Application Note

Pharma & Biopharma

Agilent

Trusted Answers

Screening of Pesticide Residues in Traditional Chinese Medicine with the Agilent Intuvo 9000 GC

Author

Jie Zhang
Agilent Technologies, Inc.

Abstract

This application note focuses on the residue measurement of 30 banned pesticides and their metabolites in Traditional Chinese Medicine (TCM) by following the standard method in the 2020 China Pharmacopoeia. A midcolumn backflush configuration was used for pesticides analysis. The sample was extracted using the QuEChERS method, and the purification kit was optimized for improved recovery rate. Seven types of TCM matrices were tested to verify the effectiveness of the modified cleanup kit. The instrument performance was investigated in terms of linearity and quantitation precision.

Introduction

The TCM teas, powders, or capsules made from plants are the key branch in the TCM system. Pesticides are applied during the plants' growth for better production yield. It is necessary to measure and control the pesticide residue in the plants to ensure the safety of TCM. The China Pharmacopoeia 2015 edition released four methods to guide the measurement of pesticides in TCM using different analytical techniques. Gas chromatography (GC), coupled with selective ambient detectors or mass spectrometry (MS), is the key technique recommended by the China Pharmacopoeia for pesticide analysis. The method No. 5[1] was developed and officially published in the China Pharmacopoeia 2020 edition. This method prescribes the analysis of 54 banned pesticide residues in TCM by GC/QQQ and LC/QQQ techniques. Among the 54 pesticides, it is recommended that 34 compounds, including 30 pesticides and their metabolites, are analyzed on a GC/QQQ platform.

Hundreds of plants are used in TCM. Different parts of plants, such as flowers, leaves, fruits, stems, barks, and roots are used in different TCM formulas. To analyze the pesticide residue in these matrices, effective pesticide extraction and purification is important. The extraction should generate adequate pesticide recovery from the matrices, and purification is needed to reduce the contamination on the analytical system as much as possible without affecting the targeted compounds. There are three types of sample preparation approaches recommended by method No. 5. These are direct homogenization, QuEChERS, and homogenization followed by SPE purification. Direct homogenization is the simplest method for extracting the pesticides. However, it requires a large amount of sample, and cross-contamination can easily occur when using the same homogenizer to prepare different TCM samples, even when intersample rinsing is made. In addition, homogenization without purification means that all contaminants are present in the sample and can much more easily enter the analytical system. That is why the homogenization-only approach is suitable for clean matrices, which are mostly flower-based. Homogenization followed by SPE purification provides the best cleanup when the SPE cartridges are properly selected. It is more suitable for complex matrices, like barks and roots. However, rotary evaporation is needed to concentrate the extracts right after homogenization and prior to the SPE cleanup, which to some extent limits the sample preparation throughput. What's more, the SPE cartridge selection and operation rely on analyst experience. Compared to the homogenization-based sample preparation procedure, the QuEChERS approach has the advantages of no cross-sample contamination, effective purification, and easy operation, which makes it the best approach to start with for TCM analysis in most government labs and pharmaceutical factories.

The samples prepared from complex matrices bring challenges to the analytical system. GC has a way of coping with this challenge. Inlet liner, precolumn, and backflush techniques are used individually or combined on a GC platform to protect the analytical column and downstream detectors from the dirty matrices.

In this work, the Agilent Intuvo 9000 GC was interfaced with an Agilent 7000 triple quadrupole mass spectrometer for pesticide residue analysis in seven types of TCM matrices. The Intuvo features a Flow Chip design that easily accommodates the backflush configuration. The Intuvo guard chip can trap the contaminants to protect the analytical column. If a guard chip is contaminated, it can be replaced with a new one, so there is no need to trim the column, and the retention time is more consistent. With the above-mentioned advantages, the Intuvo 9000 GC was selected for this analysis. In addition, the sample purification kit was optimized to improve the method recovery. The linearity, repeatability, and recovery rate were evaluated to show the suitability of the tested system for the targeted analysis.

Experimental

The Intuvo 9000 GC coupled with the 7000 triple quadrupole GC/MS was used for sample analysis. An Agilent 7650A automatic liquid sampler was used for sample injection. A midcolumn backflush Flow Chip was configured on the Intuvo to realize the separation and concurrent backflush on two sequentially connected analytical columns. Multiple reaction monitoring (MRM) mode was used for data collection and the transitions were selected based on China Pharmacopoeia recommendations. The analytical conditions and consumables are listed in Table 1. The MRM transitions are listed in the table in the appendix.

In this work, BF time was selected based on angelica roots, then tested on the other six matrices. The backflush time should be selected and tested on the matrices of interest in real analysis, considering the RT of targeted compounds varied in different matrices.

Table 1. Analytical system configuration and test conditions.

