A MULTI RESIDUE ANALYSIS OF TEN ORGANOPHOSPHOROUS INSECTICIDES IN WATER BASED ON SOLID –PHASE EXTRACTION AND GAS CHROMATOGRAPHY – MASS SPECTROMETRY

BY

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ABSTRACT

A multi residue method for analysis of 10 organophosphorous (OPs) insecticides in water has been developed. The OPs were selected among the most used as insecticides during the last 10 years in Egypt and Kuwait. Solid phase extraction (SPE) using a graphitized carbon black (GCB) (Carbopack B Cartridge) was selected as extraction method. Extracts were analyzed by Gas Chromatography with capillary column and Ion Trap Detector (GC-ITD). The recoveries were satisfactory, above 90% with relative standard deviation (%RSD) ranging from 4.5 to 10.2%. The method detection limit (MDL) and the limit of quantification (LOQ) were ranged from 14 to 25 ng/L and from 50 to 80.1 ng/L respectively. MDL and LOQ were below enough for determination multi residues of tested OPs in water at the maximum residual levels (MRLs)

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set by European Economic Community (EEC) and World Health Organization (WHO) . This analytical procedure is simple and demonstrates good accuracy, sensitivity, selectivity, precision to be applied successfully for analysis of some water samples in Egypt and Kuwait.

INTRODUCTION

OPs insecticides are more soluble in water (more mobile) [Fest and Schmidt (1982)] and may contaminated large area of aquatic environment of surrounding treated fields. Residues reach the aquatic environment through direct runoff , leaching, careless disposal of empty containers, equipment washing.. etc., Barcelo and Hennion (1997). Senseman *et al.* (1997), found that from 0.01 – 5% of insecticides applied on field are lost in runoff events. OPs are routinely detected in surface and ground water, Miliadis (1993) and (1994), Jimenez *et al*, (1997), Khalifa *et al.* (2000), Garrido *et al*. (2000) and El-Meshaan *et al*. (2002).

The European Economic Community (EEC) (1988) and WHO (1993), have strict legislation on the occurrence of pesticide chemicals in water intended for human consumption, the maximum residual level (MRL) of a pesticide chemical must be below 100 ng / L and sum of all pesticides not exceed 0.5 ug / L and in surface waters, not more than 3ug/L. Concentration over the MRL represents hazardous effects on human and environment health. Thus there is a real need for suitable analytical methods for monitoring the residues of large numbers of OPs (method for multi residues analysis) in aquatic environment at levels of MRLs (100ng/L) and below .

In practice Gas Chromatography (GC) using long capillary column and selective and sensitive detectors such as, Nitrogen Phosphorous Detector (NPD) have been employed as analytical technique for OPs residues in water, (Colina *et al.*, 1993; Miliadis *et al* 1994 and Jimenez *et al*., 1997). In GC technique, relative retention time are the criteria applied for identification of chromatographic peaks but an additional confirmatory technique is also necessary. To this end, the coupling of GC with mass spectrometry (MS) has extensively used, as it increase the selectivity and certainty of

identification avoiding false positive in determination of pesticides residues in water. GC-MS as analytical tool has been used in multi residue determination of pesticide residues in water, Miliadis *et al*. (1995), Pablos *et al*. (1999), Khalifa *et al*. (2000) and El-Meshaan *et al.* (2002). Determination of trace amounts of OPs in environmental aqueous samples requires an extraction step before chromatographic analysis. Extraction step is often necessary to raise the concentration of the analyte found in water at trace level above the sensitivity of the detector of the analytical tools such as, GC, GC-MS. The interested pesticide chemicals found in water were extracted using cartridges filled with graphitized carbon black (GCB) materials such as Carbopack B (60-80 mesh, 100cm^2/g) which used as efficient sorbent in SPE for extraction of trace amount of more polar organic pollutants from water such as OPs and their transformation products, Dicorcia and Marchetti, 1991; Dicorcia and Samperi,1993; Dicorcia *et al*. ,1993; Crescenzi *et al.*,1997; Sabik,1998 and Bolden *et al*.,2000. The present work was aimed to develop a multi residue analytical procedures based on solid phase extraction (SPE) technique using a graphitized carbon black (GCB), Carbonpack B for extraction of 10 organo-phosphorous insecticides (OPs) from water samples followed by Gas Chromatography-Mass Spectrometry (Gas Chromatography-Ion Trap Detector, GC—ITD) as analytical tool. Applications of the developed method for analysis of water samples for environmental pollutants are discussed.

