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Organochlorine Pesticides in Ambient Air in Selected Urban and Rural Residential Areas in the Philippines Derived from Passive Samplers with Polyurethane Disks

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Abstract The passive sampler with PUF disk was applied to investigate the types and concentrations of organochlorine pesticides (OCPs) in ambient air in three urban and rural residential areas simultaneously at different weather conditions in the Philippines. The concentrations of OCPs derived from the passive samplers indicated clear distinctions in the predominance of certain types and amounts of OCPs in air at different sampling sites and periods of sampling. Chlordanes were detected in concentrations ranging from 218 to 2,324 pg/m^3 in the urban residential sites in all the sampling periods, indicating the possible use of these pesticides as termiticides in houses. Endosulfans were detected in two rural sites at 491 pg/m^3 and 904 pg/m^3 during one sampling period; indicating the possible use of the pesticide in the farm areas at that period.

Keywords Organochlorine pesticides · Ambient air pollution · Passive sampling

OCPs are persistent, lipophilic and semi volatile compounds; they are mostly associated with soil but can also partition in air (Meijer et al. 2003). Some OCPs have been shown to have adverse effects on the metabolic functions of hormones. DDT, DDE and methoxychlor have shown estrogenic properties that resulted in anti-androgenic reproductive abnormalities in offsprings in animal studies (Gray et al. 2001). Other studies showed the association of long term, low dose exposure to metabolites of some OCPs to cancer such as oxychlordane to prostate cancer (Ritchie et al. 2003) and dieldrin to breast cancer (Mitra et al. 2004). Since the general population is exposed to air, it is important to investigate the OCPs present in air that can cause potential adverse effects on the population.

Several scientists used passive air samplers to investigate the atmospheric concentrations of persistent organic pollutants simultaneously; and were able to compare the toxic contaminants in both spatial and temporal scale (Harner et al. 2006; Gouin et al. 2005; Jaward et al. 2004). Polyurethane (PUF) disk passive samplers have been sufficiently characterized in terms of theory and application to derive air concentrations of persistent organic compounds (Pozo et al. 2004; Shoeib and Harner 2002). This study investigated the types and levels of OCPs in ambient air in three urban and three residential areas simultaneously at different weather conditions using passive samplers with PUF disks.

Materials and Methods

Samples were collected in three urban residential sites within the Metro Manila area and in three rural residential sites in the Philippines (Fig. 1). In each site, two samplers were mounted about 1.5 m high on a tree in an area with unobstructed flow of air. Only one field blank was obtained for each sampling site. The sampling conditions and the local weather conditions during the study period are listed in Table 1.

The passive sampler and the PUF disk are exactly as described in the study of organic pollutants in air in Chile (Pozo et al. 2004). The PUF disks (14 cm diameter; 1.35 cm thick, 365 cm^2 surface area; 4.40 g mass; 0.0213 g cm^{-3} density supplied by PacWill Environmental, Stoney Creek, Ontario) were housed in two stainless

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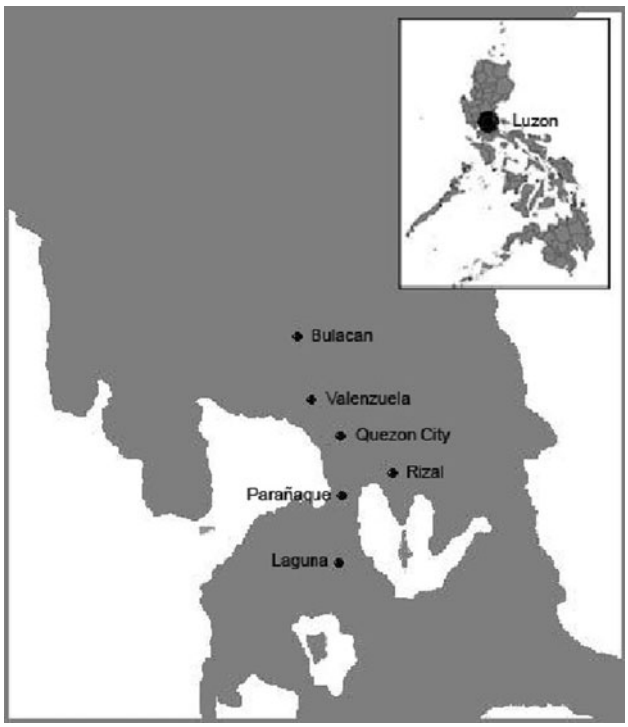

Fig. 1 Location of sampling sites

steel domes which protect the disk from precipitation, UV sunlight and particle deposition. To calibrate the adsorption phase of the field sampling, seven passive air samplers were installed close to each other at the same time in a representative sampling environment. One sampler was retrieved at days 11, 22, 34, 44, and 56 of exposure and analyzed for OCPs.