Parameter	Value
Agilent 9000 Intuvo GC	
Inert Flow Path Configuration	Agilent Intuvo D2-MS midcolumn backflush (p/n G4588-60721)
Carrier Gas	Helium
Inlet	Split/splitless in pulsed splitless mode, 265 °C
Injection Pulse Pressure	30 psi until 0.5 min
Purge Flow to Split Vent	60 mL/min at 0.5 min
Septum Purge Flow	3 mL/min
Inlet Liner	Ultra Inert, splitless with glass wool (p/n 5190-2293)
Intuvo Guard Chip	Track oven
Columns	Agilent Intuvo custom columns, Two Agilent J&W DB-17, 15 m × 0.25 mm, 0.25 µm (p/n 100-2111-INT)
Column Flow Rate	Column 1: 1.2 mL/min Column 2: 1.5 mL/min * Backflush at 23.5 min on column 1 with –1 mL/min to 26.75 min (column ramp rate 100 mL/min)
Column Flow Rate During Post-Run	–4.56 mL/min for column 1 and 4.89 mL/min for column 2 for 4 min
Oven Temperature Program	80 °C (1 minute), then 40 °C /min to 200 °C, then 2 °C /min to 230 °C, then 40 °C /min to 300 °C (6 minutes)
Agilent 7000 Triple Quadrupole GC/MS	
Transfer Line	260 °C
Ion Source	Extractor source
Source Temperature	250 °C
Quad Temperature	150 °C
Solvent Delay	6 minutes
Gain Use	Yes (gain factor: 10)
Drawout Plate	Standard (3 mm)

Chemicals

Pesticides standard mixtures and internal standard (IS) triphenyl phosphate (TPP) of 100 µg/mL in acetonitrile were purchased from Anpel Inc. There were two types of pesticides mixture used. Mixture 1 contained 33 components without *p,p'*-dicofol, and mixture 2 consisted of 34 pesticides including dicofol isomers (*o,p*-dicofol and *p,p'*-dicofol). The compound concentration in mixture 1 was 100 µg/mL, and in mixture 2 varied from 40 to 100 µg/mL. Both the pesticide standards and IS standard were diluted 100 times by acetonitrile as a working solution for preparation of matrix-matched calibration standards.

Seven types of TCM raw material—wolfberry, licorice, angelica roots, cassia seeds, celosia seeds, mulberry leaf, and Sichuan bulb of fritillary—were purchased from and ground by a local pharmacy. The powder was weighed at 3.00 g and extracted according to the procedure described in China Pharmacopoeia method No. 5. Two dispersive kits were applied for cleanup purposes after the matrices were extracted by acetonitrile and the extraction kits. The recovery results achieved on the two dispersive kits were compared.

For recovery test sample preparation, 150 and 300 µL of pesticide working solution 1 were added to weighted cassia seeds, celosia seeds, mulberry leaf, and Sichuan bulb of fritillary TCM powders. The same volume of pesticides working solution 2 were added to weighted wolfberry, licorice, and angelica roots. The spiked herb powders were extracted, purified, and concentrated to 1 mL, then added with 300 µL IS working solution for analysis.

For calibration standard preparation of each matrix, six tubes of 5 mL purified extracts obtained from 3 g raw material in each tube were concentrated to 600 µL by nitrogen blowing, then added with 10, 20, 50, 100, 150, and 200 µL pesticide working solution and ACN to 1 mL, finally added with 300 µL 100 ng/mL IS for GC/QQQ analysis.

The repeatability samples were prepared by spiking 50 µL pesticides mix 1 working solution to the matrix blank solution of wolfberry, licorice, and angelica roots with 1ml final volume.

The extraction kit and dispersive kits used during the test are listed in Table 2. Only dispersive kit 5156 was used for linearity and repeatability sample preparation.

Table 2. The extraction and dispersive kits.

Sample Preparation Consumables	Description	Part Number
QuEChERS Extraction Kits	1.5 g NaOAc, 6 g $MgSO_4$	5982-5755CH
Dispersive Kit 2048	300 mg C18, 300 mg PSA, 300 mg silica, 900 mg $MgSO_4$, and 90 mg GCB	5982-2048CH
Dispersive Kit 5156	150 mg C18, 150 mg PSA, 900 mg $MgSO_4$	5982-5156
GCB	Bond Elut Carbon Bulk	64100G

Results and discussion

Selection of dispersive kits and recovery test

The testing started from the dispersive kit 2048, containing 300 mg of C18, 300 mg of PSA, 300 mg of silica, 900 mg of $MgSO_4$, and 90 mg of GCB, which is recommended by the China Pharmacopoeia method. For most compounds, the recovery rates based on this kit were in the range of 60% to 130% except for chlordimeform below 40%, which was out of the recovery range required in method No. 5. Different types of dispersive kits were tested to evaluate their impacts on chlordimeform recovery. The test results showed that the kits without silica can improve chlordimeform recovery (as shown in Figures 1A and 1B). The absolute response of 20 ng/g chlordimeform in 3 g of mulberry leaves purified by dispersive kit 5156 was 2.8 times the response purified by kit 2048. Chlordimeform recovery rate was improved from 34.0% to 95.8% without using silica for cleanup.