MATERIALS AND METHODS

1. Chemicals and Reagents:

a- Chemicals :

The following OPs insecticides were chosen for this study, mevinophos, ethoprophos, diazinon, parathion-methyl, tertrachlorvinphos, fenthion, fensulfothion, azinphos-methyl, sulprofos and coumaphos. Reference standard materials for these OPs were obtained from Riedel-De – Haen (Seelze, Hannover, Germany) always with a purity higher than 99%. Pesticide quality solvent, n-hexane, methanol, dichloromethane and ethyl acetate were supplied by BDH, poole, UK. Ultra pure water was prepared

by Milli Q2 plus (Millipore Corporation, USA). Anhydrous sodium sulphate was purchased from Merck, Darmstadt Germany) for pesticide residue analysis and was purified by heating at 300C over night and later Soxhlet extracted for 12 hours with dichloromethane. Ascorbic acid 99% (Signma-Aldrich, Germany). Carbopack B Cartridges for Solid phase extraction, a plypropylene Cartridge (6.5 X 1.4 cm ID) packed with 500 g graphtized carbon black (GCB), 60-80 mesh were purchased from Sigma – Aldrich, GmBH, Germany.

b- Reagents :

Stock standard solution 200 ug / ml was prepared in n-hexane from each OP compound and stored at -30◦C. Working standards mixture solution was prepared by appropriate dilutions in n-hexane and stored in a refrigerator at 4ºC. Ascorbic acid solution pH2 was prepared by dissolving 10g of ascorbic acid in one liter of ultra pure water and the pH of the solution was adjusted to 2 using concentrated HCl.

2. Experimentals:

a. Recovery experiments and Solid phase extraction (SPE) procedure using Carbopack B Cartridge.

In this study, extraction of OPs from water samples was based on SPE using Carbopack Cartrdige as a sorbent. To evaluate the ability of Carbopack B to retain the investigated OP compounds from water and to release them during the desorpiton proceses, the recovery was determined for each compound. For this purpose a 1 liter of ultra pure water was fortified at levels, 0.1 and 0.5ug / L from standards mixture of OPs using methanol as a carrier solvent and continuously mixed for 30 min prior to the initiation of the experiment. The fortified water samples were extracted using a Carbopack B as a sorbent in the form of cartridge. These cartridges were first cleaned and conditioned by passing sequently 10ml of ethyl acetate, 10ml of methanol followed by 20ml. of ascorbic acid solution pH2. then the outlet of the cartridge was fitted into a side arm of filtering flask. PTFE tube (ID, 1/16in) was connected to the inlet of the cartridge through an adapter of 2 liters glass reservoir for the transferring the water sample to the cartridge. SPE extraction starting by transferring the waster sample into the reservoir and forcing it to pass through the

cartridge by vacuum (20 – 30 mmHg) (using water pump) at a flow rate 50ml./min. (Note : the sorbent inside the cartridge was never allowed to drying during conditioning and sample loading). Following sample application the cartridge was rinsed with 5 ml of ultra pure water, then aspirated for 2 min. to remove residual water on the sorbent. After the cartridge had dried, the water pump was disconnected and the cartridge was turned upside down and purged with a stream of nitrogen (2 min) for complete removing the residual of water. The OPs compounds retained on the sorbent were then eluted by running 25 ml of n-hexane: ethylacetate (90 : 10 v/v) through the cartridge into a 50ml conical flask followed by 10ml. of n-hexane at a rate of 5 ml / min. (using a hypodermic syringe). Last drops of the solvent on the sorbent were forced out into the conical flask by flashing a stream of nitrogen for 1 min. The eluent was dried immediately through a glass column (15 X 2.5 cm ID) filled with 10g anhydrous sodium sulphate. The dried extract was transferred to 50 ml round bottom flask and concentrated to near dryness by rotary vacuum evaporation at 30ºC. The flask was rinsed three times with 1 ml of n-hexane and collect the rinsed solvent into a 10ml graduated conical test tube. Lastly the extract in the tube was reduced to 0.5 ml. of n-hexane by water bath at 30 ºC under a nitrogen stream. Before analyses the extracts were centrifuged at 2000 rpm for 10min. A method blank was performed using a volume of 1 liter of ultra pure water and extracted as for fortified water samples.

