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Determination of Organochlorine Pesticide (OCPs) in Shallow Observation Wells from El-Rahawy Contaminated Area, Egypt

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The contamination of organochlorine pesticides (OCPs) from the selected sites in El Rahawy area was investigated to estimate the current status of pollution in surface and groundwater. A study was conducted to determine the concentrations of OCPs in surface and groundwater samples along El Rahaway drain. Samples were collected from six different sites during the rainy and dry seasons. The samples were extracted by liquid-liquid extraction method then screened and determined qualitatively for 18 OCPs using GC/ECD (Gas chromatograph equipped with electron capture detector). There was a variation of pesticide residue levels with season. The commonly found OCP residues in the study area were α-HCH, γ-HCH, heptachlor, heptachlor epoxide, endosulfan I, endosulfan II, p,p'-DDE, p,p'-DDD and endrin. The overall results showed that surface water was more polluted with OCPs than groundwater, especially endosulfan I which was detected in the wide range concentration of 0.021 to 0.375 μg L^{-1} and 0.083 to 0.823 μg L^{-1} during dry and rainy seasons, respectively. There was a variation of pesticide residue levels with season. The OCPs levels in all water samples were generally exceeded Canadian water quality guidelines for the protection of agricultural water uses (CWQGs).

Keywords: *Organochlorine pesticides (OCPs), residues, water, El Rahaway drain, Egypt.*

1. Introduction

Organochlorine pesticides (OCPs) are categorized as a group of persistent organic pollutants (POPs), which most of these compounds have been prohibited from use due to their toxic effects (Zhou et al., 2006). OCPs are a common name of a group of pesticides consisting of benzene and chlorine (Pandit et al., 2005). Organochlorines are grouped into 3, namely: dichlorodiphenyl ethane (eg, DDT, DDD and DDE), cyclodiene (example: aldrin, dieldrin, heptachlor and endosulfan), and chlorocyclohexane (eg, α, β, γ and δ-HCH).

In Egypt, all types of OCPs have been banned since late 1990, after being used for more than 50 years for agriculture and public health reasons. However due to its cheap price, easy to use, and effectively eradicate pests, some kind of OCPs such as DDT and γ-HCH (lindane) are still used in Egypt, coupled with a lack of law enforcement (Nasr et al., 2009).

Environmental contamination by OCPs in water bodies have been a great concern, since most of these pesticide compounds are very persistent, bioaccumulative and their toxicity can pose harmful effects to human and ecosystems, because of these compounds are lipophilic and have low chemical and biological degradation rates (Barakat et al., 2002).

A potential pathway for adverse effects of pesticides is through hydrologic systems, which supply water for both humans and natural ecosystems. Water is one of the primary ways pesticides are transported from an application area to other locations in the environment. Pesticide contamination of groundwater is especially acute in rural agricultural areas where over 95 percent of the population relies upon groundwater for drinking. The organochlorine contamination pathways to water bodies are likely to be nonpoint sources via runoff, atmospheric deposition, and leaching due to agricultural applications, vector pest control and improper waste disposal methods (Carvalho et al., 1996; Galindoet al., 1999).

El-Rahawy drain is one of the major drains of Egypt, which is considered the main source of contamination at The Nile River. El-Rahway drain receives all sewage of El-Gieza governorate in addition to agricultural and domestic wastes of El-Rahway village and discharged these wastes directly without treatment into Rosetta branch (El Bouraie et al., 2010).

The object of the research is determination the residues of OCPs in two surface water samples collected along El-Rahawy drain and in four groundwater samples collected from different water levels throughout El-Rahawy drain basin. The present study, therefore, looked at the distribution and characterized the possible sources of OCPs in water bodies.

2. Material and Methods

The study was conducted at Central Laboratory for Environmental Quality Monitoring and the Chemistry Department of Central Water Quality Laboratory, Greater Cairo Water Company from January to June 2010.

All chemicals and reagents used in this study were of high purity quality and were of analytical grade. n-Hexane and dichloromethane of special grade for pesticide residue analysis were purchased from Sigma-Aldrich, Germany. Organic solvents particularly dichloromethane which is toxic, were handled with care observing safety precautions, using efficient fume hoods and wearing protective gloves. Other materials used throughout the experimental procedure, such as cotton wool, filter paper and anhydrous sodium sulphate (Na_2SO_4) from Merck, Germany. Silicagel (60-100 mesh ASTM) was purchased from Merck, Germany. The individual reference pesticide standards (ISO 9001Certified) used for GC analysis of the organochlorines was purchased from Dr. Ehrenstorfer GmbH of Augsburg in Germany. A standard solution of each OCP was prepared in a proper way depending on being solid or liquid, to give a 100 µg mL^{-1} stock solution in n-hexane, which was stored at -20°C in glass bottles with PTFE-faced screw caps. Dilutions were prepared from the stock solutions and stored in the refrigerator at +4°C. A standard mixture solution containing all 18 pesticides was prepared with the appropriate concentrations of each pesticide, and stored at -20°C. For qualitative and quantitative interpretation of results, a concentration of 1.0 µL mixture of OCPs was used as internal standard for OCPs standard mixture and in the real sample final solutions (Abbaccy et al., 2003).

The gas chromatograph used (Hewlett Packard, 5890 series II, with its required accessories including Hp-chemistation software) was equipped with an Electron Capture Detector (ECD) and a fused silica capillary column (length of 6 m, 0.25 mm I.D. and 0.25 µm film thickness), operated as mentioned elsewhere. An ultrasonic bath (BANDELIN electronic, Germany), a mechanical shaker (Edmund Bühler, Germany), and a rotary evaporator (Janke and Kunkel, IKA-Lab., Germany) were used (Fatoki and Awofolu, 2003).

El Mouheet drain in Giza is considered one of the most polluted main drains and has one main branche: the 70.2 km El-Rahaway drain from the beginning at El-Badrasheen. El-Rahaway drain starts at Rahawy Pump Station on Mansouria Rayah lies at 30 Km, North to Cairo at El-Kanater El-Khayria area, Egypt. El-Rahawy drain lies between latitudes 30º 10' N to 30º 12' N and longitudes 31º 2' E to 31º 3' E as shown in Figure 1. El-Rahawy drain is about 12.41 km^2 with an average length of 4.5 Km. El-Rahawy drain passes through El-Rahway village and many villages dotted along it receiving agricultural and domestic wastes in addition to sewage of El-Gieza governorate and discharged these wastes directly without treatment into the Nile (Rossetta Branch).

Two types of water samples were collected from El-Rahawy drain area, namely, surface water samples (collected manually) from 2 different sites along El-Rahawy drain and groundwater samples were collected from Hand pump on the observation wells which located beside El-Rahawy drain using clean glass containers (1.5 liter capacity), stored in the refrigerator at + 4 °C and extracted within 24 hours as shown in (Fig. 1) and illustrated in (Table 1) during rainy and dry seasons, from January to June 2010.

