QuEChERS-HPLC-MS/MS Method for Determination of Residues of Multiple Fungicides in Animal-derived Foods

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Abstract [**Objectives**] This study was conducted to improve the purification agent combination conditions for QuEChERS and establish a method for detecting the residues of 18 fungicides in animal-derived foods (fish, pork, milk, eggs, and pork liver) using high-performance liquid chromatography-triple quadrupole tandem mass spectrometry. [**Methods**] The samples were extracted with acetonitrile, purified with 885 mg of magnesium sulfate, 150 mg of PSA (ethylenediamine-N-propylsilane silica gel), and 15 mg of GCB (graphitized carbon black), and analyzed using ACQUITY UPLC BEH C_{18} as the chromatographic column with a mobile phase of acetonitrile-0.1% formic acid aqueous solution. [**Results**] The 18 fungicides showed good linearity in the range of 5.00 - 200.00 μg/L, with correlation coefficients (R^2) greater than 0.991. The limit of quantification (LOQ) was 0.01 mg/kg. The average recoveries ranged from 63.7% to 117.5%, and the relative standard deviations (RSDs) were between 0.22% and 6.33%. [**Conclusions**] This method is simple, rapid, and highly accurate, and provides technical reference for the detection and risk assessment of fungicides in animal-derived foods.

Key words QuEChERS; HPLC-MS/MS; Animal-derived; Fungicide

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Fungicides, also known as biocides, bactericidal algaecides, or antimicrobial agents, generally refer to chemical agents that can effectively control or kill bacteria, fungi, and algae in water systems. Internationally, they are commonly used as a general term for agents that prevent and control various pathogenic microorganisms. Research on their residue levels in vegetables and fruits is relatively well-established[1-2], while studies in animal-derived foods remain at a preliminary stage, and only partial fungicides have been reported. QuEChERS (quick, easy, cheap, effective, rugged, safe) is a rapid sample pretreatment technique for agricultural product testing that has emerged in recent years. It was developed in 2003 by Professor Anastassiades from the U.S. Department of Agriculture. This method is primarily used in combination with gas chromatography-mass spectrometry (GC-MS)[3-6] and liquid chromatography-tandem mass spectrometry (LC-MS/MS)[7-9] for pesticide residue analysis. It has been applied to detect residues in milk[10-12], tea[13-15], plant-derived foods[16-17], and animal-derived foods[18-20]. In this study, a method for simultaneously determining residues of 18 fungicides in animal-derived foods was developed by optimizing the QuEChERS purification agent combination and combining it with liquid chromatography-tandem mass spectrometry (LC-MS/MS). The approach supplements existing research on fungicides in animal-derived products, offering a simple, accurate, and reliable analytical technique. It provides technical reference for the detection and regulation of fungicide residues in animal-derived foods.

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Materials and Methods

Equipment and agents

Waters Xevo TQ-Micro liquid chromatograph-triple quadrupole mass spectrometer with electrospray ionization (ESI) source (Waters Corporation, USA); ME203E/02 analytical balance (Mettler Toledo Instruments (Shanghai) Co., Ltd.); H1850 centrifuge (Hunan Xiangyi Centrifuge Instrument Co., Ltd.); GZY-P120-B ultrapure water system (Hunan Kertone Water Group Co., Ltd.); AutoEVA-60 automated parallel concentrator (Evertech Technology, Guangzhou); VORTEX-6 vortex mixer (Kylin-Bell Lab Instruments Co., Ltd.).

Acetonitrile (HPLC grade); formic acid (HPLC grade); sodium chloride; magnesium sulfate (analytical grade); PSA (ethylenediamine-N-propylsilane silica gel); GCB (graphitized carbon black); C_{18} (octadecylsilane-bonded silica gel) adsorbent; 0.22 μm organic filter membranes (ANPEL Laboratory Technologies Inc., Shanghai); ultrapure water prepared using the GZY-P120-B water purification system; carbendazim, ametoctradin, triadimefon, triadimenol, imazalil, flutriafol, tebuconazole, dimethomorph, trifloxystrobin, mandipropamid, metalaxyl, flusilazole, fenbuconazole, propiconazole, penthiopyrad, isopyrazam, prochloraz, and difenoconazole, all purchased from GRG Metrology & TEST (Hunan) Co., Ltd.

Preparation of standard solutions Preparation of standard stock solution: An appropriate amount of each reference standard was accurately weighed into a 10 ml volumetric flask, and diluted to constant weight with acetonitrile to obtaine a stock solution with a concentration of 1.00 mg/L, which was cold-stored at 0 - 4 ℃.