b- Analysis :

Extracts of water samples (1-2 ul) were analyzed for 10 OPs utilizing a bench top Gas Chromatography-Mass Spectrometry (Gas Chromatography-Ion Trap Detector, GC-ITD) which consisted of a Varian 3800 series Gas Chromatograph interfaced to a Saturn 2000 Ion Tap Detector (Varian Instruments, Synnvale, CA, USA). The GC equipped with a split/splitless port which was operated in splitless mode (purge time set at 1 min.) and maintained at a temperature of 250^{o}C. All Chromatographic separation were achieved using an HP-5MS capillary column, 30m x 250um ID and 0.25 film thickness. The carrier gas was helium at a constant flow rate of 1.1 ml/min. The temperature of the column was initially set at 85^{o}C for 0.3 min. It

was increased to 150°C (held for 4 min.) at a rate of 30°C/min., then to 185°C at a rate of 2°C min. and finally to 290°C(held for 5 min.) at a rate of 4°C/min. The GC was fitted with an autosampler 8200 and was programmed for injection 1-2ul. Ion Trap Detector was operated in Electron Impact Ionization mode (EI) at 70 eV and temperature at 220°C. EI Spectra were monitored by scanning ions within the range of 50-5000 amu. The computer which controlled the system had an EI-MS libraries (Willey spectral Library of more than 140 000 compound). The OPs compounds were identified by their full scan mass spectra and retention time using the total ion current as a monitor to give Total Ion Chromatogram (TIC). The use of the full scan mode allows to compare the spectrum obtained for interested OPs with the commercial EI-MS GC-ITD Libraries was obtained according to the methods described by [NBS (1988) and EPA / NIB, Heller and Miline (1980)]. In addition, Selective Ion Monitoring (SIM) mode was used for identification (mass spectra in elution time window were searched for 3 selective specific ions for the compound of interest, table 1), [safe and Hytizinger (1976) and Heller and Milne (1980)]. The most abundant ion for each compound was used for quantification and other ions were used to confirm the presence of the interested compounds (table-1).

Table - 1: Ions used for identification and quantification of OPs using GC-ITD.

No.	Compound	Molecular Weight (MW)	Molecular Formula	Ions, m/z*
1-	Mevinophos	224	C7H1306P	(127), 102, 109
2-	Ethoprophos	242	C8H1902PS2	(158), 43, 97
3-	Diazinon	304	C12H21N2O3PS	(179), 137, 152
4-	Parathion – methyl	263	C8H10NO5PS	(109), 125, 263
5-	Tetrachlorvinphos	364	C10H9Cl4O4P	(331), 329, 109
6-	Fenthion	278	C10H15O3PS2	(278), 125,109
7-	Fensulfothion	308	C11H17O4PS2	(293), 308, 141
8-	Azinophos – methyl	317	C10H12N3O3PS2	(160), 77,132
9-	Sulprofos	322	C12H19O2PS3	(140), 156, 322
10-	Coumaphos	362	C14H16ClO5PS	(109), 97, 362

* Ions between brackets were used for quantification

c- Quantification, linearity of GC-ITD response and calibration :

For quantification of OPs in extracts of water samples, a 1-2ul were injected into GC-ITD operated under conditions as described before. The concentration of each OP compound was determined from calibration curve (external standard method of calibration). The calibration curves (5 points), for all OPs under investigation were constructed using an OPs fortified ultra pure water at concentration levels, 0.01-10ug/L of each compound and applying the extraction and the analytical procedure as described before. The integrated peak areas obtained from the reconstructed Ion Chromatogram (RIC) (Total Ion Chromatogram,TIC) corresponding to the quantification ion (table -1)were ploted versus the concentrations which were used for fortification. In order to check the linearity of the calibration graphs Sigma Plot Software, versions 5 was used and the correlation of coefficient (R2) for each compound was calculated.

d.- Method detection limit (MDL) and limit of quantification (LOQ):

