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A Multiresidue Method for 20 Pesticides in *Radix paeoniae* Alba of Chinese Herb by Gas Chromatography with Electron-capture Detection

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Abstract A rapid multiresidue method was developed for the determination of 20 pesticides in *Radix paeoniae* Alba of Chinese herb by ultrasonic wave extraction, silica gel column chromatography and gas chromatography (GC) with electron-capture detection (ECD) in this study, Mean recoveries of the method ranged from 74.45 to 115.14%. The validation of the proposed approach was verified on *Isatis indigotica* Fort, *Pltycodon grandiflorum*, *Cotex mouta* and *Poria cocos* of Chinese herbs; good recoveries were also obtained in the range of 72.51–113.47%, respectively.

Keywords *Radix paeoniae* Alba · Pesticides · Multi-residue analysis

The use of herbal plants for medicines or food ingredients in China has been prevalent for thousands of years, which have become an important part of life in Chinese society (Vania and Janete 2000). Because of insect pests and diseases, pesticides, such as organochlorine pesticides, are used to combat them (Rong et al. 2006). The enormous use of organochlorine pesticides has been serious concern because of their persistant nature. The absorption of these pesticides in foods and herbs can be considered as a risk for human health due to the chronic toxicity of most of these compounds.

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In recently years, many new analytical methodologies of pesticide multiresidue in different matrices (e.g., vegetable, fruit) were reported, such as solid-phase micro extraction (SPME) (Hwang and Lee 2000), supercritical fluid extraction (SFE) (Zhao et al. 2006), matrix solid-phase dispersion micro-extraction (MSPD) (Abhilash and Singh 2008), solid phase extraction (SPE) (Wang et al. 2008), and microwave-assisted extraction (Shashi and Gregory 2004). The residue detection of pesticides has been carried out by GC, LC, GC–MS and LC–MS (Susana et al. 2006). These new methods are efficient and safe. However, many of these extraction and detection methods are costly, time-consuming and require special equipment. Moreover, there is hardly any analytical method of multi-residue in *Radix paeoniae* Alba, which is routinely used as an herb in China for dietary and medical purposes. The present study deals with developing a rapid and efficient multiresidue analysis method for the determination of the 20 pesticides in *Radix paeoniae* Alba and the validation of the proposed approach.

Materials and Methods

Certified analytical standards of pesticides, purity >99%, were obtained from the Institute for the Control of Agrochemicals, Ministry of Agriculture, P.R. China. Anhydrous sodium sulfate was analytical grade reagents (Shanghai Chemical Reagents Co.). All solvents used were of analytical grade, among them acetone, dichloromethane and petroleum were obtained from Shanghai Chemical Reagents Co. (Shanghai, China). Silica gel, heated 10 h at 130°C before use, was purchased from Qingdao Chemical Reagents Co. (Qingdao, China). Medicinal plants *Radix paeoniae* Alba, *Isatis indigotica* Fort, *Pltycodon*

grandiflorum, *Cotex mouta* and *Poria cocos* were purchased from the Herb Corporation of Bozhou (Bozhou, China).

An Agilent 6890 gas chromatograph (America) equipped with ^{63}Ni electron capture detector (ECD) was used. A fused silica capillary column (HP-5), 5% phenyl polysiloxane as nonpolar stationary phase (30 m × 0.32 mm i.d.) and 0.25 μm film thickness, supplied by Phenomenex (Torrance, CA, USA), was employed. The temperatures of the injector and the detector were maintained at 275 and 300°C, respectively. The oven temperature was programmed to increase from 50 to 150°C at a rate of 20°C/min and from 150 to 280°C at a rate of 4°C/min before being held at 280°C for 10 min the initial holding time at 50°C was 1 min, Nitrogen (N_2, 99.999% purity) was used as carrier gas, flow rate of 1.5 mL/min. Injection volume of sample was 2 μL.

Stock solutions (1,000 mg/L) of each pesticide were prepared in acetonitrile and stored in dark glass vials at −18°C. The stock solutions were further diluted with acetonitrile to make a mixed intermediate pesticide solution that contained each isomer of HCH and DDT at 100 mg/L; chlorothalonil, α-endosulfan, vinclozolin, 100 mg/L; fenpropathrin, chlorpyrifos, fipronil, 200 mg/L; and phorate, dimethoate, deltamethrin, λ-cyhalothrin, s-fenvalerate, befenthrin, 300 mg/L. Different volumes of the mixed intermediate solution were diluted with petroleum ether to prepare the working solutions used for calibration and for fortification in recovery studies. All solutions were stored in dark glass vials at 4°C.

