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Determination of multiple pesticide residues in animal foods using on-line gel permeation chromatography-gas chromatography/mass spectrometry

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Introduction

An analytical method for the determination of multiple pesticide residues in animal foods by modified QuEchERS method with on-line gel permeation chromatography/gas chromatography/mass spectrometry (GPC/GC/MS). Targets were 21 pesticides: methamidophos, dichlorvos, α-BHC, β-BHC, γ-BHC, δ-BHC, dimethoate, terbufos, chlorpyrifos-methyl, metolachlor, isocarbophos, p, p'-DDE, p, p'-DDD, o, p'-DDT, p, p'-DDT, phentriazophos, permethrin, cypermethrin, flucythrinate, fenvalerate, and deltamethrin. The samples were extracted from homogenized foods with acetonitrile assisted by n-hexane, and separated with liquid-liquid partition. The supernatant liquid was purified by PSA and C18 to remove most of the fats and pigments in samples, then after on-line GPC-GC/MS analysis which further removed macromolecular interference material, such as fat, the background interference brought about by the complex matrix in samples was effectively reduced. At the spiked level of 0.02 mg/kg, recoveries for most of pesticides were from 72.9% to 118.6%, and the relative standard deviations ranged from 0.88% to 9.67%. The limitis of detection and the limitis of quantification were 0.6~2.5 μg/kg and 2.0~8.4 μg/kg, respectively. The method is simple, rapid and characterized with acceptable sensitivity and accuracy to meet the requirements for the analysis of multiple pesticide residues in animal food.

Experimental

Sample pretreament

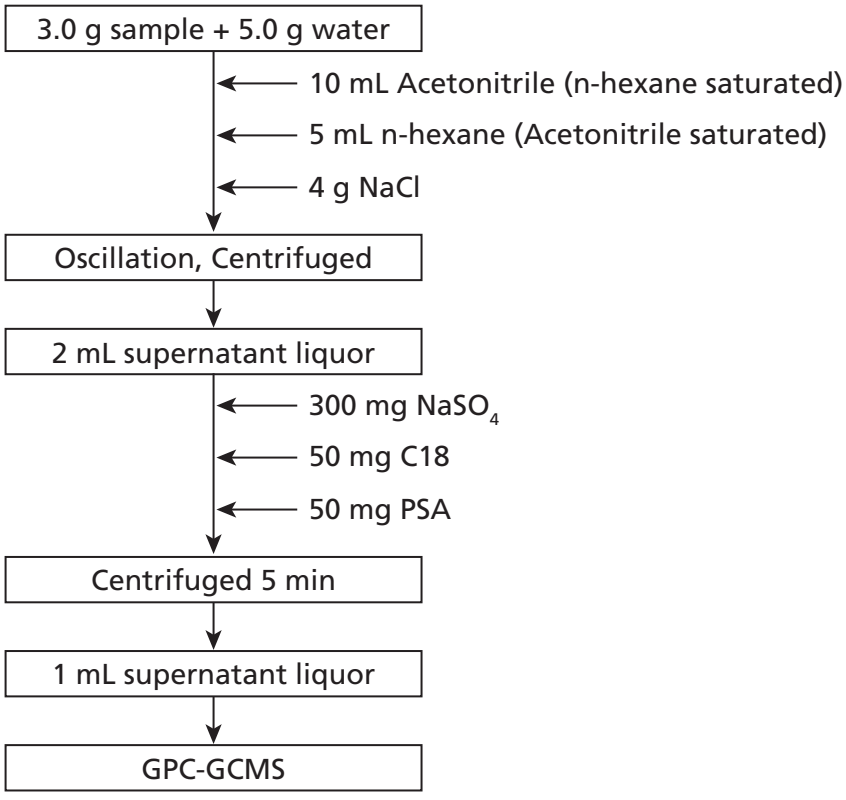

Figure 1 Schematic flow diagram of the sample preparation

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Instrument

Analytical Conditions

GPC	
Mobile phase	: Acetone/Cyclohexane (3/7 by volume)
Flow rate	: 0.1mL/min
Column	: Shodex CLNpak EV-200 (2mmx150mm)
Oven temperature	: 40 °C
Injection Volume	: 10 µL

GCMS	
Column	: deactivated silica tubing [0.53mm (ID)x5m (L)] +pre-column Rxi-5ms [0.25mm (ID)x5m (L)] Rxi-5ms [0.25mm (ID)x30m (L),Thickness 0.25µm]
Injector	: PTV
Injector time program	: 120 °C (5min)-(100 °C/min)-250 °C (33.7min)
Oven temperature program	: 82 °C (5min) (8 °C/min)-300 °C (8min)
Linear velocity	: 45.0cm/sec
Ion Source temperature	: 230 °C
Interface temperature	: 300 °C