The MDL and LOQ were determined according to PAM (1994) and EPA (1994). The MDL was determined using the fortified water samples with OPs standards mixtures at the above mentioned extraction and analytical procedures. The MDL was calculated as the lowest concentration of OP which provides a chromatographic peak height in the GC-ITD Total Ion Chromatogram (GC-ITD-TIC) 3 times the average base line noise (at the same retention time) obtained from the GC-ITD-TIC of the extract of non-fortified water sample. (Blank). The limit of quantification (LOQ) was determined corresponding a value 10 times the back noise in blank GC-ITD-TIC.

e. Quality Control :

Several quality control measures were routinely used in this study and included running blanks with each sample set and analyzing samples as triplicates. Gas Chromatographic – MS conditions were monitored daily by checking the range of response factors of the calibration standards and the recoveries of a test standard that was included with each run.

RESULTS AND DISCUSSION

1. Analytical determination by GC-ITD:

The organophosphorus compounds chosen for this study are commonly applied for insect control in Egypt and Kuwait in agricultural and public health practice. It is well known that OPs insecticides are more soluble in water, [Fest and Schmidt (1982)] and may contaminate large areas of aquatic environment of surrounding treated fields specially in Egypt. The EEC (1988) and WHO (1993) have strict legislation about the occurrence of pesticide chemicals including OPs in water intended for human consumption, the maximum residual limit (MRL) must be below 100ng/L and sum of all not exceeded 500ng/L. Thus there is a need for multi residue analytical procedure for monitoring the residues of OPs in water. So, the aim of this work was to develop an efficient analytical procedures based on SPE using a GCB cartridge, Carbopack B for extraction of 10 OPs from water samples followed by GC - ITD as analytical tool. Before SPE studies began, analytical method for the final analytical determination of selected 10 OPs was developed using GC -I TD. GC - ITD conditions were optimized to separate the OPs studied using HP-5MS capillary column. A chromatogram of a standard mixture solution of OPs (0.1ug/L) using the conditions described in the Experimental Section is shown in figure 1. It is appearent that all OPs were properly separated in less than 40 min. All the compounds were identified by their full scan mass spectra and SIM mode. The use of the full scan mode allows to compare the spectrum obtained and commercial EI - MS libraries.

It is well known that in a multiresidue analysis, the components of the sample under investigation and the standard for this sample may not always be at the same concentration. Therefore to confirm that results can be extrapolated from one concentration to the other, the linearity of the GC-ITD response for all the OPs were studied in the range 0.01 – 10ug/L. The data found in table 2 showed that the GC-ITD responses to the different OPs under study is linear with the range 0.01-10ug/L and coorelation coefficient ranging from 0.997-to 0.999. These results indicated that GC-ITD is suitable as

analytical tool for multi residue analysis of selected 10 OPs in water samples.

