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Multi-medium Distributions of HCHs, DDTs, and PCBs in Typical Petrochemical Industrial Area and Surrounding Regions of Jilin Province, Northeast China

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Abstract The levels of DDTs, HCHs and PCBs in topsoil, cereal and irrigation water from typical industrial and agricultural areas of Jilin Province in Northeastern China were evaluated by using gas chromatography coupled with an electron capture detector. The amount of $\sum$OCPs and $\sum^{7}$PCBs found in topsoils ranged from 24.7 to 98.0 and 17.2 to 98.7 ng g^{-1}, respectively. The geometric means of $\sum$HCHs, $\sum$DDTs and $\sum^{7}$PCBs in rice stem samples were 28.9, 32.4 and 49.0 ng g^{-1}, respectively. The average level of total OCPs concentration in rice field water in Meihekou area (0.849 ng g^{-1}) is higher than that in Jilin area (0.178 ng g^{-1}) and all OCPs concentrations in rice field water met the water quality standards for Grade I regulated by China's national environmental quality standard of surface water.

Keywords Organochlorine · Polychorinated biphenyl · Jilin Province · Topsoil · Cereal · Irrigation water

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Organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) had been extensively used in China, resulting in widespread environmental occurrence. These semi-volatile compounds may accumulate in cold region by long-range transport and deposition through atmosphere and water because low ambient temperature may retard their degradation in ecosystems of cold region (Gouin et al. 2004). Hence, distribution characteristics and pollution level of OCPs and PCBs in cold areas is of great significance (Wang et al. 2007; Wania and Westgate 2008). In addition, possible health risks of OCPs and PCBs in spite of low exposure concentration had increased public concerns about food quality and safety in polluted region.

Jilin Province is one of the important constitutes of northeast old industrial base and also one of the main production bases of commercial grain in the northeastern China. As we all know, Jilin Province is located in North Temperate Zone with a long and cold winter and annual mean temperature is −3–7°C. The province may be roughly divided into two parts: the eastern mountains and the western plains. Mountainous and hilly areas occupy more than 60 % of its land. Recently, the region is suffering an increasingly serious environmental pollution such as the spill of chemicals in the Songhua River after the explosion incident happened in Jilin Petrochemical Plant in 2006 (Fu et al. 2007). In addition, according to the Songhua River Water Pollution Control Plan (2006–2010) promulgated by the national government in 2006, there are 157 petrochemical factories along the Songhua River that discharged metals or organic pollutants. Hence survey on the pollution status of OCPs and PCBs in this region has intrigued the environmental experts and biologists, but the investigations were sporadic and partial. Previous studies had proved the existence of HCH and DDT residues in the region (Cai et al. 2008; Yu et al. 2007). Fan (2007)

measured 19 PCBs isomers in the Songhua River sediments and reported PCBs concentration reached maximum near Jilin City which lies on the upper reaches of the river. Yu et al. (2007) reported that HCHs and DDTs were the main OCP species in this region.

The aim of present study was to investigate multi-medium environmental spatial distribution, composition characteristics and pollution status of OCPs and PCBs in the typical petrochemical industrial region and the surrounding agricultural region. In addition, the ecologic risks of these persistent organic pollutants were also evaluated so as to improve our understanding of the pollution sources and current contamination status of these pollutants, especially the aftermath of the explosion incident in 2006.

Materials and Methods

Surface soil samples (0–20 cm) and crop samples (rice and maize) were collected from agricultural land of Jilin City, Meihekou City and Changbai County in June and October in 2009, respectively. Sampling locations (Fig. 1a) were orientated through GPS satellite navigation system. All the samples were kept in aluminum foils baked out before use. After air-dried at room temperature, all samples were ground to pass through a 60 mesh sieve and stored in refrigerator until analysis. Paddy field water samples were collected from rice field in the suburb of Jilin City and Meihekou City in early June, 2009. Two surrogate standards (PCB209 and 2,4,5,6-tetrachloro-m-xylene, TMX) were added on site to all water samples. On the same day water samples were filtered by glass fiber filter membrane (Waterman 0.7 mm GF/F, 0.47 mm i.d.) to separate soluble from insoluble substances, all of which were kept at 4°C. At the same time, rice seedlings were collected. Mature rice grain was collected for comparative analysis in October, 2009.