Liquid-liquid extraction was used for the extraction of OCPs residues from water samples. One L of each water sample was extracted with 60 ml dichloromethane in a 2-L separatory funnel. The mixture was shaken manually for 5 min, followed by collection of the lower organic layer. The extraction was repeated twice each time with 60 ml dichloromethane. The pooled 180-ml dichloromethane extracts were dried over anhydrous sodium sulfate and filtered. The solvent was evaporated to dryness under vacuum at ≤40˚C and 350 mbar. The residues were dissolved in 1 ml n-hexane containing 1 µL as internal standard.[10]

Calibration curves were prepared from a stock solution of 10.0 mg L^{-1} OCPs dissolved in hexane by serial dilution to reach calibration concentrations of 5, 10, 20, 40 and 50 µg L^{-1}. Each calibration solution was analysed in threefold by GC-ECD. The peak areas of the corresponding analytes were plotted against the calibration concentrations and the regression coefficient was calculated reaching a mean of $r^2 = 0.9993$ for all analytes. The minimum detection limits of the methods used for extraction of OCPs residue from water is 0.01 ng L^{-1} (Rezaee et al., 2006).

The retention times obtained for the components of the mixture are based on a signal-to-noise ratio of 3:1, the retention times were as follows: α-HCH (11.511 min), γ-HCH (13.288 min), heptachlor (14.514 min), aldrin (16.215 min), β-HCH (16.38 min), δ- HCH (17.311 min), heptachlor epoxide (18.221 min), endosulfan I (19.282 min), p,p'-DDE

(20.145 min), dieldrin (20.721 min), endrin (21.523 min), p,p'-DDD (23.112 min), endosulfan II (23.337 min), p,p'-DDT (23.887 min), endrin aldehyde (25.037 min), methoxychlor (26.597 min), endrin ketone (26.786 min) and endosulfan sulfate (28.824 min).

A Hewlett-Packard 5890 series II GC with ECD and HP-A1773 (length of 6 m, 0.25 mm I.D. and 0.25 µm film thickness) capillary column was used with helium as the carrier gas and nitrogen as auxiliary gas. Conditions of the GC were: injector temperature 250˚C; detector temperature 320 ˚C; oven temperature 90˚C; initial temperature 90˚C; initial time 2 minutes; ramp 1, 30˚C min^{-1}; temperature 1, 180 min^{-1}; time 1, 0.0 minute; ramp 2, 30˚C min^{-1}; temperature 2, 270 ˚C, time 2, 0.0; final time 35 minutes; purge time 0.75 minutes; injection split-splitless (Fatoki and Awofolu, 2003).

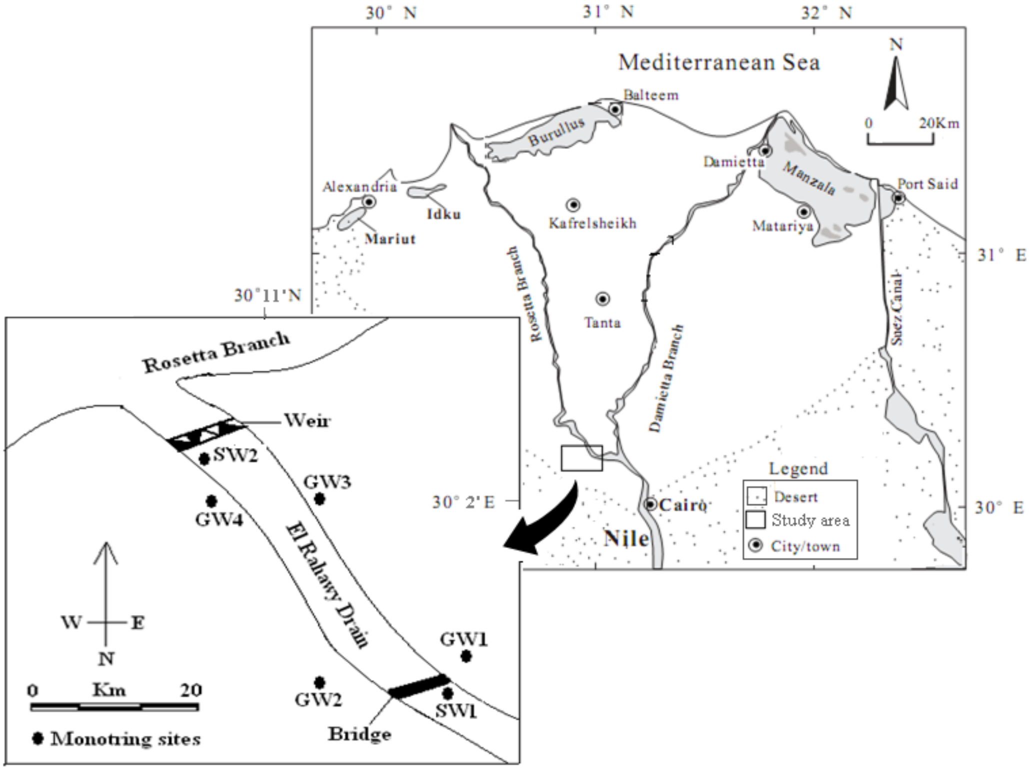

Fig. 1. Map Showing Sampling Locations at El Rahawy Drain

Table 1. Locations and description of the surface and groundwater collected

Site Code	Site Name	Location	
		Latitude (˚N)	Longitude (˚E)
SW1	El-Rahawy drain at 3 km (south Rosetta branch)	30˚ 11' 13.26"	31˚ 02' 52.84"
SW2	El-Rahawy drain at 0.5 km (south Rosetta branch)	30˚ 12' 15.76"	31˚ 02' 04.02"
GW1	Hand pump east El Rahawy drain at 2.8 km (south Rosetta branch).	30˚ 11' 14.36"	31˚ 02' 53.19"
GW2	Hand pump west El Rahawy drain at 2.7 km (south Rosetta branch).	30˚ 11' 13.52"	31˚ 02' 50.85"
GW3	Hand pump east El Rahawy drain at 0.9 km (south Rosetta branch).	30˚ 12' 02.97"	31˚ 02' 12.35"
GW4	Hand pump west El Rahawy drain at 0.6 km (south Rosetta branch).	30˚ 12'09.47"	31˚ 02' 05.57"

3. Results and Discussion

The results obtained from a comprehensive study of 18 OCPs residue in surface and groundwater samples collected from study area. Noteworthy that chromatogram of OCPs residues in the standard sample is illustrated in (Fig. 2).

Concentration of total OCPs in water samples of the current study during Rainy season varied from 0.3404 to 2.1567 µg L^{-1} and 0.006 to 0.152 µg L^{-1} for the surface and groundwater, respectively.