Known concentrations of depuration compounds phenanthrene-d10 and $^{13}C_{12}$ PCB 28 were spiked on previously cleaned PUF disks. The samplers were deployed in four field sites for two sampling periods and the concentrations of the labeled compounds on the PUF disks were determined after 56 days exposure. The $K_{PUF\text{-}A}$, the retention capacity of the disk for the compound, was estimated as the K_{OA}, which was obtained from available literature (Li et al. 2003; Beyer et al. 2002). The $K_{PUF\text{-}A}$ values were corrected against temperature (Harner and Bidleman 1998) and multiplied by the density of the PUF disk (Pozo et al. 2004) before using in the equation to calculate k_A.

A method blank was included in each batch of four field samples for extraction. Surrogate standard $^{13}C_{12}$ *p,p′* DDT (50 ng) was used to assess the performance of the procedure in each sample. The performance of the analytical process was determined by the recovery of known amounts of mixed OCP standard (50 ng) and OCP surrogate standard (50 ng) that were added to an unexposed PUF disk before extraction. The PUF disks were cleaned prior to deployment in the sampling sites. The retrieved field- exposed PUF disks, excluding the depurated samples, were spiked with the surrogate standard before extraction. The OCPs adsorbed in the disks were extracted in a Soxhlet with 1:1 DCM/Hexane for 20 h and concentrated in a rotary evaporator. The extract was passed through a 1,000 mg florisil solid phase extraction (SPE) cartridge and eluted with 10 mL of 10% acetone in hexane. The extract was concentrated by blowing with a gentle stream of nitrogen to less than 1 mL and transferred to a 1.5 mL vial. A mixture of internal standards phenanthrene-d10, pyrene-d10, chrysene-d12 (50 ng) was added to the extract prior to adjusting the volume to 1 mL and analysis by Gas Chromatography Mass Spectrometer (GCMS).

Table 1 Details of the location and weather conditions during the study

Sampling site	Coordinates		Altitude masl	Location	Average temperature
a. Location and average temperature in sampling sites					
Bulacan	14°49′.10N, 120°54′.36E		10	Rural	27°C
Valenzuela	14°41′.06N, 120°58′.93E		9	Urban	30°C
Quezon city	14°38′.92N, 121°01′.89E		42	Urban	29°C
Parañaque	14°29′.29N, 121°02′.21E		18	Urban	31°C
Rizal	14°33′.73N, 121°07′.77E		11	Rural	28°C
Laguna	14°20.21N, 121°04′.67E		18	Rural	29°C
Sampling period (year 2005)	Exposure (days)	Weather condition	Relative humidity (%)	Min–max temp, (°C)	Rainfall, (mm)
b. Weather conditions during exposure periods of passive samplers					
1. May 24 to Jul 5	42	End of dry season	79	24.6–34.1	613
2. Jul 5 to Aug 16	42	Rainy season	83	24.4–31.6	459
3. Aug 16 to Oct 11	56	Rainy season	83	24.4–31	739
4. Oct 11 to Dec 6	56	End of rainy season	82	23.6–30.6	415