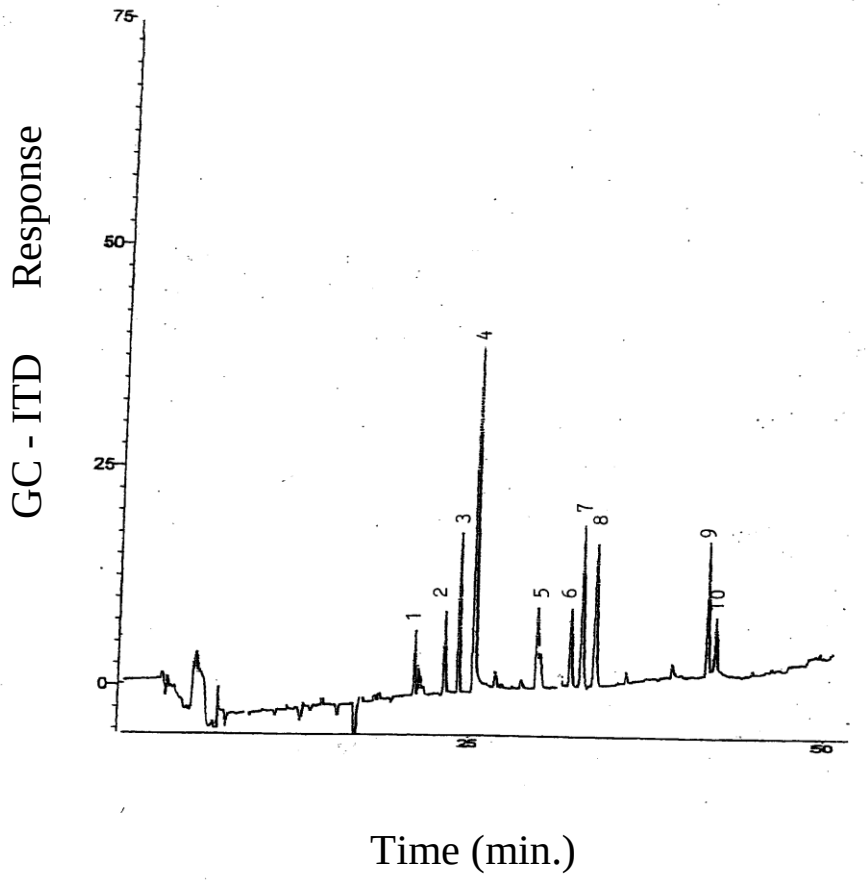

Figure- 1 : GC-ITD Total Ion Chromatogram (TIC) showing the separation of 10 OPs at 0.1ug/L (chromatographic conditions as explained in the text), peaks: 1=mevinophos, 2=ethoprophos, 3=diazinon, 4=parathion-methyl, 5=tertrachlorvinphos, 6=fenthion, 7=fensulfothion, 8=azinphos-methyl, 9=sulprofos 10=coumaphos.

Repeatability of GC-ITD response for each OP was determined from 5 repetitive injection of standard mixture solution (0.1ug/L) into GC-ITD operated under prescribed conditions. From the resulting peak areas for each OP in GC-ITD-TIC, the % RSD was calculated and expressed as repeatability of GC-ITD response for that compound. The data about repeatability are listed in table –2. It is apparent that % RSD for all tested OPs lower than 10.2. Therefore the repeatability achieved with the used GC-ITD apparatus was satisfactory according to the requirements were set by PAM (1994) and EPA (1994).

Data about method detection limit (MDL) and limit of quantification (LOQ) are found in table 2. From these data it is apparent that LOD and LOQ they were low enough to determine all tested OPs residues in water samples at the required levels set by the EEC (1988) and WHO (1993), for drinking and surface waters and below.

Table – 2 Repeatability, linearity, method detection limit (MDL) and limit of quantification (LOQ) for 10 OPs under study.

No.Compound	Repeatability (%RSD)	Linearity R2	MDL ng / L	LOQ ng /L
1- Mevinophos	7.8	0.997	14.0	50.0
2- Ethoprophos	5.2	0.999	15.0	52.0
3- Diazinon	7.7	0.998	20.0	65.0
4- Parathion – methyl	4.5	0.996	25.0	80.0
5- Tetrachlorvinphos	10.2	0.997	23.0	70.0
6- Fenthion	8.3	0.998	16.0	52.0
7- Fensulfothion	9.2	0.999	17.0	55.0
8- Azinophos – methyl	5.6	0.997	21.0	71.0
9- Sulprofos	9.3	0.981	22.0	70.0
10- Coumaphos	8.5	0.998	25.0	80.1

(1) Repeatability of GC-ITD response for standard mixture solution of 10 OPs at 0.1ug/L injected 5 times (expressed as % relative standard deviation, % RSD).

(2) Linearity of GC-ITD response for standards mixture of 10 OPs extracts obtained from fortified ultra pure water samples at different concentrations in the range of 0.01 – 10ug/L (expressed as correlation coefficient, R2).

2- Extraction on Carbopack B Cartridge (Recovery):

In this study trace amounts of OPs in water samples were enrichment on Carbopack B cartridge, (60-80 mesh). Process of the enrichment was controlled by systematic recovery experiments. For this purpose ultra pure water (1 liter) was fortified with OPs standard mixture at 2 concentration levels 0.1ug/L and 0.5ug/L, and each concentration was replicated 3 times. Prior to the extraction of the fortified water samples, the Carbopack B cartridges were conditioned by passing sequently 10ml. of ethylacetate, 10ml of methanol followed by 20ml of ascorbic acid solution pH2. As mentioned by Crescenzi *et al*. (1995), ascorbic acid was used to convert quinines present on the GCB surface to less reactive hydroquinone (improve desorption process during SPE extraction). The mean recoveries (%) as well as the relative standard deviation (%) for all investigated OPs are listed in table 3. As can seen the recoveries obtained were satisfactory, above 90% with % RSD ranging from 4.5 to 10.1%.