HCHs are considered as the less persistence OCPs. Based on the average values of contribution of each isomer, they can be arranged according to the following descending order: γ-HCH > α-HCH > β-HCH > δ-HCH. Thus, we can conclude that γ -isomer was the most dominant isomer of HCHs in water samples of the study area during winter season. A maximum (0.225 µg L^{-1}) of γ-HCH was recorded at SW2; while a minimum value BDL (below the detection limit 0.01 ng L^{-1}) was recorded at GW3 and GW4.

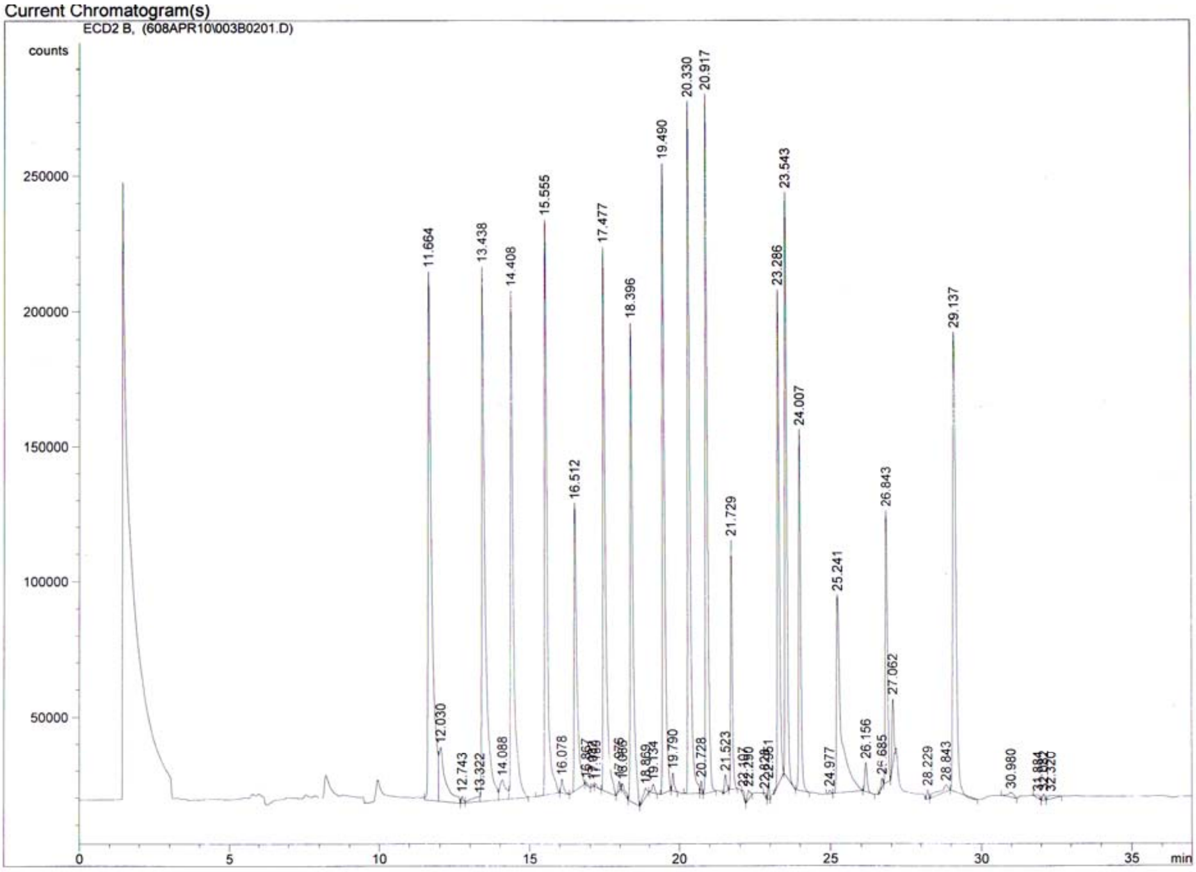

Fig. 2. GC/ECD Chromatogram of standard OCPs (0.05 µg L^{-1}).

Table 2. Concentrations of OCPs residues in water during Rainy Season (µg L^{-1})

Items	SW1	SW2	GW1	GW2	GW3	GW4
p,p'- DDT	0.029	0.064	0.003	0.003	0.004	0.049
p,p'- DDE	0.051	0.084	BDL	BDL	0.001	0.009
p,p'- DDD	0.048	0.091	BDL	BDL	BDL	0.020
ΣDDTs	0.128	0.239	0.003	0.003	0.005	0.078
α- HCH	0.0016	0.008	BDL	BDL	BDL	BDL
β- HCH	BDL	0.001	BDL	BDL	BDL	BDL
γ- HCH	0.042	0.225	0.003	0.005	BDL	BDL
δ- HCH	BDL	BDL	BDL	BDL	BDL	BDL
ΣHCHs	0.0436	0.234	0.003	0.005	0.00	0.00
Aldrin	BDL	BDL	BDL	BDL	BDL	BDL
Dieldrin	0.001	0.001	BDL	BDL	BDL	BDL
Endrin	0.0038	0.0087	BDL	BDL	BDL	0.008
Endrin aldehyde	0.001	0.001	BDL	BDL	BDL	BDL
Endrin ketone	BDL	BDL	BDL	BDL	BDL	0.066
Heptachlor	0.018	0.148	BDL	BDL	BDL	BDL
Heptachlor epoxide	0.06	0.7	BDL	BDL	BDL	BDL
Endosulfan I	0.083	0.823	BDL	BDL	BDL	BDL
Endosulfan II	0.001	0.001	BDL	BDL	0.001	BDL
Endosulfan sulfate	0.001	0.001	BDL	BDL	BDL	BDL
Methoxychlor	BDL	BDL	BDL	BDL	BDL	BDL
ΣCyclodienes	0.1688	1.6837	0.00	0.00	0.001	0.074
ΣOCPs	0.3404	2.1567	0.006	0.008	0.006	0.152

BDL: below the detection limit

Concentration of ΣCyclodienes (aldrin, dieldrin, endrin, endrin aldehyde, endrin ketone, heptachlor, heptachlor epoxide, methoxychlor, endosulfan I, II and endosulfan sulfate) varied from 0.00 to 1.6837 µg L^{-1} and from 0.00 to 0.074µg L^{-1} for the surface and groundwater, respectively. As a result, the following interpretation and discussion focused on OCPs residue in surface and groundwater are shown in (Table 2) and illustrated in Figs (3-8)

Endrin is an alicyclic chlorinated hydrocarbon and is rapidly converted to the epoxide form (endrin aldehyde and endrin ketone). The presence of a value recorded during winter season, declares that there is a renewal source of endrin in surface and groundwater.

