in 34 samples (40%) and not detected in 49 samples, and from the 25 analysed carrot samples lindane residues were detected in 14 samples (56%) and not detected in 11 samples. However, as it can be seen from the data of Table 2, lindane residues were not detected in potatoes from Messinia, and were rarely detected in potatoes from Euboea and Voeotia. It is also seen that lindane residues were mostly detected in potatoes from Achaea and Eleia, areas of Greece in the Peloponnese peninsula. Furthermore, it is evident from the data that lindane residues decreased from 1988 to 1992. Thus, the 1992 samples were found to have either nondetectable or very low concentrations (≤ 15 μg/kg) of lindane. This can be attributed to a series of actions taken by the State since 1989, concerning the distribution of lindane active ingredient and formulations. These actions were taken following our suggestions that were based on the 1988 data, which showed significant lindane residues in some of the samples, and included, among others, reduction by 20% in a year of the imported lindane quantities, restrictions of the approved lindane uses, and proper information to the Greek farmers, especially those in the Peloponnese peninsula. As it is seen from the latest data of Table 2 (1990-1992) these actions were proved to be effective and lindane residues decreased significantly.

Soil samples from the studied areas were not analysed, since the aim of the study was restricted to lindane residues in potatoes and because it is known from similar studies that approximately 1% of the soil-applied lindane is translocated into plants (Fuhremann and Lichtenstein 1980). However water samples from lakes and rivers near some of the studied areas were analysed during the years 1990-1992 (Miliadis 1993) and lindane residues were detected in the water of the lake Iliki, a lake in Voeotia adjacent to the cultivated with potatoes and carrots fields. Lindane concentrations in water of Iliki lake were found to be from 0.005 to 0.015 μg/L, values below the EEC maximum allowable concentration (0.1 μg/L).

From the results of this study it can be concluded that lindane residues in the potato tubers analysed arose after soil application of lindane formulations. These results are in accordance with findings in other European

countries (Szokolay et.al. 1977), and indicate that lindane formulations should be used carefully and according to the good agricultural practices in order to have products with low or non-detectable residues.

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