

Full Length Research Paper

Distribution of 19 organochlorinated pesticides residues in ginseng and soils in Jilin Province, China

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The organochlorinated pesticides widely applied and still remains in soils, has become toxic to ginseng production in Jilin Province. In this study, 19 trace organochlorinated pesticide residues in five types of ginseng products and soil samples from four ginseng production areas were analyzed using a gas chromatographic-mass spectrometry (GC-MS) method for simultaneous measurement. The ginseng and soil samples were extracted with acetonitrile. The extracts were cleaned up by solid phase extraction using a column of 6 ml Florisil. The 19 organochlorinated pesticides were separated through a DB-17 MS capillary column and were measured by GC-MS. The recoveries rate ranged from 71 to 112%. The results show that organochlorinated pesticides residues ranged from 0.01 to 0.04 mg/kg in these samples. Endosulfan I and endosulfan II were 0.04 mg/kg in the four places. Higher-levels organochlorinated pesticides residues were alpha-benzene hexachloride (α -BHC), pentachloronitrobenzene (PCNB), endosulfan I and endosulfan II in five kinds of ginseng products. Except for α -BHC, preserved fresh ginseng had very low concentration of organochlorinated pesticides residues. Mould pressing steamed red ginseng, white ginseng with fibrous root, red and white ginseng all showed high residue concentration of α -BHC, PCNB, endosulfan I and endosulfan II. This indicated that the hyperaccumulation of organochlorinated pesticides residues occurred in ginseng. It was therefore suggested that α -BHC, PCNB, endosulfan I and endosulfan II should not be used in ginseng production in Jilin Province.

Key words: Gas chromatographic-mass spectrometry, ginseng, organochlorinated pesticide residues, solid-phase extraction.

INTRODUCTION

Organochlorinated pesticides (OCPs) have been used for agricultural purposes for decades and they are distributed widely across China. Although these pesticides have never

been used in large quantities after they have been banned, studies indicate that their residues still persist in the top layer soils and in many places in China (Xu et al., 2004, 2006; Tang et al., 2008). These phenomenon were due to atmospheric redistribution (Shegunovaa et al., 2007), bioaccumulation by plant or animals and decays pattern of organochlorinated pesticides (Shen et al., 2005).

Ginseng is a slow-growing perennial plant with fleshy

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roots, in the *Panax* genus, in the family *Araliaceae*. It grows in the Northern Hemisphere in Eastern Asia, typically in cooler climates. The clinically active compound in ginseng is ginsenosides, and it has been used as an expensive nourishing stimulants or medicine in the treatment of type II diabetes, as well as sexual dysfunction in men. Jilin Province is one of the most important ginseng production bases in China. In the ginseng production, organochlorinated pesticide residues in ginseng and soil received more and more concerned because their toxic potential to the consumers. At present, the maximum pesticide limit in international market was downgraded from 0.2 to 0.01 µg/g of OCPs. Hence, reliable methods with high sensitivity, low detection limits are needed in pesticide residues measurement and remediation of OCPs in ginseng and soil (Can et al., 2004; Chen et al., 2007).

Unfortunately, traditional methods including isotope dilution gas chromatography-mass spectrometry (GC-MS) (Chan et al., 2007), the solid-phase extraction and capillary gas chromatography assays (SPE-CGC) (Chen et al., 2007; Chu et al., 2006), and the solid-phase extraction and gas chromatography assays (SPE-GC) (Hou et al., 2007) could not determine organochlorinated pesticide residues effectively. This was partly because the complexity of ginseng substance resulted in many false positives results determined by the GC method, and also, these methods could not determine multiple kinds of organochlorinated pesticide residues simultaneously. Besides, the measurements of pesticide residues of commonly used endosulfan and procymidone are still unknown. Allena et al. (1999) determined the total and free *trans*-39-hydroxycotinine and cotinine in the urine of subjects receiving transdermal nicotine by GC-MS method. Afterwards, this accurate method was applied in the studies of steroidal estrogens in flushed dairy manure wastewater (Hanselman et al., 2006), volatile fraction of ewes' dairy products (Povolo et al., 2007), and free thyroid hormones in serum (Yue et al., 2008).