From all previously data it can be concluded that the developed analytical procedures demonstrates satisfactory recoveries, low detection and quantification limits, good reproducibility (repeatability = precision) and accuracy. Further more, the main future of the method include extraction of OPs (more polor) using disposal GCB cartridge (Carbopack-B) with substantial saving of glass wares, reagents and time compared with liquid – liquid extraction [Miliadis (1993)]. All these favorable analytical features of the developed analytical procedures allowed its application for determination of the selected OPs residues in water samples at the levels of maximum residue levels were set by EEC (1988) and WHO (1993) for surface and drinking water.

Table - 3 : Mean recoveries (%) and relative standard deviation (% RSD) for all 10 OPs extracted from fortified water samples using carbopack B cartridge.

Sl.No.	Compound	Levels of Fortification Ug/L	% Recovery	% RSD
1	Mevinophos	0.1	92.3	6.4
		0.5	93.4	6.5
2	Ethoprophos	0.1	93.2	4.7
		0.5	95.2	7.7
3	Diazinon	0.1	100.4	8.5
		0.5	98.2	7.5
4	Parathion- methyl	0.1	91.1	8.1
		0.5	90.5	9.2
5	Tetrachlorvinphos	0.1	94.5	10.1
		0.5	95.6	9.1
6	Fenthion	0.1	99.1	6.5
		0.5	95.2	5.5
7	Fensulfothion	0.1	95.8	4.5
		0.5	96.7	4.7
8	Azinophos methyl	0.1	103.8	6.7
		0.5	102.9	7.5
9	Sulprofos	0.1	91.3	8.1
		0.5	92.5	9.1
10	Coumaphos	0.1	94.9	7.6
		0.5	95.6	6.6

*Each value is the mean of 3 replicates.

3- Application of method developed:

To determine the suitability of the proposed analytical procedures for its application for multi residue analysis of selected 10 OPs in surface and drinking waters, samples fortified (at 0.1ug/L of the mixture of tested OPs) and without fortification were extracted and analyzed using the same method. Surface water samples (2 liters) were collected from irrigation drain No.7 (in which residue of some insecticides have been detected previously by Khalifa *et al* (2000) at Khafer El Sheikh governorate, Egypt (agricultural area). Drinking water samples (2 liters) were collected from tap water at College of

Health Sciences, Kuwait and at Faculty of Agriculture, at Kafer EL-Sheikh, Egypt. Upon arrival at the laboratory, the collected water samples were filtered through a fiber glass filter. Each filtered water sample were divided into two identical volumes (1 liter). The first sample (analytical sample) was used to quantify the targeted OPs, whereas the second (fortified) was used to calculate the percent recoveries of the targeted OPs. Data on the recoveries of the tested OPs from investigated water samples are listed in table 4. The obtained percentage of recoveries are satisfactory, above 87% with %RSD ranging from 4.5 to 10.5% less recoveries obtained from surface waters can be associated with presence of the organic matter in natural water (surface waters), which could affect the sorbent efficiency during SPE processes [Molto *et al*, (1991)].

Evaluation for Total Ion Chromatogram generated from GC-ITD analysis of fortified water samples (surface and drinking waters) showed that all tested OPs can be detected and identified at 0.1ug/L. Detection of tested OPs in fortified surface water samples at 0.1ug/L is lower than the EEC (1998) limit (3ug/L). So practically the proposed analytical procedures are sensitive for multi residue analysis of selected OPs in surface and drinking waters.

Table.4 : Mean recoveries (%R) and relative standard deviation (RSD) for 10 OPs extracted from different water samples fortified at 0.1 ug/L.

Sl.No	Compound	Kuwait drinking Water		Egypt drinking Water		Surface Water	
		% R	% RSD	% R	%RSD	%R	%RSD
1	Mevinophos	93.2	7.5	91.2	8.5	88.1	8.2
2	Ethoprophos	94.2	6.5	92.3	4.5	87.0	9.1
3	Diazinon	95.3	8.6	94.2	6.5	90.1	8.2
4	Parathion methyl	96.4	7.6	96.6	6.7	91.2	10.1
5	Tetrachlorvinph	97.2	8.5	98.6	4.5	87.8	8.1
6	Fenthion	97.3	6.5	98.3	8.5	93.2	9.3
7	Fensulfothion	99.2	7.5	101.2	8.5	88.5	10.2
8	Azinophos -methyl	102.1	8.5	99.2	10.5	90.5	7.1
9	Sulprofos	91.2	7.5	98.3	9.5	93.5	10.3
10	Coumaphos	98.2	8.5	99.1	6.5	94.5	9.5

* Each Value is the mean of 3 replicates.