Preparation of mixed standard working solution: A specific volume of the standard stock solutions or standard solutions were precisely transferred into a 10 ml volumetric flask, and diluted to constant volume with acetonitrile to obtain an intermediate

standard solution with a concentration of 1.00 μg/L, which was stored at 0 – 4 ℃.

Preparation of matrix-matched standard working solutions: An appropriate volume of the mixed standard working solution was accurately transferred into six portions of pre-extracted and purified blank matrix, respectively. After evaporation with nitrogen gas to dryness in a water bath, 1 ml of acetonitrile was added to each sample, which was then vortexed for dissolution, resulting in matrix-matched standard working solutions with concentrations of 10, 20, 50, 80, 100, and 200 ng/ml.

Experimental Methods

Sample Preparation Fish meat: The head, internal organs, and bones were removed, and a 200 g of edible sample including skin and muscles was homogenized and cryopreserved in a polyethylene bottle. Eggs: Fifteen eggs were shelled, homogenized, and thoroughly mixed. The egg liquid was cryopreserved in a polyethylene bottle. Milk: A 200 ml of milk sample was thoroughly mixed and cryopreserved in a polyethylene bottle. Pork and pork liver: A 200 g of muscle or internal organ sample was homogenized and cryopreserved in a polyethylene bottle.

Sample pretreatment A 2 g of sample was weighed into a 50 ml centrifuge tube. Then, 4 g of sodium chloride and 20 ml of acetonitrile were added, followed by vortex mixing for 1 min. The mixture was subjected to ultrasonic extraction for 30 min and centrifuged at 4 000 r/min for 5 min. An 8 ml aliquot of the supernatant was transferred to a 15 ml plastic centrifuge tube containing 885 mg of anhydrous magnesium sulfate, 150 mg of PSA and 15 mg of GCB. The mixture was vortexed for 1 min and centrifuged at 4 000 r/min for 5 min. A 5 ml portion of the supernatant was evaporated to near dryness with nitrogen gas. The residue was reconstituted in 2 ml of acetonitrile, thoroughly mixed, and filtered through a membrane for instrumental analysis.

Instrumental conditions

Chromatographic conditions A C_{18} column (1.8 μm, 2.1 × 100 mm) was used for HPLC separation. The separation was performed with an injection volume of 10 μl using 0.1% formic acid aqueous solution as mobile phase A and acetonitrile as mobile phase B at a flow rate of 0.4 ml/min and a column temperature of 40 ℃. The gradient elution program is shown in Table 1.

Table 1 Gradient elution program for the mobile phase

Time//min	0.1% formic acid aqueous solution	Acetonitrile
0	90	10
0.50	90	10
1.00	80	20
2.50	10	90
4.00	10	90
4.20	90	10
5.00	90	10

MS conditions MS conditions: Ion source type and temperature: ESI, 150 ℃; ionization mode: positive/negative switching; curtain gas: 30 psi; ionization voltage: 5 500 V; desolvation temperature: 550 ℃; nebulizer gas: 55 psi; auxiliary heater gas: 55 psi; acquisition mode: multiple reaction monitoring (MRM). Detailed MS parameters are listed in Table 2.

Table 2 MS parameters of the 18 fungicides

Item	Parent ion//m/z	Daughter ion//m/z	Declustering voltage//V	Collision voltage// V
Carbendazim	192.1	132.1	30	28
		160.1*	30	18
Ametoctradin	276.1	175.9*	60	30
		149.1	60	35
Triadimefon	294.1	197.1*	14	15
		225.1	14	8
Triadimenol	296.1	70.0*	10	12
		227	10	9
Imazalil	297.1	159.0*	18	23
		201	18	16
Flutriafol	302.1	109	25	32
		123.0*	25	27
Tebuconazole	308.2	70.0*	36	15
		125	36	26
Dimethomorph	388.1	165.1	45	21
		301.1*	45	13
Trifloxystrobin	409.1	145	14	45
		186.1*	14	15
Mandipropamid	412.1	328.0*	30	15
		356	30	8
Metalaxyl	280.1	192.1	30	17
		220.1*	30	13
Flusilazole	316.1	165.1*	28	19
		247	28	13
Fenbuconazole	337.1	70.0*	13	22
		125	13	37
Propiconazole	342.1	123	18	56
		159.0*	18	32
Penthiopyrad	360.1	256	16	16
		276.0*	16	10
Isopyrazam	360.2	244.1*	14	16
		340.2	14	12
Prochloraz	378.1	70.1*	30	25
		310.1	30	20
Difenoconazole	406.1	251	13	22
		337.0*	13	13

* indicates quantitative ions.