In this study, we used a new solid-phase extraction and GC-MS method that can simultaneously determine 19 organochlorinated pesticides residues, to determine organochlorinated pesticide residues both in five kinds of ginseng products and four planting areas where ginseng are planted. Our results show that the contaminations of organochlorinated pesticide residues in soils were lower than those in ginseng products.

MATERIALS AND METHODS

Collection of samples

The samples comprised five kinds of ginseng products namely, mould pressing steamed red ginseng, white ginseng with fibrous root, red ginseng, white ginseng and preserved fresh ginseng. The soil in Fusong county, Baishan city, Changbai country and Wanliang town were also sampled at 15 cm depth and were collected at July

2008 and July 2009, respectively. These samples were weighed, dried, shattered, and then filtered through a 0.245-mm sieve.

Apparatus and reagents

A Milli-Q integral Pure/Ultrapur water production unit, Agilent 6890N GC-5973 Inert MSD GC-MS with electron bombardment ionization (EI), solid phase extraction apparatus, SHZ-88 constant temperature water bath oscillator, and a SPE Sep-Pak Florisil (1 000 mg, 6 ml, Agilent Co. Ltd., USA) were used in the determination of pesticides. Acetone, acetonitrile, hexane, pentachlorobenzene (PCB), hexachlorobenzene (HCB), alpha-benzene hexachloride (α -BHC), pentachloronitrobenzene (PCNB), γ -BHC, β -BHC, heptachlor (HEPT), δ -BHC, chlorothalonil (CTO), aldrin, procymidone, endosulfan I, p, p'-dichloro-diphenyl-dichloroethylene (p,p'-DDE), dieldrin, endrin, p,p'-dichloro-diphenyl-dichloroethane (p,p'-DDD), o, p'-dichloro-diphenyl-trichloroethane (o,p-DDT), endosulfan II, p,p'-DDT standard samples were purchased from Sigma Co. Ltd. Germany); Heptachlor-exo-epoxide (cis-, isomer B) was used as internal standard (Dr. E Co, Ltd. Germany).

Procedures

0.1 to 10 mg/L Heptachlor-epoxide used as internal standard was dissolved by acetone. The Sep-Pak Florisil column was pre-diluted by 5 ml of an acetone/hexane mixture (10:90, v/v) followed by 5 ml of hexane, respectively. Vials containing 10 g of the filtrated soil and ginseng sample were added to 20 ml pure water and 50 ml acetonitrile, and introduced into an oscillator. After a period of sample equilibration (30 min), 40 ml solution was filtered through filter paper, and then collected by a new vial containing 5 g NaCl. This was vibrated for 1 min, after which samples were stabled for 10 min at room temperature to enable the separation of acetonitrile and water into different layers. Then the upper acetonitrile was transferred to a new 100 ml breaker, put it into a water bath with 80°C and air-dried with nitrogen. Furthermore, 2.0 ml of hexane was added into it and the beaker was covered with aluminum foil. The hexane solution was put into equilibrium column, diluted with 5 ml of acetone/hexane (15: 85, v/v) for three times, air dried with nitrogen. Finally, 1 ml of petroleum ether and 1 ml 0.1 mg/L of heptachlor-epoxide was added into dry ash immediately, shaken up, and then determined by GC-MS.

GC-MS

Pesticide extract solutions were injected into an Agilent 6890N GC-5973 Inert MSD GC-MS equipped with EI and a computer operating system with the program (MSD Productivity Chemstation D.02.00). A DB-17 MS flexibility-silica capillary column was used (30 m length, 0.25 mm inside diameter, 0.25 µm film thickness; from Agilent, USA). The operation conditions were as follows: the oven temperature was set initially at 60°C (1-min hold), then increased to 180°C at 5°C/min (10-min hold), and finally increased to 260°C at 5°C/min (20-min hold); the temperatures of injector port, the ion source, the quadrupole mass analyzer, and the detector were kept at 250, 230, 150 and 280°C, respectively; and splitless mode was used for injection with a volume of 1 µL; mass spectra were recorded at an ionization energy of 70 eV; data were collected by selected ion monitoring (SIM).