The GC-ITD Total Ion chromatogram obtained from the analysis of the extract of surface water sample without fortification is illustrated in figure 2a. As shown in this figure the surface water sample is positive for 2 OPs, fensulfothion and azinophos-methyl at concentrations of 0.8 and 1.2 ug / L, respectively. Figures 2b, and 2c present the Total Ion Chromatograms of the extracts of drinking water samples collected from Egypt and Kuwait and analyzed by GC-ITD. It can be seen that there is no identified peaks related to the tested OPs on the chromatogram (the concentration of targeted OPs are below our MDL, >25 ng/L.

Conclusions

Application of developed method for analysis of the selected 10 OPs residues in environmental water samples based on solid phase extraction using a graphitized carbon black, Carbopack B, Cartridge followed by GC-ITD with good determination limits. Multi residues for studied OPs in water at maximum residual limit, 100ng/L can be determined with high sensitivity, precision and accuracy. Although the actual maximum residual levels (MRL) for individual pesticides are now set at 100ng/L, for drinking water (EEC, 1988 and WHO 1993), levels as low as 14 to 25ng/L can be determined by the proposed method. The proposed analytical procedure was applied successfully for analysis of water samples from Egypt and Kuwait. Finally the developed analytical procedure can be use as a routine method in monitoring programs in Egypt and Kuwait to determine residue levels of the studied 10 OPs in drinking and surface water.

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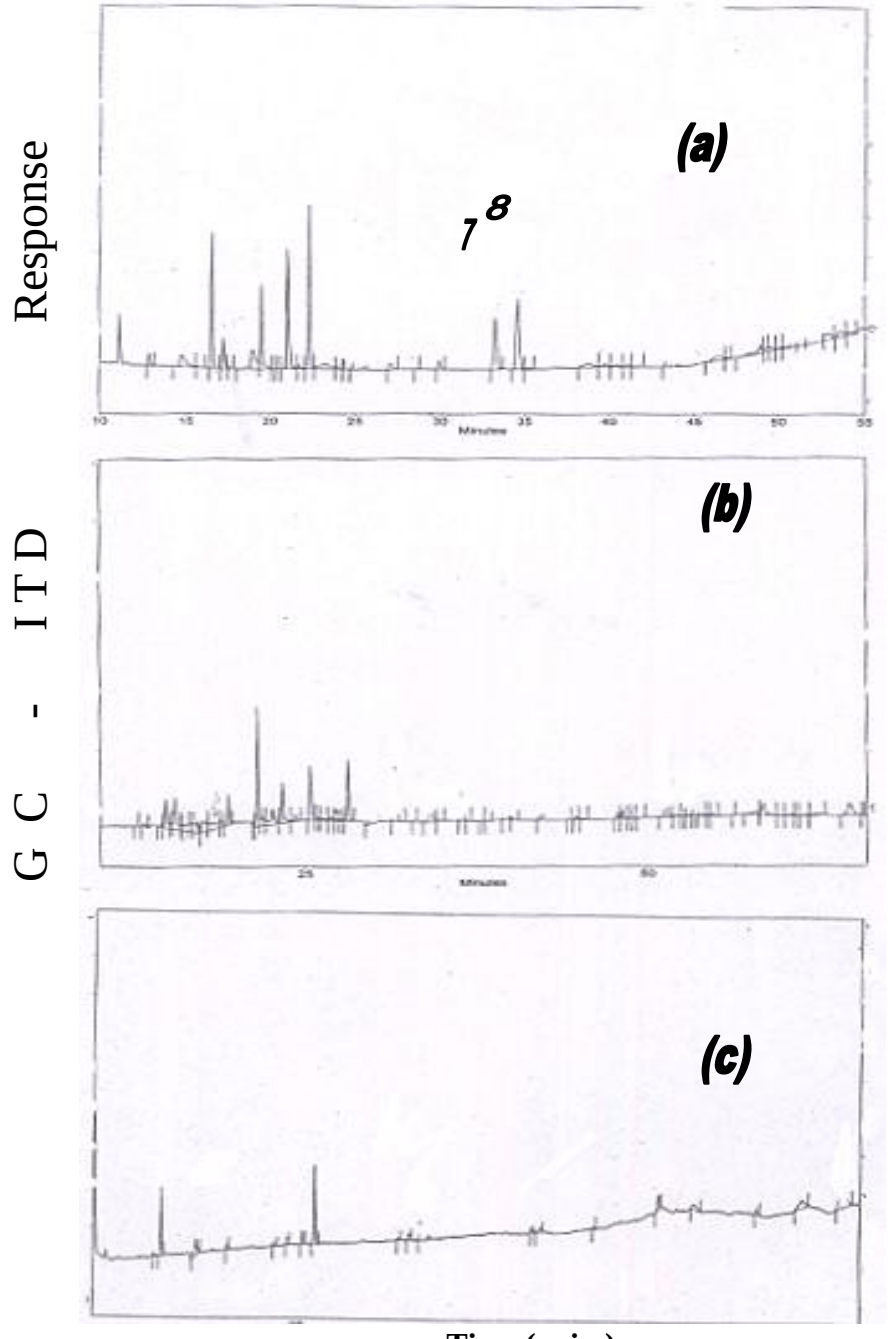