Concentration of ΣDDTs showed variation from 0.00 to 1.126 µg l^{-1} and from 0.003 to 0.049 µg L^{-1} for the surface and groundwater, respectively (Table 2). In the environment, DDT can be degraded by solar radiation or metabolised in organisms. Dehydrochlorination of DDT gives its metabolite DDE (Mladen, 2000). This is supported by the presence of a maximum (0.084 µg L^{-1}) of p,p'-DDE recorded at SW2.

The p,p'-DDE is the most dominant pesticides which followed by p,p'-DDT and finally p,p'-DDD during winter season. Table (2) indicates that DDTs are the major pollutant pesticides followed by HCHs and then cyclodienes compounds during winter season.

Generally, El-Rahawy drain is more polluted by OCPs than those in the water courses, thus may be as a result from rejecting pollutants directly in the drain or the aquifers and are usually the results routine activities or accidental events to these in a good agreement with as reported by (El-Barbary et al., 2008).

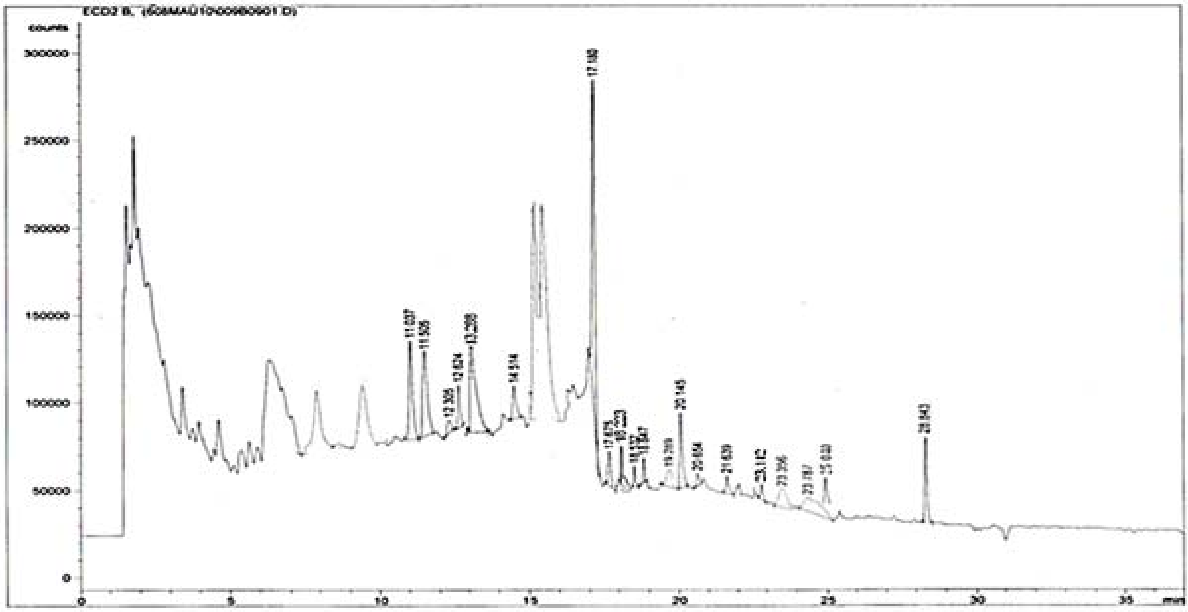

Fig. 3. GC/ECD Chromatogram of OCP residues for surface water sample in Rainy Season at SW1

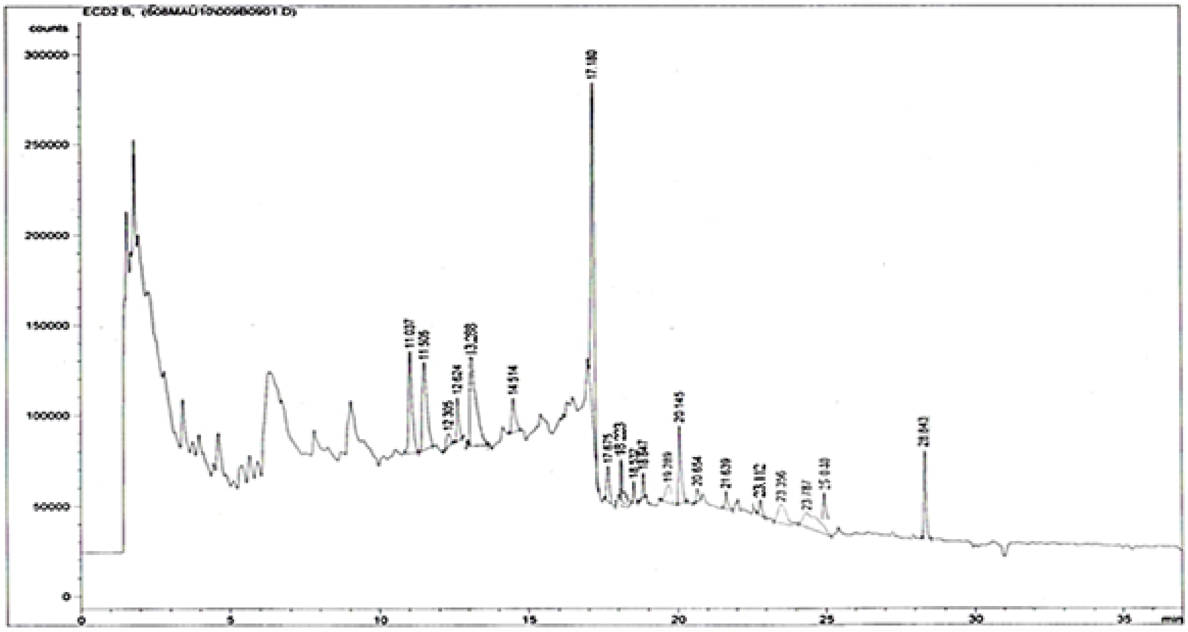

Fig. 4. GC/ECD Chromatogram of OCP residues for surface water sample in Rainy Season at SW2