Matrix effect

In MS quantitative analysis, interference from other components in the sample may lead to deviations in the results for target analytes. The evaluation method for matrix effect in this study is shown in Formula 1.

$$x = \left| \left(\frac{b}{b_1} - 1 \right) \times 100 \right| \quad (1)$$

In the formula, x is the absolute value of matrix effect (%); b is the slope of matrix-matched standard curve; and b_1 is slope of solvent standard curve. For the classification of matrix effect, $x > 50\%$ indicates strong matrix effect; $20\% \leqslant x \leqslant 50\%$ indicates moderate matrix effect; and $x < 20\%$ indicates weak matrix effect.

Data processing

MassLynx software was used for instrument signal analysis. Quantification was performed using the peak areas of fungicide quantitative ions with the external standard method.

Results and Discussion

Optimization of chromatographic conditions

Chromatographic columns determine the resolution between target compounds. Columns with different stationary phases exhibit varying separation efficiency and sensitivity for substances. Even columns with the same stationary phase may demonstrate different separation performances for identical compounds due to manufacturing process variations among different manufacturers. In this study, the peak profiles of 18 fungicides on following three chromatographic columns were compared: ACQUITY UPLC BEH C_{18} (1.7 μm, 2.1 mm × 100 mm), SHIMSEN Ankylo C_{18}-NC (2.6 μm, 2.1 mm × 100 mm), and Kinetex C_{18} (2.6 μm, 2.1 × 100 mm). The 50 ng/ml intermediate standard solution was analyzed using 0.1% formic acid aqueous solution and acetonitrile as mobile phases with gradient elution. The results indicated no difference in the elution order of the 18 fungicides on different chromatographic columns, though slight variations in retention time were observed for certain compounds. When using the ACQUITY UPLC BEH C_{18} column, penthiopyrad, ametoctradin, mandipropamid, and isopyrazam exhibited narrower peak widths, while propiconazole, metalaxyl and triadimenol showed the largest peak areas and highest sensitivity. Consequently, the ACQUITY UPLC BEH C_{18} column was selected for the residual analysis of these 18 fungicides.

Optimization of QuEChERS conditions

PSA (ethylenediamine-N-propylsilane silica gel), GCB (graphitized carbon black) and C_{18} (octadecylsilane-bonded silica gel) are currently the most commonly used adsorbent materials for impurity purification in QuEChERS methods, primarily employed to remove interfering substances such as organic acids, pigments, and sugars. While these adsorbent materials effectively capture impurities, they may also potentially adsorb target analytes. In this study, the adsorption effects of PSA, C_{18} and GCB on target compounds were investigated. First, 10 ml of mixed fungicide working solution with concentration of 20 ng/ml was prepared. Next, 1.5 ml aliquots were transferred into three 2 ml centrifuge tubes containing 10 mg of PSA, C_{18}, or GCB, respectively. After purification and centrifugation, the concentrations of target compounds were determined. The experimental data are shown in Fig. 1. The results demonstrated that C_{18} showed varying degrees of adsorption towards ametoctradin and penthiopyrad, while PSA and GCB exhibited minimal adsorption effects on the target compounds. Consequently, the combination of PSA and GCB was selected as the optimal adsorbent purification materials.

Matrix effect

Negative samples of grass carp, milk, eggs, pork liver and pork were selected. Matrix-matched standard curves were prepared following the sample pretreatment method described in section "Sample pretreatment" and analyzed simultaneously with solvent standard curves. The test results are presented in Table 3.