N-hexane, acetone, dichloromethane (HPLC grade, Tianjin Kermel Chemical Reagent Co. Inc., China), methanol (analytical grade, Jinan Chemical Reagent Co. Inc., China), sulfuric acid (guaranteed reagent, Qingdao Chemical Reagent Co. Inc., China), copper chips (analytical grade, Tianjin Huazhen Chemical Reagent Co. Inc., China) and granular anhydrous sodium sulfate (analytical grade, Tianjin Tanggu Dengzhong Chemical Reagent Co. Inc., China) were used as received. Two surrogate standards (PCB209 and TMX) and fifteen targeted analysis standards including PCB28, PCB52, PCB101, PCB118, PCB138, PCB153, PCB180, α-, β-, γ- and δ-HCH, p,p'-DDD, p,p'-DDE, p,p'-DDT, o,p'-DDT were purchased from National Research Center for Certified Reference Materials. Florisil (60–100 mesh, PR grade) and granular anhydrous sodium sulfate were activated at 650°C in a furnace for 6 h and then kept in sealed desiccators. Florisil was then activated at 130°C for 16 h before use. All glassware were cleaned in an ultrasonic cleaner and heated at 350°C for 12 h.

10 g soil sample was weighed precisely and spiked with surrogate standards TMX and PCB209, and then was Soxhlet-extracted with 150 mL mixture of hexane/acetone (1:1, v/v) for 16 h. Elemental sulfur was removed by adsorption onto activated copper chips. The crude extracts

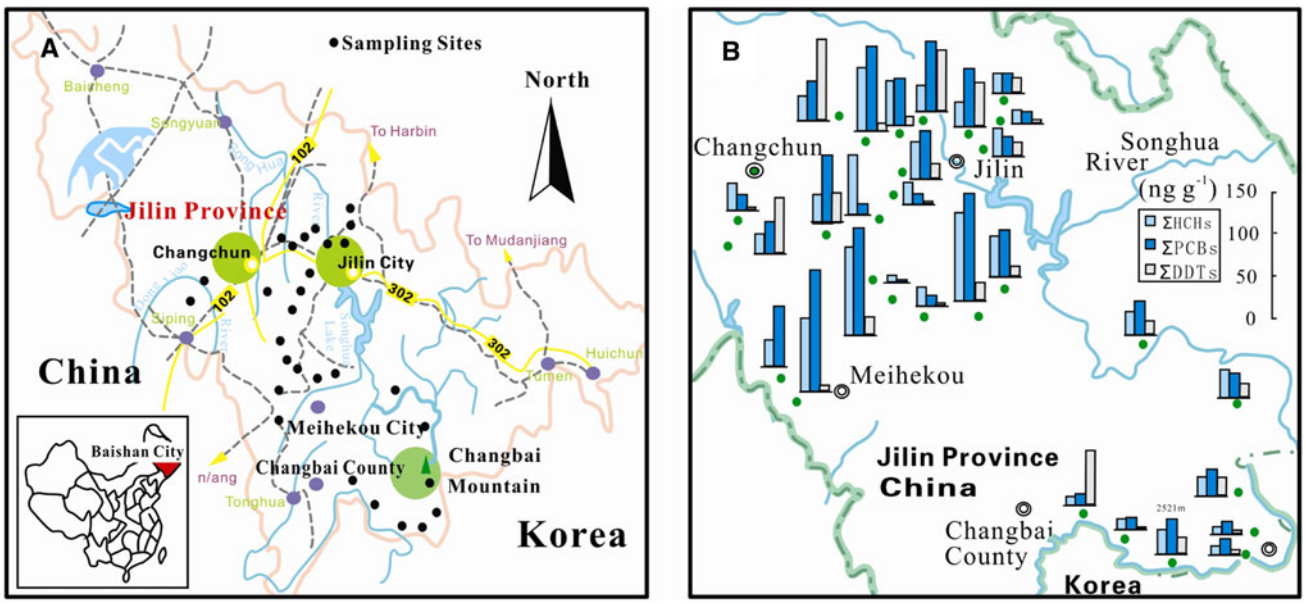

Fig. 1 Illustration of sampling sites (**a**) and the distribution characteristics of OCPs and PCBs concentrations in surface soils of Jilin Province (**b**)