Figure 2 Shimadzu GPC-GCMS

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Results

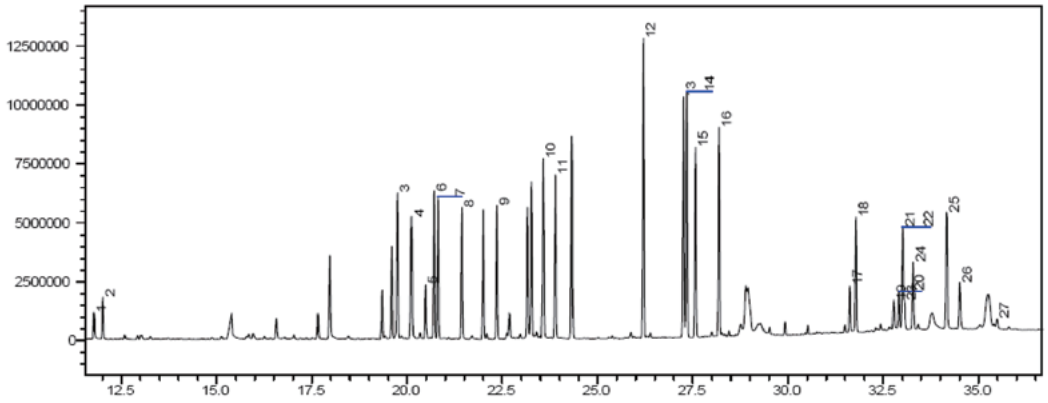

Figure 3 Chromatograms of SIM from mix standards

Table 1 Retention times, linear equation, correlation coefficients, limitis of detection (LODs, S/N≥3), limitis of quantification (LOQs, S/N≥10), the averager recoveries and the relative standard deviations (RSDs, n=3) for pesticides

No.	pesticide	t_R (min)	Correlation Coefficient*	LOD (μg/kg)	LOQ (μg/kg)	pork		shrimp	
						Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
1	Methamidophos	11.800	0.9993	1.8	6.0	72.9	2.99	83.4	4.52
2	Dichlorvos	11.983	0.9999	0.6	2.0	106.3	4.47	106.6	2.87
3	α-BHC	19.742	0.9999	0.8	2.7	97.3	8.22	91.4	6.83
4	Dimethoatee	20.108	0.9995	1.8	6.0	98.4	7.79	96.3	6.87
5	β-BHC	20.475	0.9999	1.7	5.7	95.6	6.80	97.8	6.43
6	γ-BHC	20.708	0.9999	0.8	2.7	105.2	4.22	98.0	6.15
7	Terbufos	20.817	0.9999	0.8	2.7	99.3	3.52	95.5	6.42
8	δ-BHC	21.433	0.9999	1.7	5.7	119.4	9.67	100.0	6.70
9	Chlorpyrifos-methyl	22.358	0.9999	1.6	5.4	118.6	9.52	98.3	3.58
10	Metolachlor	23.567	0.9999	1.6	5.4	110.4	3.71	95.8	4.71
11	Isocarbopho	23.883	0.9993	2.1	7.0	110.7	3.21	93.1	5.11
12	p, p'-DDE	26.192	0.9999	0.8	2.7	73.0	0.88	70.1	4.98
13	p, p'-DDD	27.250	0.9999	0.8	2.7	106.4	2.67	103.4	3.62
14	o, p'-DDT	27.342	0.9998	0.8	2.7	86.5	7.88	87.9	7.25
15	Phentriazophos	27.575	0.9996	1.0	3.4	108.6	4.25	98.7	6.07
16	p, p'-DDT	28.192	0.9998	1.6	5.4	99.0	1.32	100.9	6.93
17	Permethrin-1	31.617	0.9998	1.8	6.0	93.4	3.85	90.8	4.98
18	Permethrin-2	31.783							
19	Cypermethrin-1	32.767	0.9996	1.9	6.4	98.4	3.85	95.4	5.80
20	Cypermethrin-2	32.900							
21	Cypermethrin-3	33.000							
22	Cypermethrin-4	33.050							
23	Flucythrinate-1	33.000	0.9996	1.8	6.0	102.6	1.97	88.6	6.25
24	Flucythrinate-2	33.275							
25	Fenvalerate-1	34.167	0.9997	1.8	6.0	104.7	3.50	101.1	7.25
26	Fenvalerate-1	34.500							
27	Deltamethrin	33.475	0.9988	2.5	8.4	106.1	3.42	93.3	5.75

* Concentration range: 10μg/L~500μg/L

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Conclusion

A based on automated online gel permeation chromatography coupled with gas chromatography/mass spectrometry (GPC-GC/MS) method was developed for the determination of multiple pesticide residues in animal foods by QuEchERS method. So provides a simple, rapid, and reliable method for the detection of pesticide residues in animal foods.

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