All field sample extracts were analyzed for α-BHC, heptachlor, aldrin, heptachlor epoxide, α chlordane, γ chlordane, trans and cis nonachlor, endosulfan1, endrin, dieldrin, *o,p′*DDE, *o,p′*DDT, *p,p′*DDE, *p,p′*DDD, *p,p′*DDT, endosulfan 2, endosulfan sulfate, methoxychlor, and mirex in a Shimadzu GCMS QP2010 with electron impact (IE) ion source and auto injector. The GC/MS was equipped with a DB-5 (30 m, 0.32 mm ID, 0.25 μm thickness) column and operated with Helium as carrier gas at 3 mL/min. The sample (2 μL) was introduced into the GC by splitless injection and the peaks were acquired in the selected ion monitoring mode (SIM) mode. The GCMS analysis for OCPs was operated under the following temperature conditions: Injector temperature, 280°C; detector temperature, 200°C; interface temperature 280°C; temperature program, 50°C for 2 min, 20°C min $^{-1}$ to 130°C, 5°C min $^{-1}$ to 200°C, 15°C min $^{-1}$ to 300°C for 5 min. Scanning ranged from mz 67–411at 0.2 s SIM sampling interval. The concentration of the OCP in the sample extract was derived directly from the calibration curve using the internal standard method. A mixture of OCPs standards (Protocol Analytical, USA) was used for the quantification of OCPs. Mixed standard solutions containing 0, 5, 10, 50, 100 and 200 ng/mL OCPs with 50 ng/mL mixed internal standard and 50 ng/mL surrogate standard were used in the preparation of the calibration curves. Different sets of standards were prepared for the calibration of the labeled compounds in the depuration experiments. Phenanthrene-d10 (SUPELCO) standard solutions at concentrations of 0, 50, 100, 200, 500, 750 and 1,000 ng/mL with internal standard 50 ng/mL pyrene-d10 (SUPELCO) and standard solutions of $^{13}C_{12}$ PCB 28 (Wellington Labs) at 0, 1, 10, 20, 40 and 50 ng/mL with 10 ng/mL mixed PCB recovery standards (Wellington Lab), were used for quantification of the depurated PAH and PCB compounds respectively.

Results and Discussion

The average sampling rate obtained from depuration of phenanthrene-d10 and $^{13}C_{12}$ PCB 28 (range of 1.9–6.9 m^3/d) agrees with the sampling rate from depuration of $^{13}C_{12}$ PCB-30 (4.8 ± 2.3 m^3/d) in Chile (Pozo et al. 2004). Table 2 shows the calculated K_{OA}, the ratio of the concentrations of the depuration compounds before and after exposure and the sampling rates obtained from the depuration experiments. The average sampling rate of 4.59 m^3/day from depuration of phenanthrene-d10 and $^{13}C_{12}$ PCB 28 translates to k_A of 0.14 cm/sec for the 365 cm^2 PUF disk which compares well with the k_A of 0.15 cm/s obtained in Chile (Pozo et al. 2004) and 0.098 cm/s obtained in the Great Lakes (Gouin et al. 2005).

Table 2 Relevant data from depuration experiment for calculation of sampling rate in the passive sampler

	Paranaque	Laguna	Bulacan	Valenzuela	Average
Phenantrene-d10 [a]K_{OA} at 29°C, 7.43					
[b]C_f/C_o	0.49	0.53	0.69	0.55	0.57
[c]R	3.6	3.2	1.9	3.2	2.9
[13] C_{12} PCB 28 K_{OA} at 29°C, 7.67					
C_f/C_o	0.53	0.49	0.48	0.45	0.49
R	5.5	6.1	6.3	6.9	6.2

log [a] K_{OA} corrected at the average temperature of the sampling sites (29°C) with the regression equation log K_{OA} (29°C) = A + B/T, A = − 5.62, B = 3,942. T = 302 for phenanthrene-d10 (Harner and Bidleman 1998) and A = − 5.91, B = 4,102. T = 302 for PCB 28 (Li et al. 2003); [b] C_f/C_i – the ratio of concentrations of depuration compound retained on the PUF disk at the end of 56 days exposure period and on the PUF disk before exposure. [c] R is sampling rate in m^3/day, calculated as $R = A_{PUF} \times k_A$ where A_{PUF} is area of PUF disk; k_A was calculated at each sampling site as $k_A = -\ln(C_f/C_i) \times (\delta \times K_{PUF\text{-}A} * \text{density of PUF disk/t})$ where δ is thickness of the PUF disk (Pozo et al. 2004)

Table 3 Average recoveries of individual OCPs (50 ng) in method control samples (n = 7)

OCP	% Rec	SD	RSD	OCP	% Rec	SD	RSD
α-BHC	66	24	37	Endrin	96	48	50
Heptachlor	90	72	79	Endosulfan II	100	69	69
Aldrin	55	45	82	Endrin aldehyde	224	193	86
Heptachlor epoxide	94	29	30	*p,p′* DDD	115	28	24
γ-Chordane	83	40	48	cis-Nonachlor	111	62	56
Endosulfan1	88	80	90	*o,p′* DDT	73	54	73
α Chlordane	91	25	27	Endosulfan sulfate	67	66	98
trans-Nonachlor	87	27	31	*p,p′* DDT	129	49	38
Dieldrin	90	45	50	Endrin ketone	96	74	77
p,p′ DDE	106	13	12	Methoxychlor	70	59	84
o,p′ DDD	101	35	34	Mirex	72	43	59