Herbs were ground mechanically to homogeneous powder and sieved through a No. 40 mesh sieve. For extraction procedure, 5.0 g of such powder samples were measured into a beaker and 40 mL of acetone/dichloromethane (2/1, v/v)was added. The samples were extracted for 15 min (8 and 7 min, respectively) in ultrasonic extraction instrument at room temperature. The samples were equilibrated for the separation of the solvents and metric, and the organic phase was filtered and concentrated to near 1 mL with a vacuum rotary evaporator. Then the concentrated extract was transferred to a silica gel (7.5 g) column (25 cm × 1.5 cm i.d.) that was pre-washed with petroleum ether. The silica gel column was eluted by 20 mL mixture solvent of petroleum ether/acetone (40/60, v/v). Eluates were collected and concentrated to near dryness by vacuum rotary evaporator. Thereafter, the extract was reconstituted in 5 ml of petroleum ether for final analysis. For recovery evaluation purposes, herb samples were fortified by adding a mixture standard of 20 pesticides to obtain serial concentration (Table 1). After the fortification, the samples were kept at room temperature to equilibrate for 2 h prior before extraction. Five replicates at each fortification level, including un-spike controls, were extracted, cleaned-up as described above and analyzed by GC–ECD.

Results and Discussion

In an earlier study, the theory and methodology of orthogonal array design (OAD) for optimization of analytical procedures have been emplied. Many factors, which controlled the efficiency of supersonic extraction from solid matrices and cleaned-up of extract were considered, including solvent of extraction, time of supersonic extraction, the volume of extraction solvent, cleaned-up material, the volume of eluting solvent and the eluting solvent, etc. The cleaned-up material was most important to organochlorine pesticides, and the eluting solvent was most important to pyrethroid pesticides. Thinking of the recovery rate, cost and applicability, the optimal extract factors were 40 mL solvent of acetone/dichloromethane (2/1, v/v), 15 min (8 and 7 min, respectively) of supersonic extraction and the optimal cleaned-up method was established that the concentrated extract was transferred to a silica gel (7.5 g) column (25 cm × 1.5 cm i.d.) that was pre-washed with petroleum ether. The silica gel column was eluted by 20 mL mixture solvent of petroleum ether/acetone (40/60, v/v). Pesticides residue levels were determined by GC/ECD (Fig. 1). The ECD response for all of the 20 pesticides was linear in the dosages range (0.02–30 ng) assayed with correlation coefficients (r) > 0.999, except β-HCH, δ-HCH, chlorothalonil and λ-cyhalothrin >0.995. The limits of detection (LOD) and the limits of quantification (LOQ) of different analytes are shown in Table 2. LODs of method ranged between 0.1 μg/kg for γ-HCH and 2.5 μg/kg for β-HCH in the 20 pesticides, LOQs were varied from 0.4 to 8.2 μg/kg for various analytes studied. Mean recoveries for different pesticides were found in the range of 74.45–115.14% and relative standard deviation (RSD) were all lower 10.0% (Table 1). Comparing with the study of Wu et al. (2006), the recovery efficiency and LOQ of the proposed approach is similar or even better; 20 pesticides were analyzed simultaneously in our study, including organochlorine, pyrethroid, Organophosphorus and fungicide, however, Wu JL researched only 15 pesticides of organochlorine and pyrethroid. Moreover, the extract time was only 15 min in present study, which was much shorter than that of 6 h in research of Wu et al. (2006). In addition, fewer pesticides were studied in experiment of Tang et al. (2006). In contrast with the method of Chinese Pharmacopeia (The Chinese Pharmacopoeia Commission 2005), organochlorine and pyrethroid pesticides were analyzed by the developed method simultaneously, but according to the method of Chinese Pharmacopeia, these pesticides must be analyzed, respectively.