The recovery tests were as follows: one sample was added to 0.05, 0.25 and 2.0 mg/kg standard solutions, purified and determined by the methods described above. The recovery rate was calculated according to peak area. All experiments were repeated

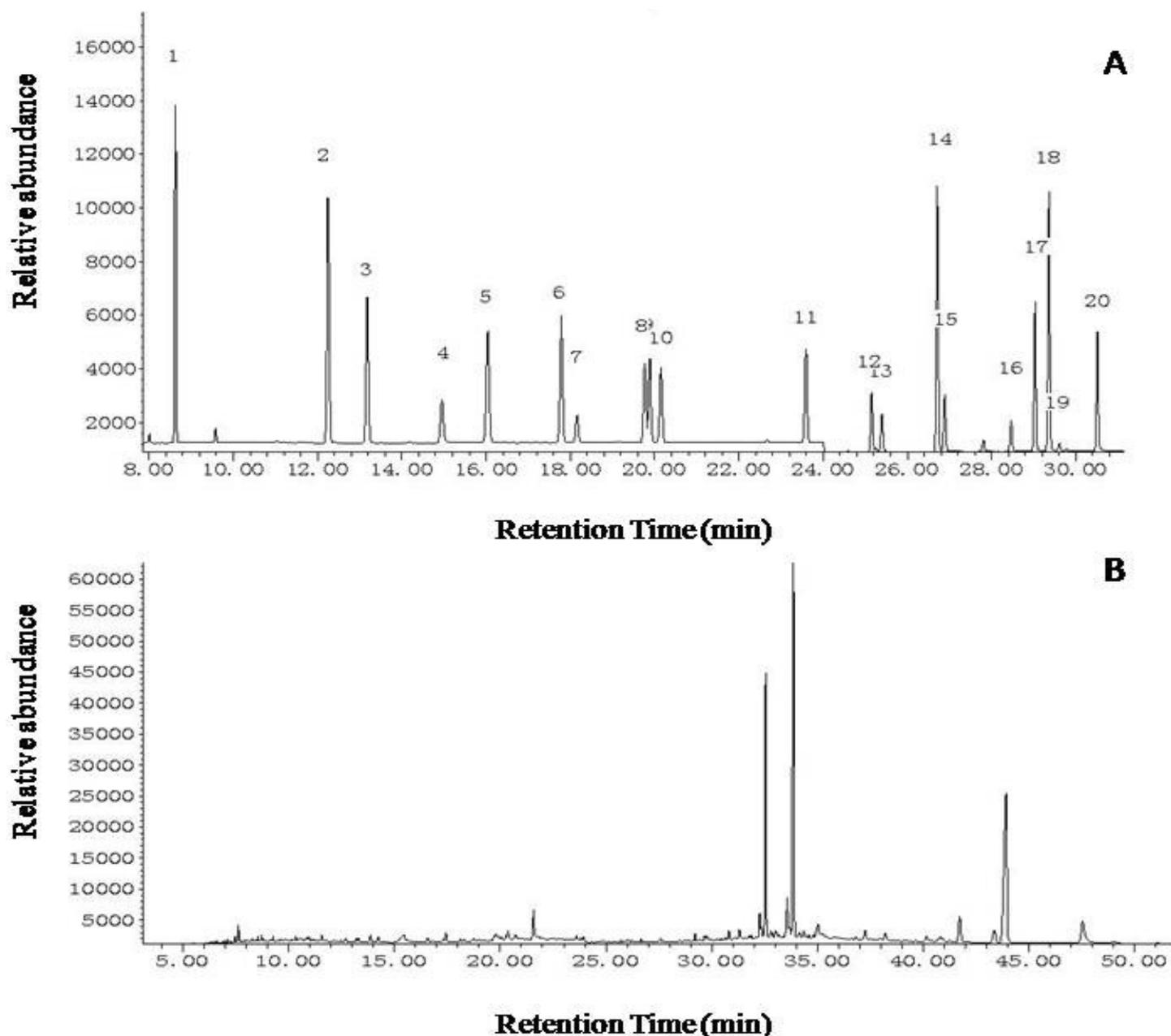


Figure 1. SIM chromatogram of the organochlorinated pesticides in standard (A) and in mould pressing steamed red ginseng (B). 1, PCB; 2, HCB; 3, α -BHC; 4, PCNB; 5, γ -BHC; 6, β -BHC; 7, HEPT; 8, δ -BHC; 9, CTO; 10, aldrin; 11, HCE; 12, procymidone; 13, endosulfan I; 14, *p, p'*-DDE; 15, endrin; 16, di-endrin; 17, *o, p'*- DDT; 18, *p, p'*- DDD; 19, endosulfan II; 20, *p, p'*- DDT. BHC, Benzene hexachloride; *p, p'*-DDE, *p, p'*-dichloro-diphenyl-dichloroethylene; *p, p'*-DDD, *p, p'*-dichloro-diphenyl-dichloroethane; *o, p'*- DDT, *o, p'*-dichloro-diphenyl-trichloroethane; PCB, pentachlorobenzene; HCB, hexachlorobenzene; PCNB, pentachloronitrobenzene; HEPT, heptachlor; CTO, chlorothalonil; SPE, solid-phase extraction; RSD, relative standard deviation; SIM, selected ion monitoring; HCE, hexachloroethane.

five times and relative standard deviation (RSD) was calculated by Excel software to show the degree of precision.

RESULTS AND DISCUSSION

Chromatogram of the mixed organochlorinated pesticides

standard and internal standard recorded by SIM are shown in Figure 1. The whole retention time in GC-MS was 35 min, while the pre-treatment time was about 60 min. All 19 components were identified. Their retention times, the ion used for the quantification in each case are shown in Table 1. Semi-quantification was based on the

Table 1. Retention times and characterization of the representative pesticides.

S/N	Pesticide	Retention time (min)	Quantification ion (m/z)	Qualities ion (m/z)		
1	PCB	8.63	250 (100)	252 (64)	215 (24)	