The average recovery of $^{13}C_{12}$ *p,p′* DDT from samples was 121% (26% SD). Forty three samples (57%) gave % recoveries within the acceptance criteria for recovery (range of 100 ± 40%) and thirty three samples (43%) gave recoveries outside of the acceptable range. The average recoveries of the individual OCPs except endrin aldehyde and β, γ, δ BHCs in the spiked PUF disk ranged from 59% to 119% (Table 3). Endrin aldehyde gave consistently high recoveries with an average recovery of 224% which was not acceptable. On the other hand, β, γ, δ BHC were not detected in most of the spiked samples. Endrin aldehyde and β, γ, δ BHCs were not reported in the samples.

Table 4 Method detection limits (MDLs) of OCPs

OCP	MDL, ng/disk	OCP	MDL, ng/disk
alpha-BHC	0.4	trans-Nonachlor	5
Heptachlor	2	*p,p′* DDD	0.1
Aldrin	8	cis-Nonachlor	3
Heptachlor epoxide	3	*o,p′* DDT	3
gamma-Chordane	10	Endosulfan sulfate	3
Endosulfan1	10	*p,p′* DDT	3
alpha Chlordane	6	Endrin ketone	3
Endosulfan1	10	Methoxychlor	0.2
alpha Chlordane	6	Mirex	2

The Method Detection Limit (MDL) was derived as the average blank concentration (n = 13) plus three times the standard deviation of the blank concentrations. The MDLs for the OCPs ranged from 0.2 to 12 ng/disk (Table 4). When target compounds were not detected in blanks, ten times the standard deviation of the instrument detection limit (IDL) was used as MDL. The total OCPs in the four field blanks ranged from 3 to 30 ng/disk. The concentrations of OCPs in the field blanks are comparable to the total concentrations of the OCPs obtained in the method blanks. Only data with concentrations greater than the MDLs and with Relative Standard Deviations not exceeding 70% for two trials were reported.

No linear trend was shown by the DDT adsorption profile; the concentrations detected for this compound in the extracts were very low (3–6 ng/disk) and near the MDL (3 ng/disk) for *p,p′* DDT. Heptachlor epoxide showed a high concentration (75 ng/disk) at 11 days of exposure; its concentration reached a plateau at 20 days, indicating that equilibrium may have been reached early. It suggests that the concentration of this compound in ambient air at the site is higher than the other OCPs. However, the presence of oxygen may have made heptachlor epoxide vulnerable to decomposition in air or in the PUF disks such that its concentration did not show an increase with longer time of exposure. For this non-linear adsorption behavior, heptachlor epoxide was not included in the report. The adsorption behavior of aldrin, *p,p′* DDE, *p,p′*DDD, endosulfan 2, endrin ketone, methoxychlor and mirex were not determined because these compounds were not detected in the site during the exposure period. Dieldrin, endosulfan1 and endosulfan sulfate showed linear phase adsorption up to day 46 and showed a decrease in adsorption beyond that period. The decrease in the recovered pesticides could be due to some decomposition of the pesticides; the presence of reactive sites (O and S) could have initiated structural