were approximately concentrated to 3 mL in a vacuum rotary evaporator at a temperature below 38°C. The extracts were transferred to the Florisil column (packed with 20 mm anhydrous sodium sulfate, 5 g Florisil and 20 mm anhydrous sodium sulfate in ascending order) and eluted with 100 mL hexane/ether mixture (94:6, v/v). The eluate was concentrated to 5 mL and then transferred to a 250 mL separatory funnel with 40 mL of n-hexane. A total of 40–60 mL of concentrated sulfuric acid was added for sulfonation. The sulfonated extract was mixed with 2 % sodium sulfate solution. The remaining extract was concentrated to dryness in a vacuum rotary evaporator and then diluted with n-hexane. The solution was evaporated to nearly dryness by a gentle stream of nitrogen at room temperature, and then dissolved in 0.5 mL n-hexane. The samples were sealed in vials and stored at −4°C before analysis. Plant samples were subjected to an identical procedure for soil samples except that 5 g of dried sample was used.

The procedure for the pretreatment of water sample was shown as follows: 500 mL water sample was placed into 1 L separatory funnel and 10 g sodium chloride was added. The funnel was shaken to dissolve sodium chloride completely. After that, 50 mL of n-hexane was added and then the funnel was shaken vigorously for 5 min. The separatory funnel was kept undisturbed for 30 min to separate the two layers. The lower aqueous layer was extracted two more times using fresh 25 mL n-hexane. The extracts were combined and dried by passing through an adsorbent containing 5 cm layer of anhydrous Na_2SO_4 over a small filter paper. The extracts were concentrated to 5 mL with an evaporator at 35°C and then evaporated to nearly dryness by a gentle stream of nitrogen at room temperature, and then reconstituted in 1 mL of n-hexane.

Samples were quantitatively analyzed on a Shimadzu GC-2010 (Shimadzu Corp., Kyoto, Japan) with electron capture detector, equipped with fused-silica capillary column of DB-5 (30 m, 0.25-mm i.d. and 0.25 μm film thickness). Temperature programming was initialized with a 2 min hold at 100°C, then a ramp at 15°C/min to 180°C. Thereafter, the temperature was increased at 5°C/min to 300°C and held for 4 min. The temperature for injection port and detector was set at 260 and 320°C, respectively. The carrier gas was ultrahigh-purity nitrogen with a flow rate of 1.5 mL/min. Sample aliquots were analyzed by injecting 1 μL of each sample with the split ratio of 50:1. Retention time and reference materials were used to identify the analytes. External standard and calibration curve method were used for quantification.

Solvent blank and duplicate sample were processed for monitoring procedural performance and reproducibility. In addition, surrogate standards TMX and PCB209 were added to each of samples. The recoveries of surrogate spiked samples ranged from 75 % to 109 %. The detection limits calculated by standard reference materials addition for α-HCH, β-HCH, γ-HCH, δ-HCH, *p,p′*-DDE, *p,p′*-DDD, *o,p′*-DDT and *p,p′*-DDT were 0.018, 0.017, 0.024, 0.020, 0.017, 0.018, 0.025 and 0.029 ng g^{-1}, respectively. The detection limits for PCBs were 0.03–0.21 ng g^{-1}.