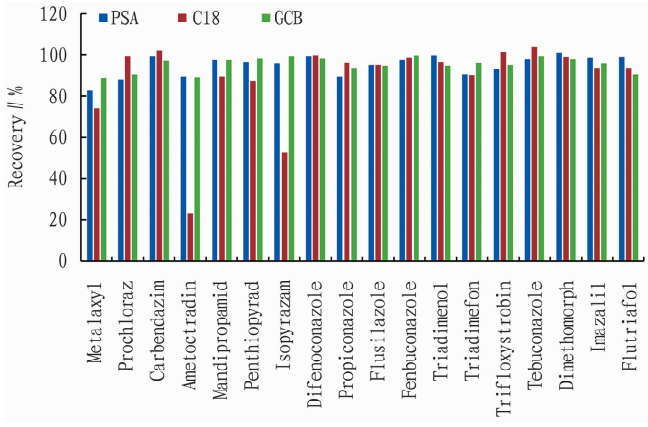

Fig. 1 Recovery data of different purification reagents

Table 3 Matrix effects of 18 fungicides in different matrices %

Fungicide	Grass carp	Pork	Egg	Milk	Pork liver
Metalaxyl	61	60	64	62	61
Prochloraz	73	76	76	73	75
Carbendazim	94	94	94	94	94
Ametoctradin	70	70	75	73	71
Mandipropamid	79	79	81	79	78
Penthiopyrad	32	34	31	24	32
Isopyrazam	47	38	48	48	39
Difenoconazole	29	51	23	13	47
Propiconazole	29	27	23	24	24
Flusilazole	45	47	44	43	40
Fenbuconazole	42	40	40	37	36
Triadimenol	84	80	79	80	83
Triadimefon	50	54	52	53	50
Trifloxystrobin	57	44	46	52	49
Tebuconazole	69	56	65	66	58
Dimethomorph	65	65	67	64	67
Imazalil	58	58	58	56	57
Flutriafol	59	63	59	59	57

As shown in Table 3, the absolute values of matrix effects for 18 fungicides in different matrices were generally greater than 20%, indicating that the matrices significantly influenced the accuracy of quantitative analysis. Twelve fungicides exhibited strong matrix effects, with carbendazim showing the most pronounced effect (absolute values >90% in all cases). Six fungicides demonstrated moderate matrix effects. Therefore, in this study, matrix-matched standard curves were employed to calibrate the contents of target analytes, so as to minimize the impact of matrix effects to the greatest extent.

Limits of detection, limits of quantitation and linear ranges

Five different types of blank samples (fish, milk, eggs, pork liver, and pork) were selected to prepare matrix-matched standard curves following the sample pretreatment method described in section "Sample pretreatment". The results demonstrated good linearity for all 18 fungicides within the range of 5.00 – 200.00 μg/L, with correlation coefficients (R^2) exceeding 0.991. The limits of detection (LODs) and quantification (LOQs) were calculated based on the signal-to-noise ratio (S/N) at the lowest spiked concentration.