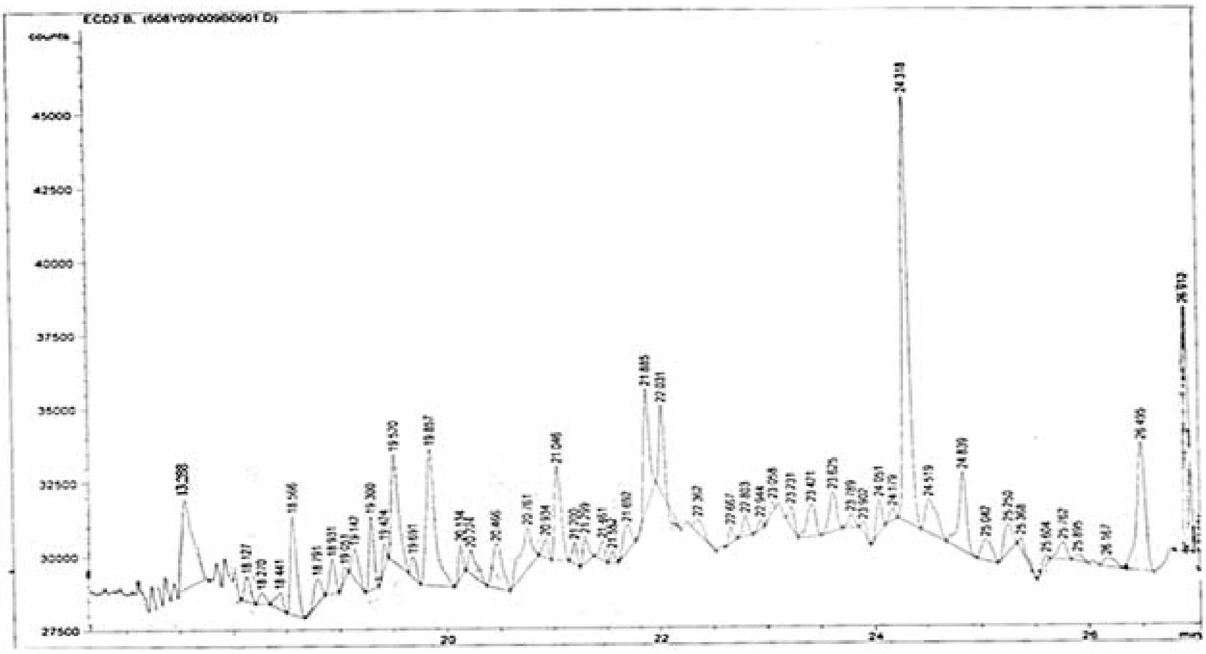

Fig. 5. *GC/ECD Chromatogram of OCP residues for groundwater sample in Rainy Season at GW1*

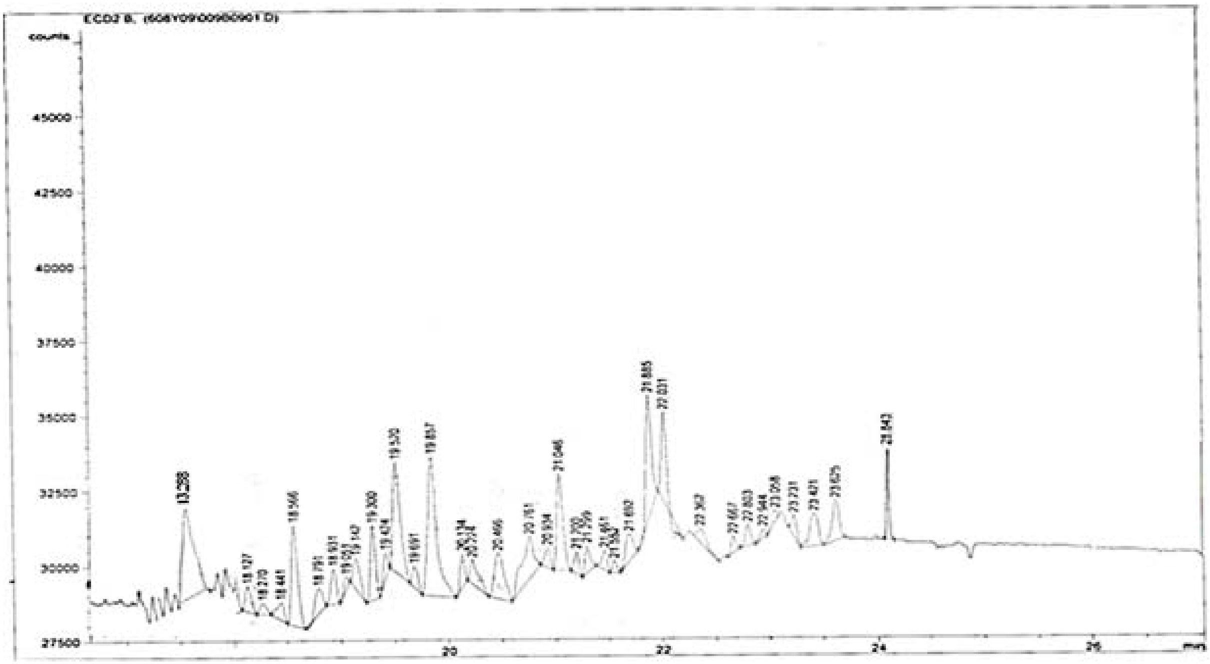

Fig. 6. *GC/ECD Chromatogram of OCP residues for Water sample in Rainy Season at GW2*