Results and Discussion

OCPs and PCBs contamination levels in the surface soil samples from Jilin City were investigated in detail (data not shown). HCH isomers were found and the total HCHs concentration (∑HCHs) was in the range of 16.4–32.3 ng g^{-1} with the geometric mean level of 27.0 ± 6.0 ng g^{-1} which was higher than that in cultivated soils in the Yangtze River Delta area, East China (An et al. 2004). ∑DDTs was in the range of 3.3–95.8 with 44.0 ng g^{-1} as geometric mean which was lower than that in agricultural soils in the Yangtze River Delta area. The ratios of α-HCH to γ-HCH in detected soil samples were in the range of 0.6–1.8, over 80 % samples with the ratio higher than 1.0, suggesting that the α-HCH has experienced long-year degradation. In addition, the usage of lindane cannot be excluded in this area considering the higher volatility of γ-HCH than that of α-HCH (Mackay et al. 1997). In most of sampling sites, (DDD + DDE)/DDT ratio ranges from 0.1 to 1.3 with the mean value 0.5 indicating that a long time has elapsed since the use of technical DDTs in the area. ∑PCBs concentration is in the range of 14.6–84.3 ng g^{-1} with the averaged content of 44.0 ng g^{-1}. PCB52, PCB101, PCB118 and PCB138 are the main existing forms. In addition, the contamination induced by the isomers with relative much chlorine substitute is much more distinct, which may be attributed to the abundance of petroleum and chemistry industry in this area.

The pollution status of OCPs and PCBs in rice field topsoils in the suburb of Meihekou City is surveyed (data not shown). ∑OCPs in the samples collected in summer contributed mainly from HCHs is in the range of 56.9–124.7 ng g^{-1} with the average of 98.0 ng g^{-1}, which was obviously higher than that in Jilin City area (Fig. 1b). It should be pointed out that total concentration of HCHs shows an increasing trend with the sampling site moving from Jilin City to Meihekou City (Fig. 2a). This may be aroused by the colder temperature in Meihekou area and the wind direction during the winter in the research region. Meihekou City is located in the down-wind of Jilin City and the pollutants may be transported by the North-East wind from Jilin City to Meihekou area. Another intriguing phenomenon is that the average ∑HCHs, ∑DDTs, and ∑PCBs concentration in the soil samples collected in October (the harvest season for rice), was remarkably lower than that in seedling period in spring and early

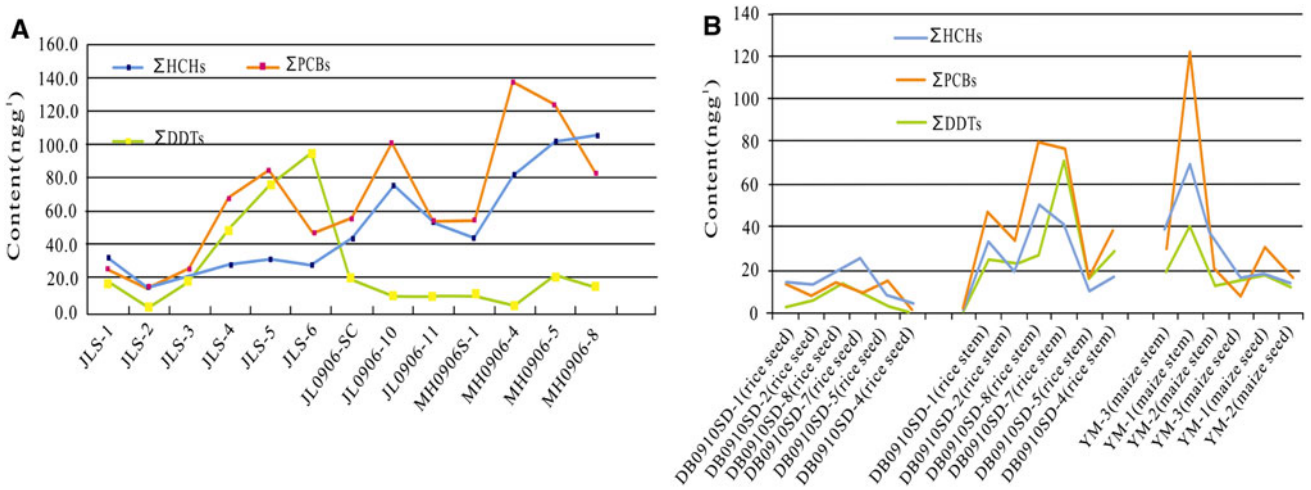

Fig. 2 Distribution patterns of POPs in rice field topsoils (**a**) and seed and stem samples (**b**) from Jilin City to Meihekou City (i.e. from north to south)