Table 4 Recovery and precision data of 18 fungicides ($n=6$) %

Fungicide	Spiked concentration μg/kg	Grass carp		Pork		Egg		Milk		Pork liver	
		Recovery	*RSD*	Recovery	*RSD*	Recovery	*RSD*	Recovery	*RSD*	Recovery	*RSD*
Metalaxyl	20	90.1	1.21	85.6	2.31	103.4	2.76	97.2	0.97	95.0	2.51
	50	93.0	2.13	95.6	0.54	97.1	3.97	101.7	3.87	99.6	5.42
	100	84.8	3.54	87.2	1.65	100.2	4.69	94.0	2.98	95.5	4.30
Prochloraz	20	90.6	2.05	83.7	0.89	117.2	5.30	107.3	1.03	102.1	2.19
	50	110.8	3.54	108.1	0.33	97.3	0.98	90.4	1.89	115.9	0.59
	100	86.4	4.30	105.8	1.37	91.6	6.33	97.6	1.57	103.2	4.32
Carbendazim	20	73.3	1.09	103.8	2.87	84.5	1.07	92.5	1.86	117.5	1.98
	50	96.7	0.93	99.8	2.69	84.4	4.34	109.5	0.68	84.2	2.59
	100	78.8	5.01	81.7	4.82	90.8	2.69	96.6	4.69	100.5	0.48
Ametoctradin	20	89.6	2.98	80.0	2.60	96.8	5.39	111.3	2.89	93.0	2.86
	50	92.2	3.22	99.9	5.39	90.0	6.01	101.9	2.95	88.6	1.78
	100	89.7	1.93	87.0	4.38	82.8	3.21	85.5	0.58	94.8	2.85
Mandipropamid	20	90.0	2.67	98.5	2.35	95.1	3.27	104.8	1.35	102.8	3.21
	50	114.9	3.25	97.7	4.68	80.9	3.88	100.6	1.66	68.4	3.86
	100	101.4	1.11	111.7	3.29	81.8	3.90	72.5	1.23	93.8	1.93
Penthiopyrad	20	83.5	4.20	92.1	4.32	77.8	0.79	85.9	3.93	92.2	1.11
	50	90.8	3.28	103.4	2.14	71.9	1.53	88.3	3.01	95.8	2.78
	100	82.9	1.89	97.9	4.27	82.8	1.25	88.8	2.86	97.5	5.32
Isopyrazam	20	96.3	4.29	85.7	2.25	83.2	5.35	98.1	0.22	102.1	3.20
	50	93.2	3.68	94.5	3.67	63.7	4.21	91.4	1.38	102.2	4.81
	100	93.0	1.18	90.3	2.84	80.7	3.06	92.9	0.91	94.7	2.39
Difenoconazole	20	96.0	1.64	88.2	0.33	75.9	4.26	106.9	4.26	100.1	0.57
	50	108.5	1.84	79.7	2.41	64.2	3.63	102.2	2.64	100.3	3.75
	100	107.2	1.25	83.1	5.31	81.2	5.31	113.3	3.16	103.1	3.84
Propiconazole	20	85.3	1.43	94.1	2.53	83.7	3.16	98.9	2.67	96.2	3.57
	50	97.0	2.87	105.0	1.52	65.9	3.27	88.9	3.75	92.5	4.86
	100	96.8	1.56	93.7	2.65	83.5	0.53	92.7	2.65	97.1	4.39
Flusilazole	20	83.4	3.21	94.2	2.56	87.6	4.13	99.3	1.76	93.5	2.78
	50	102.6	2.85	111.0	1.78	77.8	6.33	96.9	2.98	92.4	1.85
	100	96.3	1.04	93.5	3.67	89.9	4.36	95.3	3.56	93.4	0.56
Fenbuconazole	20	91.8	0.49	90.7	3.89	90.7	3.61	98.1	2.16	99.1	3.17
	50	101.8	1.02	111.2	5.75	79.8	3.69	100.1	4.38	100.9	3.75
	100	97.5	0.84	93.6	2.68	93.7	4.21	91.6	1.97	100.1	2.17
Triadimenol	20	114.6	1.28	86.0	0.31	113.9	5.31	112.6	1.69	93.1	4.64
	50	103.8	0.79	83.9	1.75	106.4	3.78	70.5	4.85	80.9	2.16
	100	111.5	1.23	100.6	2.09	107.5	1.22	93.0	2.64	108.9	2.78
Triadimefon	20	82.3	1.29	83.5	1.76	94.4	0.63	105.2	4.21	91.6	3.16
	50	96.3	1.86	103.1	3.42	76.0	1.74	90.1	3.68	93.3	2.11
	100	85.7	1.46	90.9	1.37	85.6	1.89	95.8	1.52	104.7	3.18
Trifloxystrobin	20	90.4	0.76	98.2	0.53	66.9	1.33	95.8	1.85	108.2	2.61
	50	101.0	4.39	92.1	1.36	72.9	3.15	93.7	1.35	90.6	2.63
	100	99.8	1.90	94.9	5.31	69.0	4.26	88.3	3.16	99.5	4.12
Tebuconazole	20	89.0	3.20	94.7	3.13	90.1	3.16	90.8	2.75	81.6	1.78
	50	109.2	1.96	97.4	2.16	78.4	4.22	82.2	1.44	101.5	1.69
	100	91.5	0.48	95.3	2.68	88.1	4.86	90.9	0.89	84.3	2.55
Dimethomorph	20	90.1	0.88	85.6	2.41	106.8	2.31	100.8	3.57	92.3	2.14
	50	93.0	2.87	97.5	4.15	84.2	2.86	92.8	3.61	96.5	2.16
	100	83.8	2.59	90.8	3.27	97.0	2.57	85.3	2.15	94.7	4.62
Imazalil	20	101.2	4.38	87.8	1.76	109.1	2.53	95.9	2.17	104.0	3.11
	50	101.8	3.86	100.5	2.86	99.3	3.16	100.0	3.76	92.0	1.64
	100	94.4	1.77	92.8	1.88	94.7	0.43	95.2	4.86	81.8	1.89
Flutriafol	20	90.9	0.58	89.4	2.16	92.3	1.42	94.6	1.26	88.5	0.64
	50	94.3	1.59	84.2	2.44	97.6	1.66	92.1	1.32	84.3	2.53
	100	85.7	0.64	95.3	1.80	91.3	3.74	96.5	3.22	92.5	2.63

The LODs ($S/N \geqslant 3$) and LOQs ($S/N \geqslant 10$) for the 18 fungicides were determined to be 0.003 and 0.01 mg/kg, respectively. The LOQs of the method met the requirements of GB 2763-2021 *National Food Safety Standard—Maximum Residue Limits for Pesticides in Food* for the maximum residue limits of fungicides in animal-derived foods.