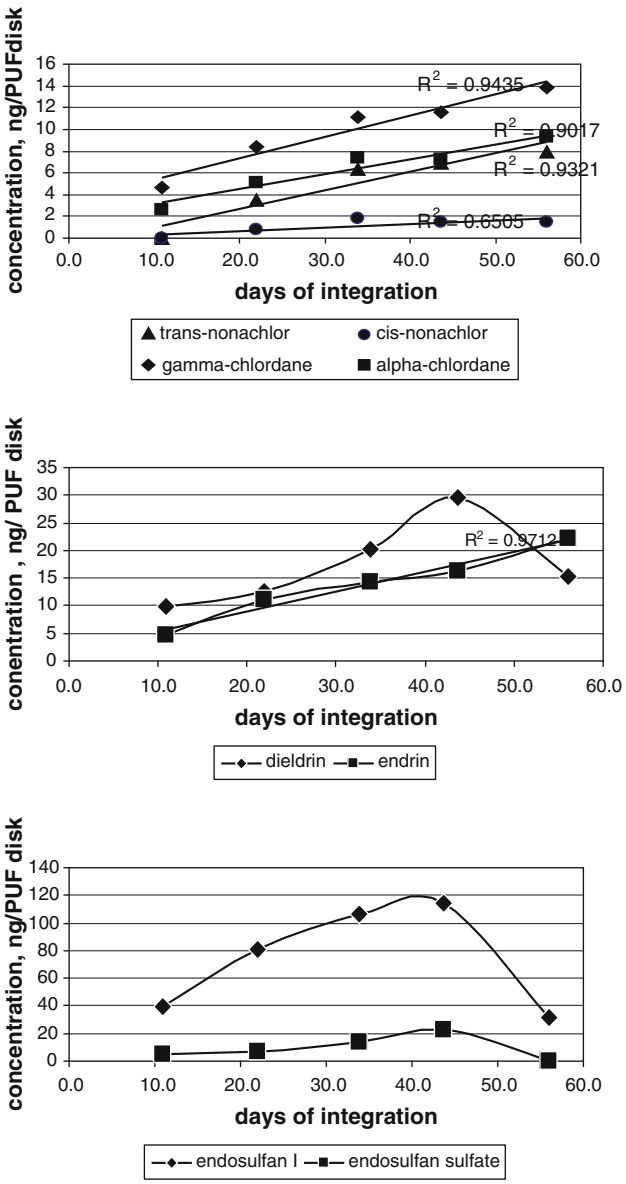

Fig. 2 The uptake of OCPs in the PUF disk within 56 days

changes in the pesticides with prolonged exposure. The linear phase adsorption of α chlordane, γ chlordane, trans nonachlor, and endrin in the PUF disk within the exposure period of 56 days were confirmed (Fig. 2).

Generally, chlordanes (α and γ chlordane and cis and trans nonachlors), endosulfans (endosulfan 1, endosulfan 2 and endosulfan sulfate) and endrins (aldrin, endrin and dieldrin) were the major contributors to the OCP contamination in air in all the sampling sites. The group of DDTs (DDT, DDE and DDD) gave the lowest concentrations (<25 ng/disk) among the OCPs monitored. The total OCPs sequestered on the PUF disk (sum of concentrations of detected OCPs) in all the sites ranged from 33 to 398 ng/disk, with the highest concentrations found in most urban sites. Among the urban sites,

Table 5 Concentrations (pg/m^3) of OCPs in urban (U) and rural(R) residential sites for the four sampling periods