summer. This may be explained by the backing of the wind direction during the winter and spring season. The contents of β-HCH higher than those of α-HCH at some sites may be due to comparatively stable characteristics of β-HCH and its higher water solubility than α-HCH, therefore more easily transported via wet precipitation (Middeldorp et al. 1996). The geometric mean level of (DDD + DDE)/DDT were 2.6 and 2.4 in early summer and autumn, respectively. All above facts prove that DDTs has been degraded for many years. The distribution of DDTs and PCBs exhibits more significant fluctuation than that of HCHs in the samples collected from north hilly area to south mountain area. The average value of $\sum$PCBs in Meihekou City area is lower than that in Jilin City area, whether in summer or in autumn. The main PCB pollutants are PCB28, PCB52 and PCB138, which may be transferred through atmospheric wet precipitation because of high water-solubility and vapor pressure of PCB28 and PCB52, and abundant rains in that region.

OCPs and PCBs concentrations in the topsoil of Changbai County were significantly lower than those of Jilin City and Meihekou (Fig. 1b). For the sampling sites at high altitude, $\sum$HCHs were in the range of 3.09–25.6 ng g^{-1} with the geometric mean level of 10.5 ng g^{-1}. $\sum$DDTs ranged from 0.96 to 19.4 ng g^{-1} with 9.08 ng g^{-1} as geometric mean. The geometric mean level of $\sum$HCHs and $\sum$DDTs at low altitude were 16.2 and 18.7 ng g^{-1} respectively, suggesting a stronger agricultural activity at the low altitude area. The ratios of α-HCH to γ-HCH were in the range of 0.2–3.3 indicating that the usage of lindane cannot be excluded in this area. The OCPs and PCBs contaminations were found at low content level for most of the soil samples. In addition, the ratio of *o,p′*-DDT/*p,p′*-DDT in Changbai County is 8.0, which is unexpectedly high, and the average ratio of (DDD + DDE)/DDT is 0.55. This may imply that DDT residues in the soils of Changbai County may originate from recent input of kelthane. According to the toxicity standard of pollutant on invertebrates in soils from Urzelai et al. (2000), the ecological risk of HCHs in Changbai County is fairly low. In addition, according to the Jongbloed's food-chain model (Jongbloed et al. 1996), DDT residues in Changbai County may lead to potential ecological risk on birds and soil organisms. The total PCBs concentration were in the range of 7.32–31.9 ng g^{-1} with the geometric mean level of 17.2 ng g^{-1} and the main compounds were PCB28 and PCB 52. The sequence for the PCB average concentration is as follows: PCB28 > PCB52 > PCB180 > PCB138 > PCB153 > PCB101 > PCB118. This may be explained by the higher vapor pressure of PCB28 and PCB52 so as to be transferred more easily through atmosphere and enriched in cold areas than other isomers (Wania and Daly 2002). The pollution level of PCB residues in the topsoils from Changbai County was higher than that in Tibet (Sun 1984) and was comparable with that in Songhua River sediments. Our results suggest that the surrounding areas of the petrochemical industry may be affected by atmospheric transport of organic pollutants.