Application of this method was verified by analyzing four other herbal medicines. Good recovery rates were also obtained in *Isatis indigotica* Fort, *Pltycodon grandiflorum*, *Cotex mouta* and *Poria cocos* (Table 2). The mean

Table 1 The recovery rates of 20 pesticides in *Radix paeoniae* Alba, *Isatis indigotica* Fort, *Pltycodon grandiflorum*, *Cotex mouta* and *Poria cocos*

Pesticides	Concentration (mg/kg)	Recovery (%) ± RSD (%) (n = 5)				
		Radix paeoniae	*Poria cocos*	*Isatis Indigotica*	*Pltycodon grandiflorum*	*Cotex mouta*
α-HCH	5.00	110.39 ± 9.76	82.67 ± 7.05	83.52 ± 1.25	82.58 ± 1.00	80.25 ± 4.92
	1.00	81.16 ± 0.66	80.38 ± 3.98	83.23 ± 5.37	87.32 ± 1.02	88.47 ± 2.09
	0.05	93.08 ± 6.90	110.16 ± 7.33	89.29 ± 9.50	93.79 ± 7.21	92.98 ± 10.83
β-HCH	5.00	95.52 ± 6.52	82.07 ± 1.31	85.33 ± 5.71	81.84 ± 0.87	90.15 ± 2.90
	1.00	83.99 ± 2.57	86.81 ± 10.9	82.78 ± 10.12	80.12 ± 0.95	97.04 ± 1.43
	0.05	90.61 ± 7.60	86.92 ± 9.84	90.60 ± 7.82	92.06 ± 4.29	79.25 ± 3.43
γ-HCH	5.00	101.12 ± 4.07	81.54 ± 5.06	85.59 ± 3.60	84.53 ± 0.63	86.83 ± 3.53
	1.00	85.08 ± 0.04	84.28 ± 5.48	80.46 ± 6.30	83.75 ± 0.19	110.02 ± 2.71
	0.05	115.14 ± 4.03	106.25 ± 13.51	86.77 ± 13.22	106.16 ± 4.42	81.86 ± 1.51
δ-HCH	5.00	113.30 ± 3.37	80.83 ± 1.57	82.07 ± 4.28	91.51 ± 1.27	90.70 ± 4.21
	1.00	80.66 ± 3.28	82.77 ± 4.28	83.71 ± 6.54	86.43 ± 0.25	91.57 ± 6.14
	0.05	92.77 ± 5.10	81.50 ± 7.62	100.35 ± 3.23	86.78 ± 6.18	91.17 ± 4.79
Chlorothalonil	5.00	89.63 ± 6.90	101.48 ± 5.91	80.26 ± 2.08	105.94 ± 1.88	83.59 ± 10.81
	1.00	88.61 ± 5.67	86.40 ± 11.57	82.11 ± 5.63	83.38 ± 2.08	116.71 ± 1.48
	0.05	108.99 ± 5.93	86.90 ± 7.36	81.68 ± 7.05	94.98 ± 7.48	94.89 ± 6.01
Vinclozolin	5.00	85.15 ± 6.07	84.40 ± 0.93	85.78 ± 2.98	84.06 ± 3.02	85.74 ± 5.18
	1.00	90.91 ± 5.10	85.17 ± 3.37	80.95 ± 5.91	82.26 ± 1.47	102.17 ± 5.11
	0.05	86.89 ± 1.87	80.43 ± 12.71	91.62 ± 2.20	94.33 ± 7.09	83.70 ± 6.80
α-Endosulfan	5.00	95.61 ± 0.29	85.60 ± 11.19	89.21 ± 2.03	90.53 ± 2.00	88.35 ± 5.62