	Paranaque (U)				Valenzuela (U)				Quezon city (U)			
Sampling period	1	2	3	4	1	2	3	4	1	2	3	4
alpha-BHC	ND	ND	ND	205	ND	ND	ND	138	ND	ND	ND	207
Heptachlor	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	275
γ-Chordane	578	569	437	364	155	104	88	288	168	111	85	94
α Chlordane	423	414	743	664	96	68	71	191	108	75	62	74
trans-Nonachlor	368	409	975	628	98	75	66	139	102	86	59	56
cis-Nonachlor	57	72	169	ND	17	15	17	38	bdl	bdl	12	ND
$\sum$ chlordanes	1,426	1,464	2,324	1,656	367	262	242	656	378	272	218	224
Aldrin	ND	ND	ND	96	ND	ND	ND	76	ND	ND	ND	121
Dieldrin	72	94	ND	ND	ND	ND	ND	61	ND	129	ND	62
Endrin	32	ND	27	90	bdl	32	49	64	ND	ND	14	ND
Endrin ketone	ND	ND	ND	507	ND	ND	ND	281	ND	ND	ND	ND
$\sum$ Endrins	104	94	27	693	ND	32	49	482	ND	129	14	183
Endosulfan1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	139	ND
Endosulfan II	ND	ND	142	ND	440	338	ND	ND	387	226	322	ND
Endosulfan sulfate	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
$\sum$ Endosulfan	ND	ND	142	ND	440	338	ND	ND	387	226	461	ND
o,p′ DDD	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	28	ND
o,p′ DDT	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
p,p′ DDT	bdl	ND	29	ND	23	ND	ND	ND	ND	ND	ND	ND
$\sum$ DDTs	ND	ND	29	ND	23	ND	ND	ND	ND	ND	28	ND
Mirex	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	178
	Laguna (R)				Rizal(R)				Bulacan(R)			
Sampling period	1	2	3	4	1	2	3	4	1	2	3	4
alpha-BHC	ND	ND	ND	181	ND	ND	ND	ND	ND	ND	ND	ND
Heptachlor	ND	ND	ND	ND	ND	ND	31	125	ND	ND	ND	ND
γ Chordane	49	bdl	ND	ND	82	bdl	39	54	83	75	ND	ND
α Chlordane	33	bdl	ND	ND	52	45	25	45	51	49	ND	ND
trans-Nonachlor	26	33	ND	bdl	43	52	29	31	47	49	ND	ND
cis- nonachlor	ND	ND	ND	ND	bdl	ND	bdl	ND	ND	bdl	ND	ND
$\sum$ chlordanes	108	33	ND	ND	177	97	93	130	182	173	ND	ND
Aldrin	ND	ND	ND	127	ND	ND	ND	118	ND	ND	ND	ND
Dieldrin	ND	ND	ND	ND	234	95	41	55	ND	ND	ND	ND
Endrin	ND	61	90	47	ND	ND	20	ND	ND	ND	16	ND
Endrin ketone	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
$\sum$ Endrins	ND	61	90	174	234	95	61	173	ND	ND	16	ND
Endosulfan1	ND	ND	ND	ND	ND	bdl	55	93	ND	686	ND	ND
Endosulfan II	ND	491	ND	ND	ND	ND	148	ND	293	218	112	375
Endosulfan sulfate	47	ND	ND	ND	ND	ND	ND	ND	52	ND	ND	ND
$\sum$ Endosulfan	47	491	ND	ND	ND	ND	203	93	345	904	112	375
o,p′ DDD	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
o,p′ DDT	34	ND	ND	64	bdl	ND	ND	ND	ND	bdl	ND	66
p,p′ DDT	ND	ND	ND	ND	ND	ND	ND	ND	bdl	ND	ND	ND
$\sum$ DDTs	34	ND	ND	64	ND	ND	ND	ND	ND	ND	ND	66
Mirex	88	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

[a] *ND* non detected

[b] *bdl* below detection limit

Parañaque consistently showed the highest concentrations of OCPs in all the sampling periods (300–400 ng/disk). Bulacan showed the highest total concentration of OCPs (175 ng/disk) among the rural sites during the second sampling and this could be attributed to the high concentration of endosulfans detected during this period. It is possible that there was recent application of endosulfans during the second sampling period, as this season coincided with planting season.

Table 5 shows the concentrations of OCPs detected in air during the four sampling periods using the average sampling rate derived from the passive sampler. Chlordanes, which could be attributed to its use as termiticides in the urban residential villages, were found in the ambient air in the urban sites consistently in relatively higher concentrations (218–2,324 pg/m^3) than endrin (ND-693 pg/m^3) and endosulfans (ND-461 pg/m^3) during the four periods of investigation. Endosulfans were detected in the farming area in Bulacan in all the sampling periods (102–904 pg/m^3) while endrins, principally dieldrin, was detected (61–234 pg/m^3) in Rizal. The concentrations of chlordanes detected in air in the urban sites in this study are much higher than the reported chlordane concentration (35 pg/m^3) in air in Concepcion City in Chile (Pozo et al. 2004). The urban sampling sites in this study are middle class residential villages with houses which were treated against termite infestation. The historical treatment of the soil with chlordane during construction of the houses before the ban of the pesticide could be responsible for the high concentrations of chlordanes in air in the urban villages. The predominance of endosulfans and dieldrin in the rural sites agrees with the result of the passive sampling of air in the Great Lakes Basin; which indicated the predominance of endosulfans (40–1,090 pg/m^3) and dieldrin (15–165 pg/m^3) in agricultural sites of the basin (Gouin et al. 2005).

Passive sampling using PUF disks was useful in determination of the types and concentrations of predominant persistent organochlorine pesticides such as chlordanes, nonachlors, endrin and endosulfans in ambient air in the urban and rural residential areas in the Philippines. Based on the experiments on the calibration of the adsorption phase under local conditions, exposure of the PUF disks at 42 days is most applicable for the determination of the concentrations of the prevalent organochlorine pesticides in air.

Passive sampling of OCPs showed that the residents in the urban sites in this study are continuously exposed to chlordanes in ambient air. It may be worth to investigate the blood levels of chlordane and its metabolite oxychlordane in the residents to assess their risk to prostate cancer.

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