Rice and maize were also chosen as the investigation targets to evaluate the pollution status of OCPs and PCBs in Jilin Province. As shown in Table 1, the total HCH concentration in rice seeds varied from 5.2 to 25.9 ng g^{-1} with the geometric mean level of 14.4 ng g^{-1}. The content of δ-HCH was in the range of 2.2–11.7 ng g^{-1} with the average value of 7.4 ng g^{-1}, suggesting that HCHs residues mainly come from the historical usage. In addition, the import and usage of lindane may be excluded in the area for the reason that the geometric means of α-HCH/γ-HCH in rice seed and stem were 0.5 and 0.2, respectively (Table 2). The detection rate of DDTs was about 50 % in rice seed samples and the total DDT concentration varied

Table 1 OCPs and PCBs concentrations in rice seed samples (ng g^{-1})

Number	DBSD1 (Seed)	DBSD2 (Seed)	DBSD8 (Seed)	DBSD7 (Seed)	DBSD5 (Seed)	DBSD4 (Seed)	Average	SD*
α-HCH	1.7	1.3	1.8	4.8	1.2	0.4	1.9	1.5
β-HCH	1.6	2.6	0.8	4.2	0.9	0.9	1.8	1.3
γ-HCH	2.3	2.5	4.4	6.1	2.9	1.6	3.3	1.7
δ-HCH	8.9	6.8	11.7	10.8	3.8	2.2	7.4	3.8
α-HCH/γ-HCH	0.7	0.5	0.4	0.8	0.4	0.2	0.5	0.2
∑HCHs	14.5	13.2	18.7	25.9	8.8	5.2	14.4	7.3
PCB-28	7.2	3.8	8.0	5.9	5.4	0.7	5.2	2.6
PCB-52	3.5	0.6	3.9	1.6	7.0	nd	3.3	2.5
PCB-101	0.5	0.6	0.8	0.6	0.3	nd	0.5	0.2
PCB-118	nd	nd	1.3	nd	0.3	nd	0.8	0.7
PCB-138	nd	nd	nd	nd	1.1	nd	1.1	
PCB-153	nd	0.7	nd	nd	nd	nd	0.7	
PCB-180	1.9	3.0	1.1	2.1	0.9	52.2	10.2	20.6
∑PCBs	13.0	8.7	15.1	10.2	15.1	52.9	19.2	16.7
p,p′-DDE	0.7	1.2	nd	1.7	0.3	nd	1.0	0.6
p,p′-DDD	nd	nd	4.0	2.1	0.8	nd	2.3	1.6
o,p′-DDT	2.2	4.8	7.7	6.4	2.6	nd	4.7	2.3
p,p′-DDT	nd	nd	0.4	nd	nd	nd	0.4	
DDD/DDE	0.0	0.0		1.2	2.5		0.9	1.2
(DDD + DDE)/DDT		10.8				10.8		
∑DDTs	2.9	6.0	12.0	10.3	3.8	0.0	5.8	4.6
Recovery	0.9	0.9	0.8	0.8	0.7	0.8	0.8	0.1

*SD** Standard deviation; *nd* not detected

from nd (not detected) to 12.0 ng g^{-1}, with the average of 5.8 ng g^{-1}. The predominant DDT isomer was *o′,p′*-DDT. In rice stem samples, the detection rate of OCPs and PCBs residues was over 80 % and the geometric means of ∑HCHs, ∑DDTs and ∑PCBs in all samples were 28.9, 32.4 and 49.0 ng g^{-1}, respectively. Similar with the distribution of DDTs in rice seeds, *o,p′*-DDT was dominant isomer in DDTs, indicating the usage of technical kelthane. The sequence for the average concentration of PCB species in rice stem samples is as follows: PCB138 > PCB52 > PCB153 > PCB101 > PCB28 > PCB180 > PCB118. Compared with that in the topsoils, higher chlorinated isomers were relatively enriched in the rice stem. It should be noticed that the average content of ∑HCHs, ∑DDTs and ∑PCBs in rice stem samples significantly exceeded that in rice seed samples, which may be ascribed to the difference in enrichment ability of crop's diversified parts for different species. Similar phenomenon had been noticed by Bi et al. (2001). The maxima of HCHs and PCBs in rice seed and stem samples appeared in Hailong County of Meihekou City (Fig. 2b). The distribution trend of HCHs and PCBs may be caused by the abundant wet precipitation in this area. The distribution trend for DDTs was in a reverse way, i.e. decreasing southwards. This may be caused by the low vapor pressure and water solubility of DDTs that the transport via wet precipitation was less significant (Shen and Wania 2005).