Figure-2 : **GC ITD Total Ion Chromatogram obtained from 1 Liter of water samples extracted and analyzed according to the proposed analytical procedures. Numbers of the peaks indicates the compounds listed in table 1. (a) surface water (b) drinking water from Egypt, (c) drinking water from Kuwait.**

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الملخص العربي

تحليل متبقيات عشرة من المبيدات الفوسفورية العضوية في الماء باستخدام تقنية الوجه الصلب للاستخلاص وكروماتوجرافيا الغاز – مطياف الكتلة

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تمثل المبيدات الفوسفورية العضوية احد أهم الملوثات الكيمائية العضوية للوجه المائي في البيئة . لذلك وضع الاتحاد الأوربي ومنظمة الصحة العالمية حدود قصوي لتواجد تلك المبيدات (الملوثات) في البيئة المائية (1و 0 ميكروجرام / لتر في ماء الشرب ، 1-3 ميكروجرام / لتر في الماء السطحي الطبيعي). مما لا شك فيه وجود هذا المبيدات بتركيزات اعلي من الحدود المسموح بها يمثل خطورة على صحة البيئة المائية وبالتالي على صحة الإنسان . حفاظا علي صحة الإنسان يجب دراسة مدي تلوث البيئة المائية بتلك الملوثات . وهذا يحتاج الي وجود طرق تحليل سهلة وبسيطة ذات كفاءة ودقة عالية . لذا يصف هذا البحث طريقة تحليلية سهلة ذات كفاءة ودقة عالية تعتمد على تقنية الوجه الصلب للاستخلاص (استخدام الفحم الأسود النشط كمادة دامصة) في استخلاص عشرة (10) من المبيدات الفوسفورية العضوية الملوثة للماء واستخدام كروماتوجرافيا الغاز – مطياف الكتلة كوسيلة تحليل متخصصة لها درجة حساسية ودقة عالية .

أوضحت نتائج هذا البحث انه يمكن الاعتماد على هذه الطريقة في تحليل عشرة من المبيدات الفوسفورية العضوية الملوثة للماء وهي .

mevinphos, ethoprophos, diazinon, parathion-methyl, tertrachlorvinphos, fenthion, fensulfothion, azinphos-methyl, sulprofos and coumaphos

ودلت النتائج على أن اقل تركيز يمكن تقديره لهذه المبيدات في الماء (حساسية الطريقة) كان 14 -25 نانو جرام / لتر . أي أن هذه الطريقة تفي بالاحتياجات المطلوبة من قبل الهيئات الدولية المعنية بالحفاظ علي صحة الإنسان والبيئة من مخاطر تلك الملوثات هذه الطريقة التحليلية موضوع البحث سهلة موفرة للوقت يمكن تطبيقها في الرصد الدوري لهذه الملوثات في البيئة المائية في كل من جمهورية مصر العربية ودولة الكويت .