	1.00	100.47 ± 5.63	83.29 ± 7.23	84.00 ± 6.19	85.32 ± 0.50	84.65 ± 2.08
	0.05	88.10 ± 4.25	78.07 ± 8.44	96.97 ± 6.38	84.32 ± 6.49	88.94 ± 6.26
pp′DDE	5.00	89.89 ± 1.89	90.74 ± 7.14	87.60 ± 2.26	91.08 ± 2.58	91.39 ± 6.58
	1.00	98.76 ± 4.87	77.62 ± 4.63	105.83 ± 9.13	92.26 ± 0.37	84.29 ± 3.71
	0.05	84.12 ± 5.04	86.89 ± 4.71	86.96 ± 2.66	90.98 ± 5.42	83.18 ± 8.79
pp′DDD	5.00	93.18 ± 2.30	82.58 ± 0.77	86.41 ± 3.93	90.49 ± 3.70	87.92 ± 3.31
	1.00	92.17 ± 7.74	81.36 ± 2.59	87.24 ± 5.66	91.36 ± 3.48	88.00 ± 4.98
	0.05	85.15 ± 8.22	81.21 ± 0.10	79.87 ± 4.01	89.87 ± 5.63	84.58 ± 11.23
op′DDT	5.00	96.93 ± 1.80	92.22 ± 0.16	87.77 ± 2.48	91.08 ± 0.22	87.57 ± 3.24
	1.00	92.20 ± 5.07	82.55 ± 5.65	82.31 ± 3.28	84.46 ± 2.97	84.30 ± 3.40
	0.05	84.28 ± 6.34	83.63 ± 10.09	87.14 ± 3.92	104.08 ± 7.44	82.84 ± 9.85
pp′DDT	5.00	98.59 ± 1.72	91.76 ± 1.30	90.56 ± 2.42	91.00 ± 1.09	85.20 ± 1.46
	1.00	88.17 ± 6.07	79.59 ± 5.31	84.18 ± 2.61	81.33 ± 6.13	86.58 ± 0.36
	0.05	77.76 ± 5.40	84.16 ± 5.00	88.46 ± 0.29	87.07 ± 5.33	84.56 ± 9.05
Fenpropathrin	10.0	110.39 ± 9.76	83.21 ± 1.05	87.45 ± 0.27	92.15 ± 1.24	86.10 ± 5.45
	2.00	81.16 ± 0.66	84.76 ± 0.10	83.32 ± 2.96	87.82 ± 3.30	83.80 ± 4.46
	0.10	93.08 ± 6.90	83.84 ± 6.28	85.86 ± 5.46	83.79 ± 2.53	81.70 ± 10.41
Chlorpyrifos	10.0	83.53 ± 3.26	82.96 ± 1.91	86.06 ± 0.71	89.02 ± 2.88	81.77 ± 8.77
	2.00	91.62 ± 2.98	81.47 ± 3.78	83.93 ± 10.61	87.55 ± 0.89	92.20 ± 4.18
	0.10	86.50 ± 1.10	78.90 ± 6.29	97.09 ± 4.20	89.11 ± 6.20	80.89 ± 3.87
Fipronil	10.0	83.49 ± 6.74	88.55 ± 4.48	80.48 ± 2.17	92.69 ± 0.20	83.81 ± 6.29
	2.00	88.09 ± 7.15	81.78 ± 2.27	81.63 ± 11.38	85.07 ± 1.92	88.79 ± 7.11
	0.10	83.53 ± 0.45	84.00 ± 12.68	80.27 ± 1.11	95.87 ± 2.81	87.27 ± 6.50
Phorate	15.0	90.09 ± 9.74	82.74 ± 6.00	95.56 ± 2.25	83.18 ± 4.43	95.18 ± 2.19
	3.00	84.59 ± 1.05	89.38 ± 3.25	94.96 ± 8.35	87.07 ± 0.21	95.12 ± 2.16
	0.15	101.64 ± 8.55	96.94 ± 1.77	95.51 ± 3.75	94.45 ± 3.05	80.67 ± 9.07