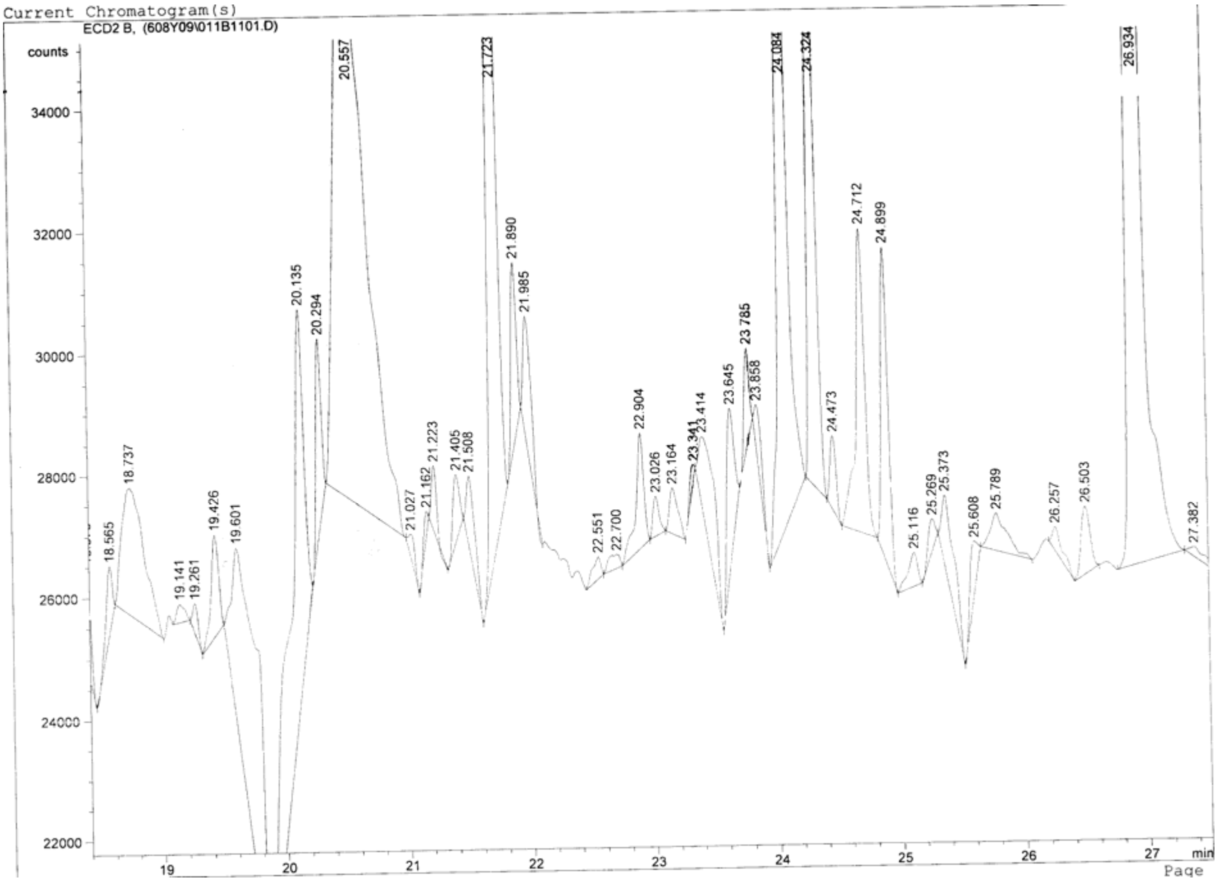

Fig. 7. Chromatograms of OCP residues for Water sample in Rainy Season at GW3

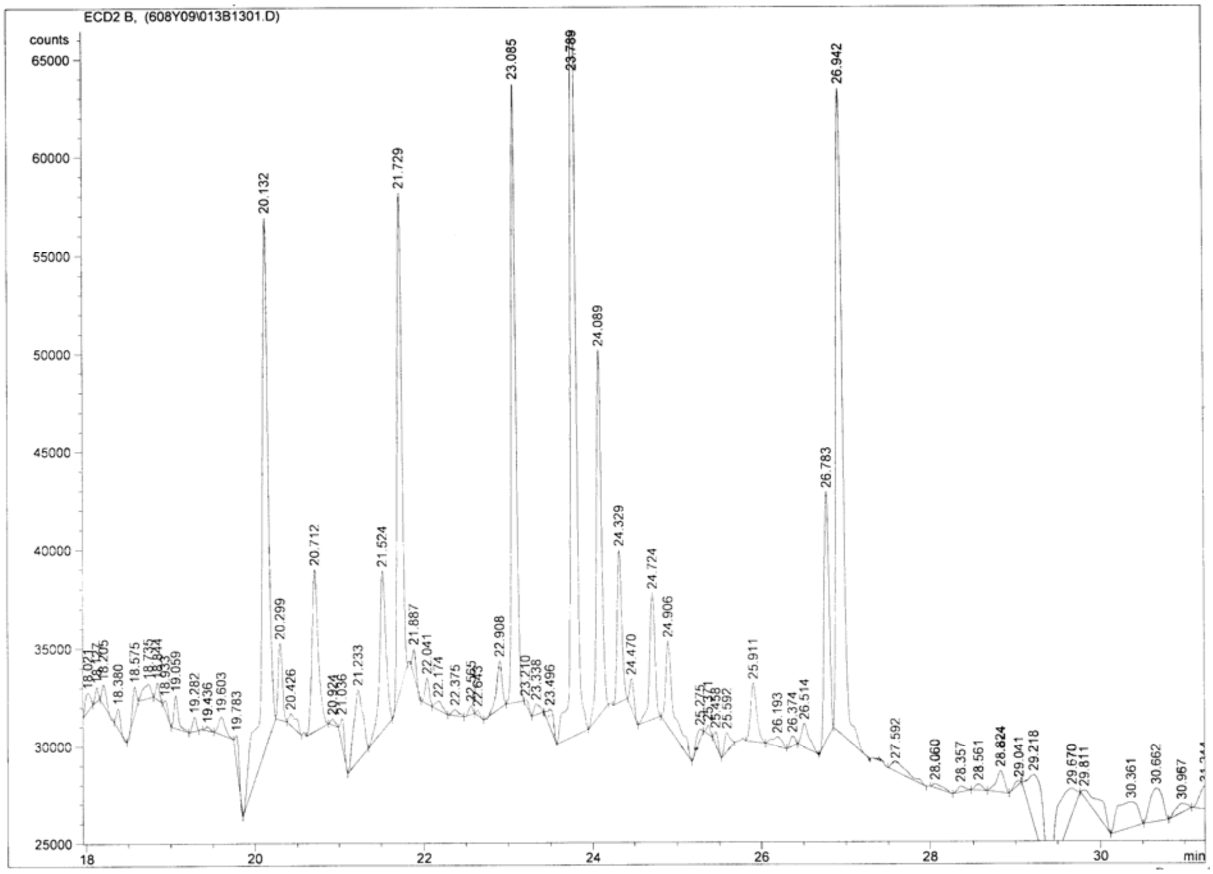

Fig. 8. GC/ECD Chromatogram of OCP residues for Water sample in Rainy Season at GW4

In all analyzed water samples, none OCPs are detected (below the detection limit 0.01 ng L^{-1}) as shown in Figs (9-11) and tabulated in Table 3, although they are used in agricultural purposes. This can be attributed to the fast rate of degradation of this class of pesticides that was accelerated through the variation of climatic conditions in the study area. OCPs, especially DDT, were used intensively during past years; therefore, it is still detected with its metabolites (DDE and DDD) in low concentrations in El-Rahawy drain. This can be related to the currently flushing processes, its use during the past years, low rate of application and attachment to the sediments along their flow as they are associated with solid phase or due to its low solubility and low photo-oxidation (Dubus et al., 2000). The HCHs concentrations have lower values than DDTs' in water. Because of their differences in physico-chemical and biological properties, having HCHs a higher water solubility, vapor pressure, biodegradability, lower lipophilicity and particle affinity as compared to DDTs properties (Tang et al., 2007). Therefore, the HCHs concentrations are found in low concentration under the different flow conditions.

OCs residues seep from different agricultural, domestic and industrial usage in the study area into drains, irrigation water and finally into pose serious environmental and health risks. It is worth mentioning that, this phenomenon could also happen even under controlled application methods, which is not always the case in Egypt. This leaching mainly depends on the type of pesticide, soil characteristics, hydrogeological conditions, climatic factors, agro-technical factors, and human factors.