Accuracy and precision

Standard solutions were spiked into blank samples at three concentration levels (2 × LOQ, 5 × LOQ, and 10 × LOQ), and each level was determined in parallel for 6 times. The detailed data are shown in Table 4. The results demonstrated that the 18 fungicides showed average recovery values ranging from 63.7% to 117.5%, with relative standard deviations (RSDs) between 0.22% and 6.33%. Both the recovery and precision values met the requirements for pesticide residue analysis methods.

Analysis of actual samples

A total of 80 commercial samples (including perch, grass carp, crucian carp, pork, pork liver, milk, and eggs) were randomly collected from local farmers' markets, supermarkets, and grocery stores. The established method was applied to analyze the residues of 18 fungicides in these samples. All test results were negative.

Conclusions and Discussion

In this study, optimal purification reagents were identified through investigation of different purification material combinations, and the chromatographic column with optimal peak shape and resolution was selected by comparing various columns. Additionally, matrix effects were evaluated. The results demonstrated significant matrix effects, and matrix-matched curves should be drawn to carry out methodological research. The developed method demonstrated satisfactory accuracy and precision meeting relevant standard requirements. This rapid and accurate method enables testing personnel to efficiently and precisely conduct batch detection of fungicide residues in animal-derived foods, while also providing technical reference for fungicide regulation and risk assessment.

References

[1] LI QL, WANG YS, LUO JY, *et al.* Simultaneous determination of 43 kinds of fungicides in fruits and vegetables by high performance liquid chromatography-triple quadrupole linear ion trap mass spectrometry[J]. Food & Machinery, 2024, 40(7): 53 – 62. (in Chinese).

[2] ZHANG Y, WU LG, MA GJ, *et al.* Simultaneous determination of 10 kinds of fungicides in tomatoes and cucumbers by GC-MS/MS combined with solid phase extraction[J]. Food & Machinery, 2020, 36(7): 67 – 71. (in Chinese).

[3] ZHANG Q, LIU YD, TONG YC, *et al.* Determination of 13 new amide fungicides in fruits and vegetables by QuEChERS-gas chromatography-tandem mass spectrometry[J]. China Food, 2023(4): 78 – 83. (in Chinese).

[4] FAN LB, WU QS, HUANG LL, *et al.* QuEChERS-GC-MS/MS detetermination of residual amount of 6 methoxy acrylate fungicides in 3 edible mushrooms[J]. Physical Testing and Chemical Analysis (Part B: Chemical Analysis), 2020, 56(6): 655 – 660. (in Chinese).

[5] BI SY, CAO T, ZHU ZQ, *et al.* QuEChERS-GC-MS/MS determination of residual amount of 7 amide fungicides in berries[J]. Physical Testing and Chemical Analysis (Part B: Chemical Analysis), 2020, 56(9): 955 – 959. (in Chinese).

[6] YANG K, SUN LP, OUYANG Y. Determination of 58 kinds of pesticide residues in animal derived food by QuEChERS-gas chromatography-tandem mass spectrometry[J]. Journal of Food Safety and Quality, 2025, 16(6): 197 – 206. (in Chinese).

[7] XIAO Y, ZENG XM, LI Z, *et al.* Rapid determination of residues of six herbicides in partly animal-derived foods by QuEChERS-ultra high-performance liquid chromatography-tandem mass spectrometry[J]. Food Research and Development, 2024, 45(11): 178 – 184. (in Chinese).

[8] HUANG ZJ, HUANG PM, LUO KN, *et al.* Determination of chlorantraniliprole and penoxsulam residues in rice by QuEChERS-UPLC-MS/MS [J]. Food and Machinery, 2024, 40(1): 63 – 67. (in Chinese).

[9] ZHANG XY, ZHANG Y, WANG ZY, *et al.* Determination of 10 fungicide residues in fresh vegetable condiments by QuEChERS-liquid chromatography-tandem mass spectrometry[J]. China Brewing, 2021, 40(10): 196 – 200. (in Chinese).

[10] ZHANG SP, QIN Y, GU YJ. Determination of 21 veterinary drugs in cow and goat milk and milk powder by QuEChERS combined with ultra performance liquid chromatography-quadrupole/orbitrap mass spectrometry[J]. Journal of Dairy Science and Technology, 2024, 47(2): 24 – 29. (in Chinese).