Table 1 continued

Pesticides	Concentration (mg/kg)	Recovery (%) ± RSD (%) (n = 5)				
		Radix paeoniae	*Poria cocos*	*Isatis Indigotica*	*Pltycodon grandiflorum*	*Cotex mouta*
Dimethoate	15.0	77.59 ± 4.45	78.79 ± 5.67	78.63 ± 9.89	80.15 ± 6.75	85.24 ± 5.92
	3.00	76.13 ± 8.21	79.25 ± 2.08	81.51 ± 1.44	72.51 ± 2.56	75.97 ± 9.89
	0.15	74.45 ± 8.53	73.25 ± 9.92	74.14 ± 7.71	74.17 ± 7.95	74.85 ± 8.87
Befenthrin	15.0	76.45 ± 8.73	87.44 ± 0.56	90.94 ± 1.46	93.12 ± 0.08	90.78 ± 0.88
	3.00	84.59 ± 1.05	88.12 ± 3.82	85.03 ± 1.73	84.12 ± 1.12	80.41 ± 0.89
	0.15	101.64 ± 9.55	86.65 ± 11.98	80.62 ± 8.00	82.53 ± 7.01	80.30 ± 3.98
λ-Cyhalothrin	15.0	95.52 ± 6.52	81.95 ± 0.93	95.08 ± 2.47	91.52 ± 0.66	85.44 ± 3.83
	3.00	83.99 ± 2.57	90.26 ± 5.34	89.58 ± 2.12	88.88 ± 0.02	89.36 ± 6.75
	0.15	90.61 ± 7.60	77.31 ± 11.24	79.50 ± 6.00	83.30 ± 10.41	78.05 ± 6.30
s-Fenvalerate	15.0	113.30 ± 3.37	83.63 ± 1.47	93.97 ± 2.96	92.24 ± 5.24	84.56 ± 10.63
	3.00	80.66 ± 3.28	85.53 ± 2.35	82.67 ± 6.30	90.21 ± 2.84	81.34 ± 2.14
	0.15	92.77 ± 5.10	79.63 ± 2.64	80.89 ± 11.08	98.46 ± 6.63	80.51 ± 6.08
Deltamethrin	15.0	85.15 ± 6.07	83.31 ± 2.18	92.85 ± 5.80	91.67 ± 0.68	86.04 ± 9.18
	3.00	90.91 ± 5.10	94.25 ± 4.13	90.07 ± 4.36	91.49 ± 4.80	88.20 ± 10.52
	0.15	86.89 ± 1.87	85.34 ± 5.26	94.47 ± 10.16	83.28 ± 8.32	78.29 ± 6.96