Table 3. Concentrations of OCPs residues in water samples during Dry Season (µg L^{-1})

Items	SW1	SW2	GW1	GW2	GW3	GW4
p,p'- DDT	BDL	0.011	BDL	BDL	BDL	BDL
p,p'- DDE	0.006	0.028	BDL	BDL	BDL	BDL
p,p'- DDD	0.002	0.027	BDL	BDL	BDL	BDL
Σ DDTs	0.008	0.066	0.000	0.000	0.000	0.000
α- HCH	BDL	BDL	BDL	BDL	BDL	BDL
β- HCH	BDL	BDL	BDL	BDL	BDL	BDL
γ- HCH	0.006	0.1	BDL	BDL	BDL	BDL
δ- HCH	BDL	BDL	BDL	BDL	BDL	BDL
Σ HCHs	0.006	0.1	0.000	0.000	0.000	0.000
Aldrin	BDL	BDL	BDL	BDL	BDL	BDL
Dieldrin	BDL	BDL	BDL	BDL	BDL	BDL
Endrin	BDL	BDL	BDL	BDL	BDL	BDL
Endrin aldehyde	BDL	BDL	BDL	BDL	BDL	BDL
Endrin ketone	BDL	BDL	BDL	BDL	BDL	BDL
Heptachlor	BDL	0.065	BDL	BDL	BDL	BDL
Heptachlor epoxide	0.01	0.35	BDL	BDL	BDL	BDL
Endosulfan I	0.021	0.375	BDL	BDL	BDL	BDL
Endosulfan II	BDL	BDL	BDL	BDL	BDL	BDL
Endosulfan sulfate	BDL	BDL	BDL	BDL	BDL	BDL
Methoxychlor	BDL	BDL	BDL	BDL	BDL	BDL
ΣCyclodienes	0.031	0.79	0.000	0.000	0.000	0.000
ΣOCPs	0.07	0.977	0.000	0.000	0.000	0.000

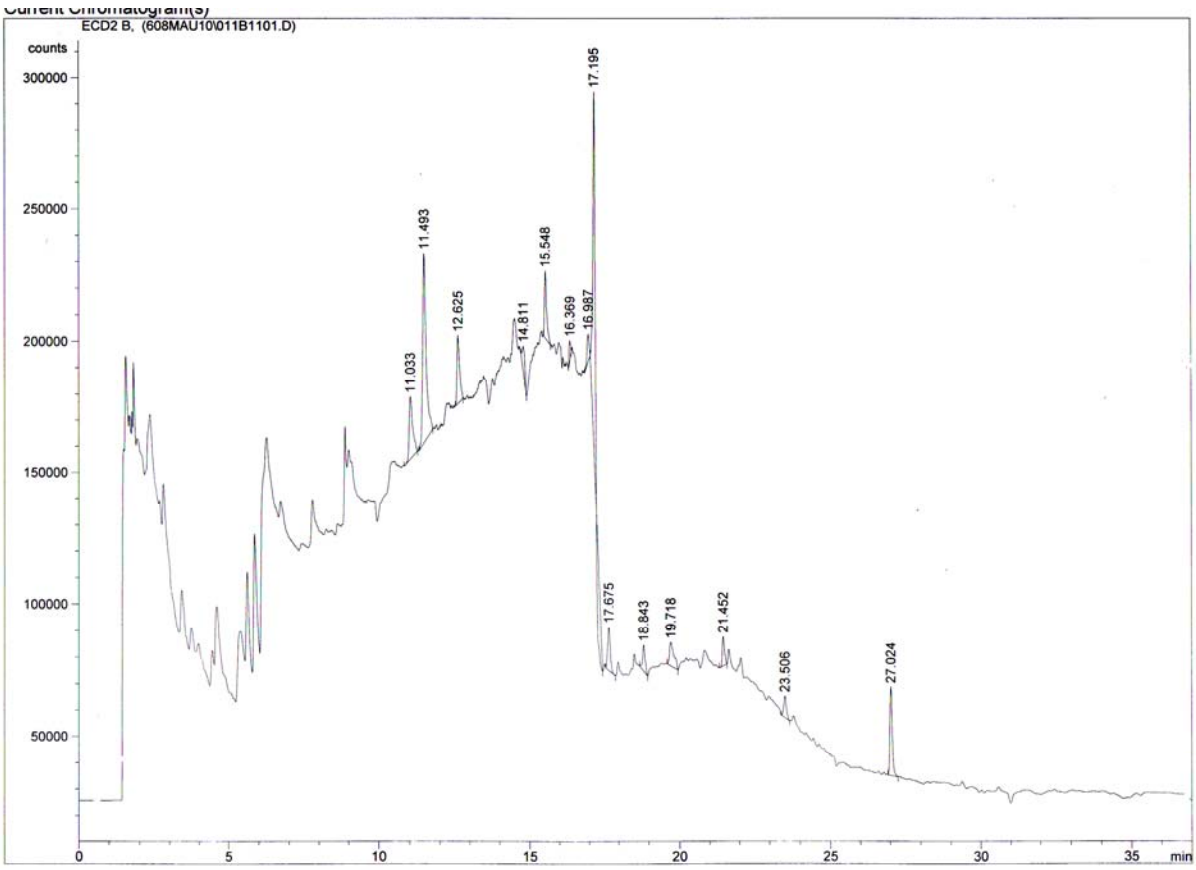

Fig. 9. *GC/ECD Chromatogram of OCP residues for Water sample in Dry Season at SW1*