2	HCB	12.25	284 (100)	286 (81)	282 (51)	
3	α -BHC	13.18	219 (100)	183 (98)	221 (47)	254 (6)
4	PCNB	14.96	295 (100)	237 (159)	249 (114)	
5	γ -BHC	16.05	183 (100)	219 (93)	254 (13)	221 (40)
6	β -BHC	17.79	219 (100)	217 (78)	181 (94)	254 (12)
7	HEPT	18.16	272 (100)	237 (40)	337 (27)	
8	δ -BHC	19.78	219 (100)	217 (80)	181 (99)	254 (10)
9	CTO	19.88	266 (100)	264 (72)	268 (49)	
10	Aldrin	20.15	263 (100)	265 (65)	293 (40)	
11	HCE	23.60	353 (100)	355 (75)	351 (52)	
12	Procymidone	25.16	283 (100)	285 (70)	255 (15)	
13	Endosulfan I	25.40	241 (100)	265 (66)	339 (46)	
14	p,p'-DDE	26.71	318 (100)	316 (80)	246 (39)	248 (70)
15	Dieldrin	26.89	263 (100)	277 (82)	380 (30)	345 (35)
16	Endrin	28.46	263 (100)	317 (30)	345 (26)	
17	o,p-DDT	29.03	235 (100)	237 (63)	165 (37)	199 (14)
18	p,p'-DDD	29.36	235 (100)	237 (64)	199 (12)	165 (46)
19	Endosulfan II	29.61	241 (100)	265 (66)	339 (46)	
20	p,p'-DDT	30.51	235 (100)	237 (65)	246 (7)	165 (34)

BHC, Benzene hexachloride; p,p'-DDE, p, p'-dichloro-diphenyl-dichloroethylene; p, p'-DDD, p,p'-dichloro-diphenyl-dichloroethane; o, p'-DDT, o, p'-dichloro-diphenyl-trichloroethane; PCB, pentachlorobenzene; HCB, hexachlorobenzene; PCNB, pentachloronitrobenzene; HEPT, heptachlor; CTO, chlorothalonil; SPE, solid-phase extraction; RSD, relative standard deviation; SIM, selected ion monitoring; HCE, hexachloroethane.

base specific peak ion area counts divided by internal standard. Tables 2 gives the average recovery rates obtained for components along with the average deviations in each case. As depicted in Table 2, the recovery rates were 71 to 110%, 82 to 112% and 75 to 112% when the 0.05, 0.25, and 2.0 mg/kg standard solution were added, respectively. The detection limits of the 19 organochlorinated pesticides were lower than 0.01 mg/kg which could meet the criterion of international market.