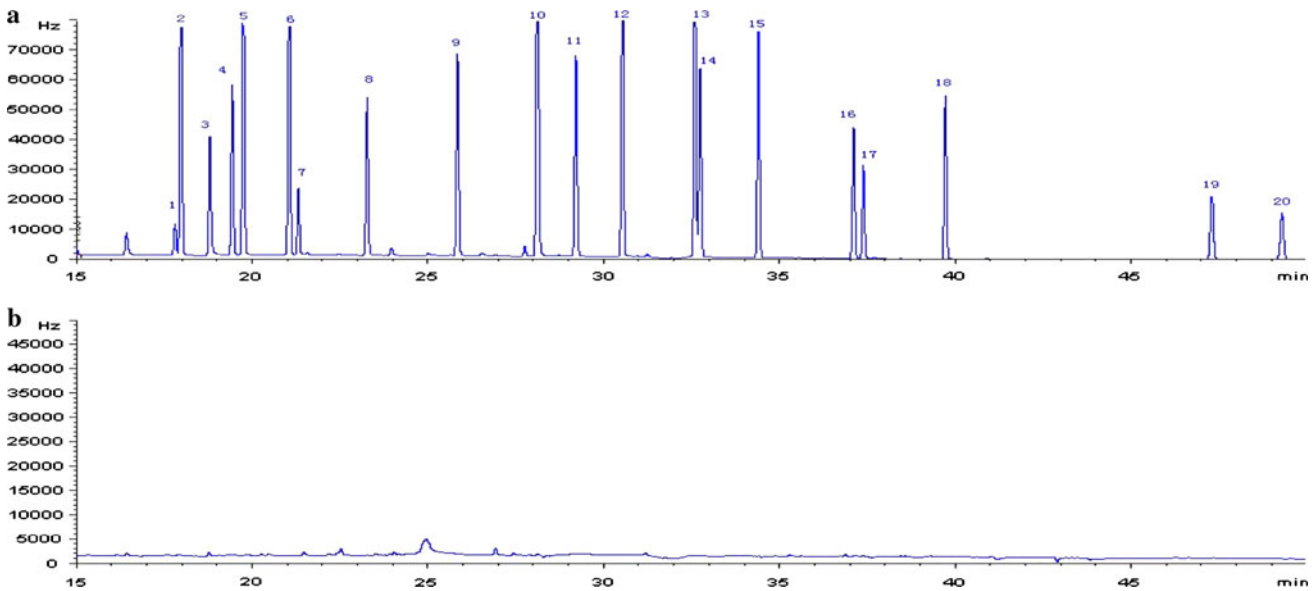

Fig. 1 Capillary GC-ECD chromatograms of 20 pesticides. **a** Mixture solution of 20 pesticides for separation of standards; **b** extract solution of *Radix paeoniae* Alba for the effect of metric; *1* phorate (3.0 mg/kg), *2* α-HCH (1.0 mg/kg), *3* dimethoate (1.0 mg/kg), *4* β-HCH (1.0 mg/kg), *5* γ-HCH (1.0 mg/kg), *6* δ-HCH (1.0 mg/kg), *7* chlorothalonil (1.0 mg/kg), *8* vinclozolin (1.0 mg/kg), *9* chlorpyrifos (2.0 mg/kg), *10* fipronil (2.0 mg/kg), *11* α-endosulfan (1.0 mg/kg), *12* pp′DDE (1.0 mg/kg), *13* pp′DDD (1.0 mg/kg), *14* op′DDT (1.0 mg/kg), *15* pp′DDT (1.0 mg/kg), *16* befenthrin (3.0 mg/kg), *17* fenpropathrin (2.0 mg/kg), *18* λ-cyhalothrin (3.0 mg/kg) *19* s-fenvalerate (3.0 mg/kg), *20* deltamethrin (3.0 mg/kg)

recoveries obtained from 72.51 to 113.47%, and RSDs < 14.0%. Therefore, these indicates that the optimized method could be used to supervise and monitor 20 pesticides residue in *Radix paeoniae* Alba, *Isatis indigotica* Fort,*Pltycodon grandiflorum*, *Cotex mouta* and *Poria cocos*.

To versify the developed method, samples of *Radix paeoniae* Alba from the field were analyzed using our newly developed multiresidue detection method. The result demonstrated a few of the samples contains the residue of chlorpyrifos, the level was 0.026 ± 0.003 mg/kg, which is lower the maximum residue limit (MRL 0.2 mg/kg) (Chen

Table 2 The regression equation, linear range, correlation coefficients and instrument LODs for 20 pesticides in a *Radix paeoniae* Alba matrix sample

Pesticides	Retention time	Regression equation	Linear (ng)	r	LOD (μg/kg) n = 5	LOQ (μg/kg) n = 5
Phorate	17.794	$y = 2143.6x + 541.52$	0.06–30	0.9998	2.2	7.3
α-HCH	17.984	$y = 84383x + 2624$	0.02–10	0.9999	0.3	1.0
Dimethoate	18.787	$y = 28094x + 3481.9$	0.06–30	0.9999	0.5	1.8
β-HCH	19.423	$y = 43575x - 8701.1$	0.02–10	0.9965	2.5	8.2
γ-HCH	19.724	$y = 84069x + 2748.7$	0.02–10	0.9999	0.1	0.4
δ-HCH	21.077	$y = 72375x - 8831.2$	0.02–10	0.9986	0.4	1.2
Chlorothalonil	21.303	$y = 32163x - 4274.8$	0.02–10	0.9975	1.2	4.1
Vinclozolin	23.266	$y = 35403x - 1592.8$	0.02–10	0.9991	0.6	2.1
Chlorpyrifos	25.830	$y = 25116x + 3766.9$	0.04–20	0.9999	1.0	3.3
Fipronil	28.109	$y = 42093x - 1623$	0.04–20	0.9997	0.3	1.1
α-Endosulfan	29.193	$y = 49886x + 4370.5$	0.02–10	0.9995	0.5	1.8
pp′DDE	30.517	$y = 69480x - 892.52$	0.02–10	0.9997	1.0	3.2
pp′DDD	32.590	$y = 67739x - 2129.6$	0.02–10	0.9996	0.2	0.7
op′DDT	32.727	$y = 49905x + 1693.7$	0.02–10	0.9998	0.3	1.1
pp′DDT	34.394	$y = 58162x - 3756.2$	0.02–10	0.9992	0.3	1.0
Befenthrin	37.094	$y = 10372x + 2886.9$	0.06–30	0.9998	2.2	7.2
Fenpropathrin	37.373	$y = 14858x - 470.46$	0.04–20	0.9995	0.4	1.4
λ-Cyhalothrin	39.702	$y = 14562x - 2246.7$	0.06–30	0.9989	0.3	1.1
s-Fenvalerate	47.282	$y = 6280.1x - 338.23$	0.06–30	0.9995	1.0	3.3
Deltamethrin	49.274	$y = 5500x - 3.015$	0.06–30	0.9997	1.4	4.7

y the area of peak, x the quantity of injection (ng)

and Jia 2005) of European regulation, and the other pesticides were not detected.

In general, the prompt analytical methods would be quite useful for other regulatory guidelines in the management of pesticide residues in herbal products. Considering the above aspects, a rapid, simple, sensitive, and versatile method has been developed for the determination of 20 pesticides in herbs.

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