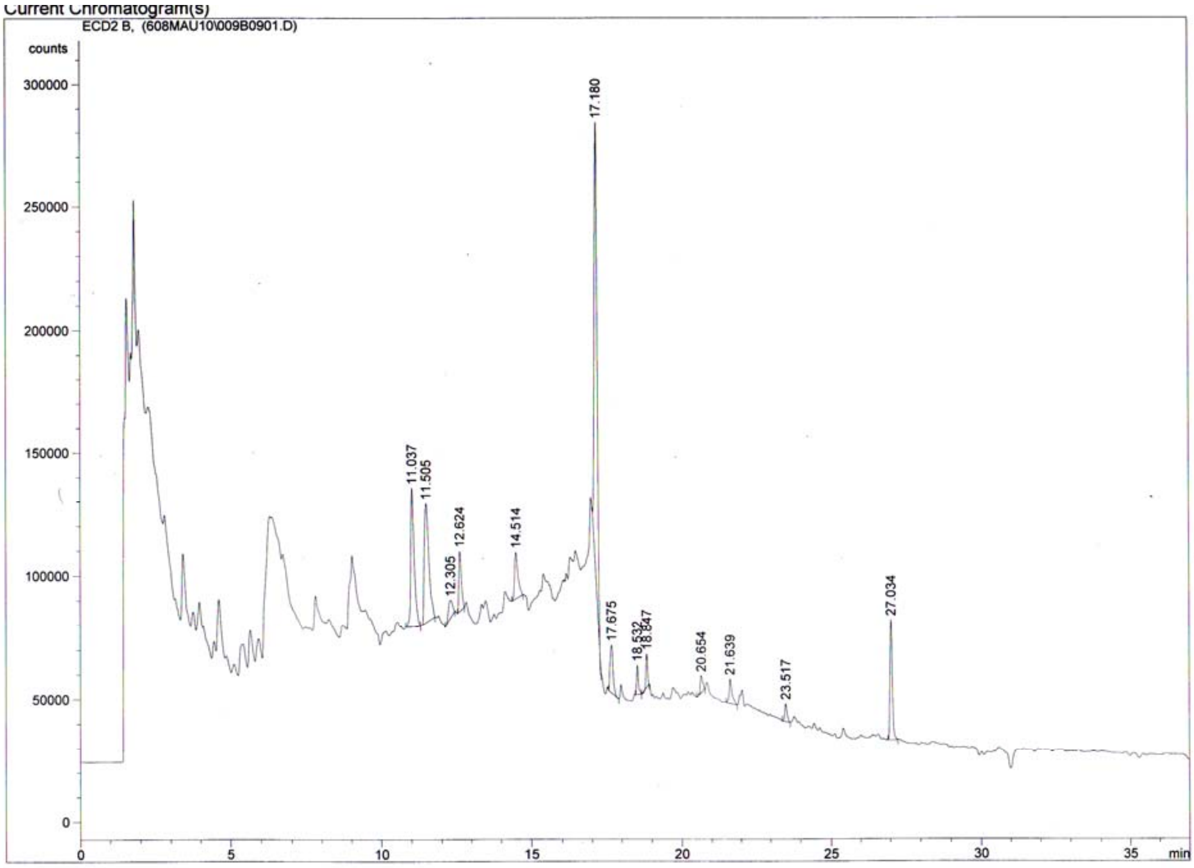

Fig. 10. *GC/ECD Chromatogram of OCP residues for surface water sample in Dry Season at SW2*

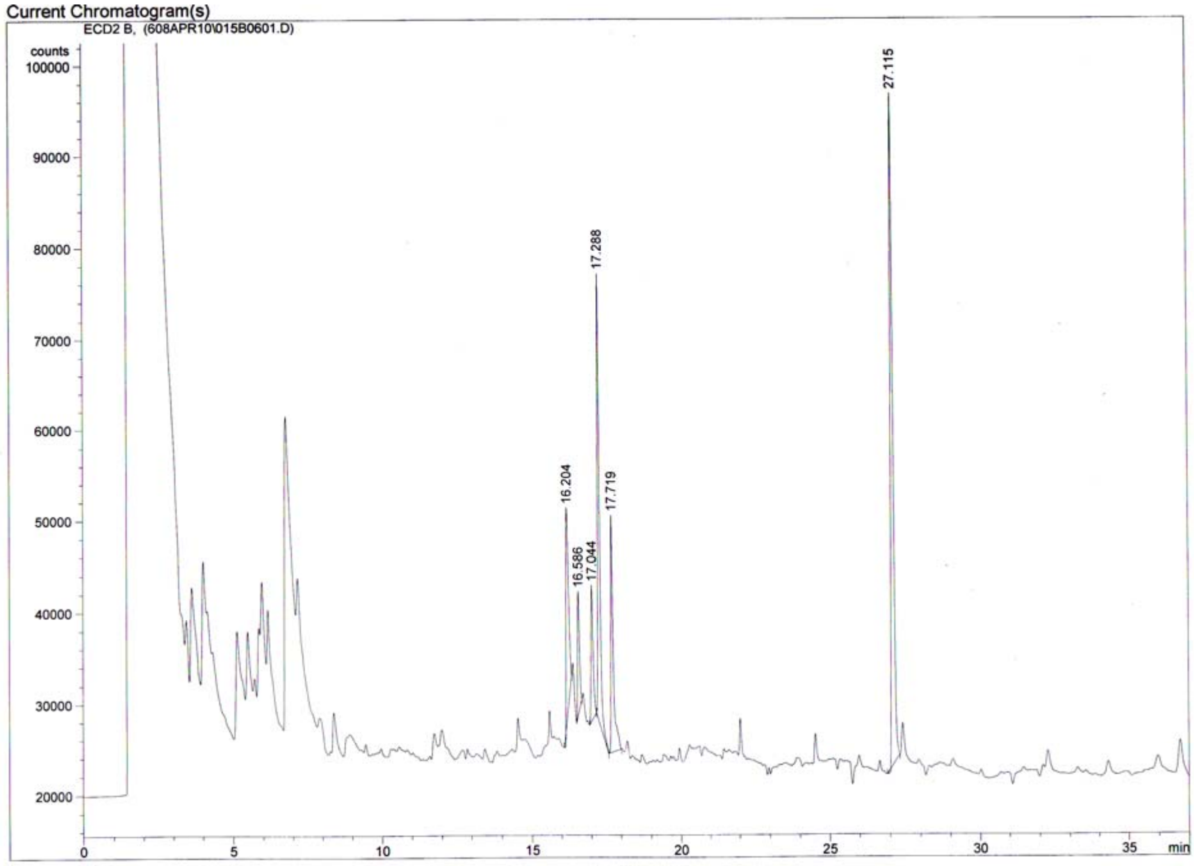

Fig. 11. GC/ECD Chromatogram of OCP residues for groundwater samples in Dry Season

In general, the levels of OCPs in the study area for the surface and groundwater are still within safety margins compared to Canadian water quality guidelines for irrigation and fresh water (CWQGs, 2005) as shown in Table 4.

Finally, the residue levels of OCPs found in surface water are higher than the concentrations in the groundwater. Since these compounds degrade very slowly and tend to accumulate in the sediments, they may subsequently leach out into the surrounding aquatic system.

Table 4. Concentration of OCPs residue in water according to CWQGs, 2005

OCPs	CWQGs (µg L^{-1})	
	Irrigation water	Fresh water
Total HCHs	—	0.01
Total DDE	—	0.001
Total DDD	—	—
Total DDT	—	—
Aldrin	—	0.004
Endrin	—	0.0023
Dieldrin	—	0.004
Heptachlor	—	0.01
Endosulfan	—	0.02
Methoxychlor	—	—

CWQGs: Canadian water quality guidelines for the protection of agricultural water uses;
—: No guideline available.

4. Conclusion

Despite the long time restriction or bane of the use of these organochlorine compounds, the contamination pattern for the selected surface and groundwater samples collected of the above parameters from El-Rahawy drain area are still within safety margins compared to Canadian water quality guidelines for irrigation and fresh water during rainy and dry seasons. This may be attributed to the pollution from large number of anthropogenic and agricultural activities throughout the year.

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Pesticidų organochlorinų matavimai paviršiniuose šuliniuose, esančiuose užterštoje Egipto El Rahawy teritorijoje

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Straipsnyje aprašomas El Rahawy teritorijos paviršinių ir požeminių vandenų užterštumo pesticidais organochlorinais tyrimas, analizuojant parinktas vietas. Tyrimai rodo, kad didesnis užterštumas pesticidais nustatytas paviršiniuose vandenyse. Nustatyta taršos kaitos priklausomybė nuo sezoniškumo.