[11] LI ZH, GAO H, HE Q, *et al.* Simultaneous determination of 83 pesticide residues in raw goat milk using QuEChERS coupled with ultra-high performance liquid chromatography-tandem mass spectrometry[J]. Chinese Journal of Analysis Laboratory, 2024, 43(10): 1435 – 1442. (in Chinese).

[12] YANG Q, JIAO XZ, MENG H, *et al.* Rapid determination of 54 pesticide residues in fresh milk by QuEChERS pretreatment and ultra-high performance liquid chromatography-tandem mass spectrometry[J]. Journal of Dairy Science and Technology, 2024, 47(6): 43 – 50. (in Chinese).

[13] TANG YH, GAO YN, CHEN JY, *et al.* Rapid determination of paraquat and diquat residues in tea by QuEChERS/ultra-high performance liquid chromatography-tandem quadrupole mass spectrometry[J]. Analytical Chemistry, 2024, 52(10): 1619 – 1627. (in Chinese).

[14] YAO LJ, CHEN YQ, LIN H, *et al.* Simultaneous determination of 27 pyrrolizidine alkaloids in tea by ultra-high performance liquid chromatography-tandem mass spectrometry with integrated QuEChERS purification [J]. Journal of Tea Science, 2024, 44(5): 831 – 842. (in Chinese).

[15] JIANG XQ, YUAN HF, CHENG Y, *et al.* Determination of residues of fipronil and its metabolites in tea by gas chromatography-negative chemical ionization-tandem mass spectrometry with QuEChERS[J]. Physical Testing and Chemical Analysis (Part B: Chemical Analysis), 2024, 60(3): 300 – 304. (in Chinese).

[16] SHEN WH, ZHANG MS, YANG GH. Determination of inositol content in plant-derived sports food by QuEChERS-GC-MS/MS[J]. China Food Additives, 2022, 33(11): 171 – 177. (in Chinese).

[17] LIU SL, WANG Z, LI CL, *et al.* Determination of pydiflumetofen in seven kinds of plant-derived foods based on QuEChERS-liquid chromatography-tandem mass spectrometry[J]. Agrochemicals, 2021, 60(7): 500 – 503. (in Chinese).

(Continued on page 23)