Hou et al. (2007), used gas chromatography (GC) method for the trace analysis of PCNB in ginseng. According to their results, only 4 pesticides could be detected simultaneously, and the average recoveries were within 90.7 to 97.8% and RSD was 0.7 to 2.4%. Chen et al. (2007) used the solid-phase extraction and capillary gas chromatography to determine 19 organochlorinated pesticide residues in preserved ginseng, ginseng honey plate and ginseng tea. And from their results, the average recoveries ranged was from 73.7 to 96.6%. In this paper, at three different internal solutions, average recoveries ranged from 71 to 110%, 82 to 112%, and 75 to 112%, respectively. It was therefore concluded that this GC/MS was more convenience and accurate than simply using GC method (Mezcua et al., 2009).

As shown in Table 3, PCNB and procymidone were

excess in the standard of international market, while other pesticides met the standard of international market, namely less than 0.02 mg/kg. This was particularly because most of pesticides were no longer in use, or because most pesticide was used on leaves of ginseng. However, the concentration of one of the commonly used CTO was lower, thus suggesting that it was easily dissipated in soil and should be used to replace PCNB. As shown in Table 4, preserved fresh ginseng had high concentrations of α -BHC, PCNB, and procymidone, while other pesticides were low in it. However, mould pressing steamed red ginseng had high concentrations of α -BHC, PCNB, endosulfan I and endosulfan II. White ginseng with fibrous root had high concentrations of PCB, HCB, α -BHC, PCNB, endosulfan I, and endosulfan II. White ginseng had high concentrations of PCB, HCB, α -BHC, PCNB, γ -BHC, δ -BHC, procymidone, endosulfan I, endrin and endosulfan II. Red ginseng had high concentrations of α -BHC, PCNB, γ -BHC, δ -BHC, procymidone, endosulfan I and endosulfan II. These results suggest that accumulation of organochlorinated pesticides were common characteristics in ginseng. More attention should therefore be paid on the proper use of organochlorinated pesticides in ginseng production. The precise assays of organochlorine pesticide residues concentrations by this methods was

Table 2. Average recoveries of 19 pesticides [means \pm relative standard deviation (RSD), n = 5].

Pesticide	Standard solution added		
	0.05 mg/kg	0.25 mg/kg	2.0 mg/kg
PCB	75 \pm 19.5	84 \pm 11.9	75 \pm 10.4
HCB	72 \pm 3.9	82 \pm 9.7	79 \pm 4.8
α -BHC	110 \pm 15.0	94 \pm 12.6	110 \pm 2.1
PCNB	78 \pm 2.4	86 \pm 5.3	92 \pm 2.9
γ -BHC	73 \pm 13.9	98 \pm 12.4	112 \pm 6.2
β -BHC	71 \pm 4.0	100 \pm 6.6	106 \pm 4.7
HEPT	71 \pm 7.7	112 \pm 7.8	111 \pm 5.4
δ -BHC	77 \pm 9.7	101 \pm 9.5	98 \pm 4.4
CTO	79 \pm 10.6	102 \pm 8.2	87 \pm 9.9
Aldrin	99 \pm 14.3	94 \pm 7.6	82 \pm 6.0
Procymidone	97 \pm 7.2	89 \pm 6.5	86 \pm 1.4
Endosulfan I	99 \pm 10.8	90 \pm 9.3	84 \pm 6.6
p,p'-DDE	78 \pm 12.7	102 \pm 9.8	103 \pm 7.3
Dieldrin	80 \pm 19.2	94 \pm 2.5	94 \pm 11.1
Endrin	87 \pm 13.1	98 \pm 16.1	87 \pm 4.4
o,p-DDT	81 \pm 11.7	96 \pm 8.5	86 \pm 9.6
p,p'-DDD	89 \pm 16.9	103 \pm 17.1	102 \pm 9.8
Endosulfan II	87 \pm 11.9	98 \pm 18.7	89 \pm 7.9
p,p'-DDT	98 \pm 15.3	90 \pm 9.3	89 \pm 4.4

Table 3. Pesticide residue in soils of 4 places in Jilin Province (mg/kg).