In maize seed and stem, total content of HCHs residue was in the range of 14.8–18.6 and 32.6–70.3 ng g^{-1}, respectively. The sequence for the contents of HCH isomers in stem samples is as follows: δ-HCH > γ-HCH > β-HCH > α-HCH. The geometric mean level of main isomer δ-HCH was 9.69 ng g^{-1} in seed samples and 14.8 ng g^{-1} in stem samples, indicating a long-year α-HCH degradation in the area. The averaged ratio of α-HCH/γ-HCH in maize seeds and stems were 0.91 and 0.50 respectively, suggesting a usage of lindane in the maize fields. The averaged ratio of *o,p′*-DDT/*p,p′*-DDT in maize seeds and stems was 2.47 and 1.51 respectively, which hints a few usage of technical kelthane in study area. PCB28 was the predominant species in PCB isomers. Similar with the distribution characteristics in rice samples, the total OCPs and PCBs concentrations in maize stem were higher than those in maize seed and maximum of PCBs occurred in Hailong County of Meihekou City.

The concentrations of OCPs and PCBs in rice field water were also investigated (data not shown). The geometric mean level of total OCPs concentration in Meihekou area

Table 2 OCPs and PCBs concentrations in rice stem samples (ng g^{-1})

Number	DBSD1 (Stem)	DBSD2 (Stem)	DBSD8 (Stem)	DBSD7 (Stem)	DBSD5 (Stem)	DBSD4 (Stem)	Average	SD*
α-HCH	2.1	1.3	2.6	4.4	0.2	1.7	2.0	1.4
β-HCH	7.2	6.9	10.7	9.3	0.5	4.6	6.5	3.6
γ-HCH	10.6	6.1	8.7	16.3	3.4	3.7	8.1	4.9
δ-HCH	13.5	5.1	20.2	21.2	6.5	6.7	12.2	7.2
α-HCH/γ-HCH	0.2	0.2	0.3	0.3	0.1	0.4	0.2	0.1
∑HCHs	33.4	19.5	42.1	51.2	10.5	16.7	28.9	15.9
PCB-28	10.3	1.3	3.2	15.8	3.3	3.0	6.1	5.7
PCB-52	0.8	3.5	47.5	7.6	2.9	5.1	11.2	17.9
PCB-101	7.5	6.5	11.4	4.9	0.7	15.8	7.8	5.2
PCB-118	nd	nd	1.1	nd	nd	1.3	1.2	0.1
PCB-138	17.4	16.3	9.8	17.0	7.8	5.8	12.4	5.2
PCB-153	8.9	4.6	1.7	30.5	1.2	4.0	8.5	11.1
PCB-180	2.4	2.0	2.2	4.3	0.3	4.1	2.5	1.5
∑PCBs	47.2	34.2	76.9	80.0	16.3	39.1	49.0	25.0
p,p′-DDE	1.9	2.5	13.8	2.7	1.8	11.1	5.6	5.4
p,p′-DDD	9.4	10.4	2.5	8.1	7.6	12.9	8.5	3.5
o,p′-DDT	6.0	5.2	54.4	6.4	5.5	6.1	13.9	19.8
p,p′-DDT	8.9	5.6	1.2	10.6	nd	nd	6.5	4.2
DDD/DDE	5.0	4.2	0.2	3.0	4.1	1.2	2.9	1.9
(DDD + DDE)/DDT	1.3	2.3	14.1	1.0	-	-	4.7	6.3
∑DDTs	26.2	23.7	71.8	27.8	14.9	30.1	32.4	20.0

*SD** Standard deviation; *nd* not detected

(0.849 ng g^{-1}) is higher than that in Jilin area (0.178 ng g^{-1}). The total HCH concentrations were higher than the total DDT concentrations, whether in Jilin City area or in Meihekou area. The reason for the phenomenon may be attributed to the higher octanol–water partition coefficient (K_{ow}) of DDTs than that of HCHs and corresponding higher water solubility of HCHs than that of DDTs (Shen and Wania 2005).

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