52. (in Chinese).
[37] ENDO N, HIRONAKA M, HONDA Y, *et al.* Combination of UV and green light synergistically enhances the attractiveness of light to green stink bugs *Nezara* spp[J]. Scientific Reports, 2022, 12(1): 12279.
[38] BRCKA C, MCSORLEY R, FREDERICK JJ. Effect of hot water treatments on root-knot nematodes and *Caladium* tubers[J]. Proceedings of the Florida State Horticultural Society, 2000: 158 – 161.
[39] PAN XG. Prevention and treatment of pests during pear planting[J]. Agricultural Technology & Equipment, 2019(5): 75, 77. (in Chinese).
[40] DU JH. Investigation on pests of *Fraxinus mandshurica* and occurrence regularity and control of several new *F. mandshurica* pests[D]. Tai'an: Shandong Agricultural University, 2016. (in Chinese).
[41] HOU YL, WANG F, LI YJ, *et al.* Chemical control of vegetable aphids in open field and net houses[J]. China Plant Protection, 2021, 41(6): 84 – 86, 96. (in Chinese).
[42] ZOU Y, LIU C, LIU Y, *et al.* Preliminary report on the occurrence and control of *Pseudaulacaspis pentagona* on *Actinidia arguta*[J]. South China Agriculture, 2019, 13(17): 13 – 14. (in Chinese).
[43] LI XJ, HAO Z, TAO W, *et al.* Control effects of 12 different kinds of pesticides against *Grapholitha molesta* and *Pseudaulacaspis pentagona* [J]. Plant Protection, 2013, 39(4): 168 – 172.
[44] LI J. Pollution-free planting techniques of kiwifruit[J]. Beijing Agriculture, 2016(2): 51 – 52. (in Chinese).
[45] YANG QP, XIE ZB, WANG LJ, *et al.* Life habit and OPM technology of main pests of organic kiwifruit in Hubei Province[J]. South China Fruits, 2014, 43(6): 132 – 134. (in Chinese).
[46] MA J. Main control points of main pests and diseases of kiwifruit in Weining[J]. Deciduous Fruits, 2022, 54(1): 78 – 80. (in Chinese).
[47] WANG XX, REN JD. Green prevention and control measures of diseases and pests in Xiuwen kiwifruit[J]. Agricultural Technical Services, 2019 (4): 75 – 77. (in Chinese).
[48] LIU LQ. Investigation and control of postharvest diseases and pests of kiwifruit[J]. Beijing Agriculture, 2015(6): 81 – 82. (in Chinese).
[49] HUANG LN. Integrated control of kiwifruit diseases and insect pests [J]. Nong Min Zhi Fu Zhi You, 2015(2): 78. (in Chinese).
[50] WANG YS, XU H, HU MR. Control of three main stink bugs in orchard [J]. Northwest Horticulture, 2019(6): 29 – 31. (in Chinese).
[51] WANG XY, BAI GY, WU LL, *et al.* Effect of biodiversity through planting companion plants on the control of *Meloidogyne incognita*[J]. Chinese Journal of Ecology, 2016, 35(5): 1233 – 1239.
[52] GAO LL, HUANG JL, LU XH, *et al.* Indoor control effect of compost extract and fermentation broth on root-knot nematodes in southern China [J]. Jiangsu Agricultural Sciences, 2016, 44(11): 151 – 154. (in Chinese).
[53] OZDEMIR E, GOZEL U. Efficiency of some plant essential oils on root-knot nematode *Meloidogyne incognita*[J]. Journal of Agricultural Science and Technology, 2017, 7(3): 178 – 183.
[54] KUMAR V, JAIN RK. Management of root-knot nematode, *Meloidogyne incognita* by *Trichoderma viride*, *T. harzianum* and bacterial antagonist *Pseudomonas fluorescens* as seed treatment on okra[J]. Indian Journal of Nematology, 2010, 40(2): 226 – 228.
[55] WANG C, ZHANG JH, FANG G. Study on the *Artemisia capillaris* inhibitory activity of *Meloidogyne* extraction in different growth periods[J]. Northern Horticulture, 2015(9): 102 – 105. (in Chinese).
[56] HOU QC, WANG WH. Control effects of extracts from three types of Chinese herbal medicines on vegetable root-knot nematodes[J]. Guizhou Agricultural Sciences, 2017, 45(5): 66 – 67. (in Chinese).
[57] LI C, ZHAO DX, ZHANG JC, *et al.* Toxic effect of ethanol extracts from 12 kinds of Chinese herbal medicines on root-knot nematodes of banana[J]. Journal of Anhui Agricultural Sciences, 2019, 47(18): 150 – 151, 166. (in Chinese).
[58] SARDARI AA, JALALI AAH, BAHRAMINEJAD S, *et al.* Effect of plant extracts on the mortality of root-knot nematodes' J2, *Meloidogyne javanica*[J]. Archiv Für Pflanzenschutz, 2015, 48(4): 365 – 375.
[59] UMAR I, MAMMAN A. Nematicidal potential of *Faidherbia albida* fruit against *Meloidogyne javanica* on cowpea[J]. Pakistan Journal of Nematology, 2014, 32(1): 77 – 83.
[60] WEN Y, CHITWOOD DJ, VINYARD BT, *et al.* Suppression of *Meloidogyne incognita* by extracts and powdered fruit of *Gleditsia sinensis* (Chinese honeylocust)[J]. Nematropica, 2017, 47(2): 155 – 164.
[61] WANG LY. Analysis on green prevention and control techniques of kiwifruit diseases and pests[J]. South China Agriculture, 2021, 15(23): 48 – 49. (in Chinese).

Editor: Yingzhi GUANG Proofreader: Xinxiu ZHU

(Continued from page 13)
[18] SUN J, YANG J, ZHAO CH, *et al.* Modified QuEChERS method combined with ultra-high performance liquid chromatography-tandem mass spectrometry for simultaneous determination of 19 quinolones and sulfonamides in aquatic products[J]. Food and Machinery, 2023, 39(7): 40 – 47. (in Chinese).
[19] LI H, XIANG J, LI D, *et al.* Simultaneous determination of 80 veterinary drug residues in chicken by QuEChERS-UPLC-MS/MS[J]. Food and Machinery, 2023, 39(6): 48 – 54. (in Chinese).
[20] HUANG XB, LIU WP. Simultaneous determination of 80 veterinary drug residues in chicken by QuEChERS-UPLC-MS/MS[J]. Food and Machinery, 2021, 37(10): 56 – 60. (in Chinese).

Editor: Yingzhi GUANG Proofreader: Xinxiu ZHU