Pesticide	Songshan county	Baishan city	Chanbai county	Wanliang town
PCB	≤ 0.010	≤ 0.010	≤ 0.010	≤ 0.010
HCB	≤ 0.010	0.013	≤ 0.010	≤ 0.010
α -BHC	≤ 0.010	≤ 0.010	≤ 0.010	≤ 0.010
PCNB	≤ 0.020	0.022	0.031	≤ 0.020
γ -BHC	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020
β -BHC	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020
HEPT	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020
δ -BHC	≤ 0.020	≤ 0.020	≤ 0.020	0.022
CTO	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020
Aldrin	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020
Procymidone	≤ 0.020	≤ 0.020	≤ 0.020	0.024
Endosulfan I	≤ 0.040	≤ 0.040	≤ 0.040	≤ 0.040
p,p'-DDE	≤ 0.015	≤ 0.015	≤ 0.015	≤ 0.015
Dieldrin	≤ 0.025	≤ 0.025	≤ 0.025	≤ 0.025
Endrin	≤ 0.025	≤ 0.025	≤ 0.025	≤ 0.025
o,p-DDT	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020
p,p'-DDD	≤ 0.015	≤ 0.015	≤ 0.015	≤ 0.015
Endosulfan II	≤ 0.040	≤ 0.040	≤ 0.040	≤ 0.040
p,p'-DDT	≤ 0.020	≤ 0.020	≤ 0.020	≤ 0.020

important in the determination and put forward remediation strategy of organochlorinated pesticide residues in ginseng production.

Conclusion

In this study, GC-MS method for the simultaneous

Table 4. Organochlorinated pesticides residues in Panax ginseng (mg/kg).

Pesticide	Mould pressing steamed red ginseng	White ginseng with fibrous root	Red ginseng	White ginseng	Preserved fresh ginseng
PCB	0.021	0.022	≤0.010	0.023	≤0.010
HCB	0.015	0.023	≤0.010	0.045	≤0.010
α-BHC	0.032	0.041	0.078	0.051	0.051
PCNB	0.023	0.112	0.063	0.112	0.021
γ-BHC	≤0.020	0.021	0.032	0.031	≤0.020
β-BHC	≤0.020	≤0.020	≤0.020	≤0.020	≤0.020
HEPT	0.024	≤0.020	≤0.020	≤0.020	≤0.020
δ-BHC	≤0.020	≤0.020	0.023	0.120	≤0.020
CTO	≤0.020	≤0.020	≤0.020	≤0.020	≤0.020
Aldrin	≤0.020	≤0.020	≤0.020	≤0.020	≤0.020
Procymidone	≤0.020	0.021	0.026	0.076	0.021
Endosulfan I	0.041	0.042	0.045	0.062	≤0.020
p,p'-DDE	≤0.015	≤0.015	≤0.015	≤0.015	≤0.015
Dieldrin	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025
Endrin	≤0.025	≤0.025	≤0.025	≤0.025	≤0.025
o,p-DDT	≤0.020	≤0.020	≤0.020	≤0.020	≤0.020
p,p'-DDD	≤0.015	≤0.015	≤0.015	≤0.015	≤0.015
Endosulfan II	0.042	0.044	0.078	0.096	≤0.020
p,p'-DDT	≤0.020	≤0.020	≤0.020	≤0.020	≤0.020

measurement of 19 trace organochlorinated pesticide residues in five Panax ginseng and soil samples from four locations was reported. Compared to solid-phase extraction and capillary gas chromatography, this method is rapid, sensitive and suitable for the analysis of organochlorinated pesticide residues in ginseng and soil on a larger scale. Although the organochlorinated pesticide residues in the four planting areas were low, the organochlorinated pesticide residues contents in ginseng products were high. This study is helpful for organochlorinated pesticide residues remediation of soil, as well as improvements of ginseng production in Jilin Province, china.

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Abbreviations: BHC, Benzene hexachloride; p,p'-DDE, p, p'-dichloro-diphenyl-dichloroethylene; p, p'-DDD, p,p'-dichloro-diphenyl-dichloroethane; o, p'- DDT, o, p'-dichloro-diphenyl-trichloroethane; PCB, pentachlorobenzene; HCB, hexachlorobenzene; HCE, hexachloroethane; PCNB, pentachloronitrobenzene; HEPT, heptachlor; CTO, chlorothalonil; SPE, solid-phase

extraction; RSD, relative standard deviation; SIM, selected ion monitoring.

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