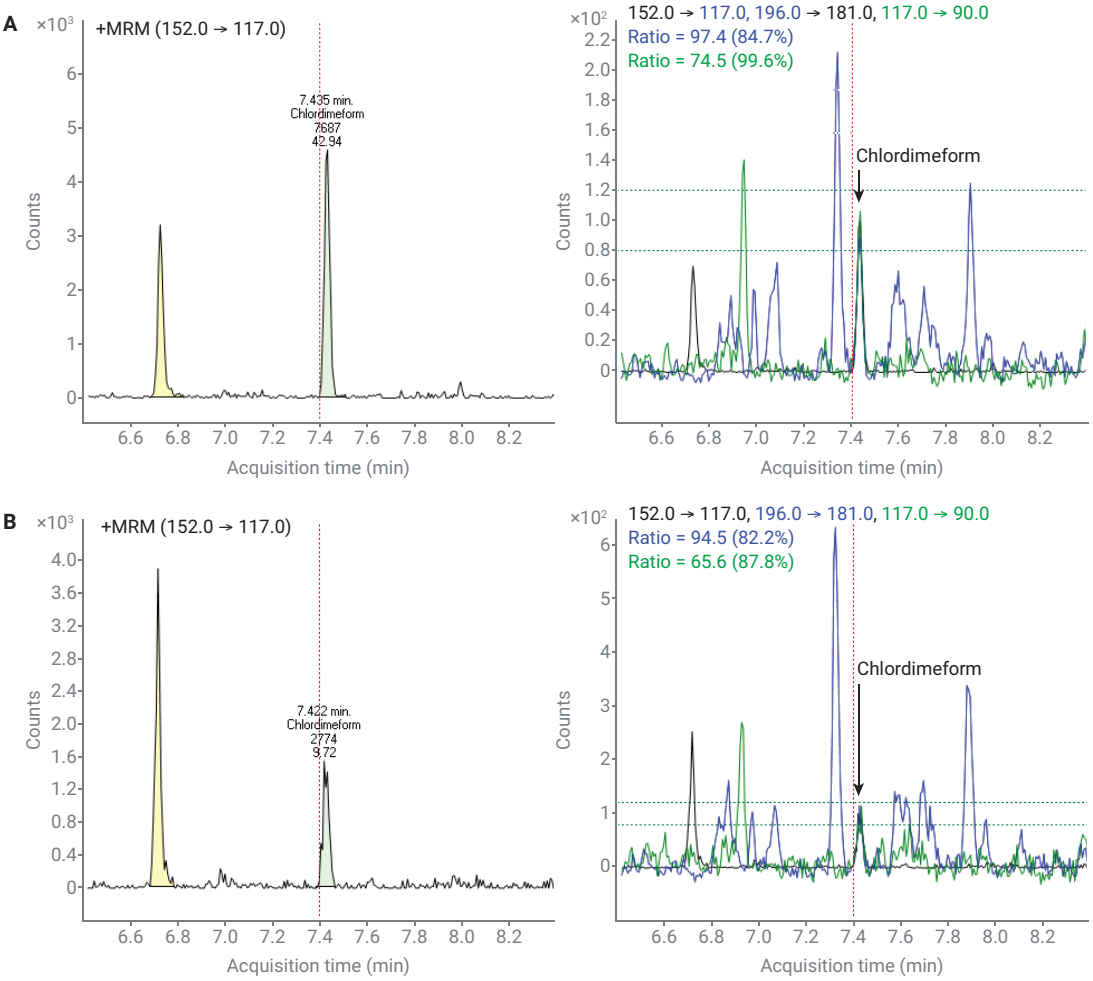

Figure 1. Extracted MRM transitions of chlordimeform in mulberry leaf extract, prepared by dispersive kit 5156 (A) and kit 2048 (B).

The recovery rate of 34 pesticides at the required LOQ level (in mulberry leaf) based on kit 5156 and 2048 was compared in Figure 2. Kit 5156 gave recovery ranging from 72.7% to 124.6%. The rates achieved on kit 2048 ranged from 34.0% to 133.6%.

Besides the selected dispersive kit 5156, it is recommended to add graphite carbon black (GCB) to samples containing high amounts of pigments to effectively get rid of the pigments. In the test, 90 mg GCB was used together with the dispersive kit 5156 for mulberry leaf and cassia seeds extract purification.

The selected dispersive kit 5156 was further evaluated on four matrices. The spiked samples were prepared according to the procedures described in the Experimental section and quantitated using the calibration curves developed in the linearity test. For each matrix, the recovery samples were prepared at two concentration levels with two replicates for each concentration level. The calculated recovery rates were shown in Figures 3A and 3B. The average recovery rates for mulberry leaf ranged from 71.5% to 114.5%, 60.2% to 104% for celosia seed, 85.3% to 121.0% for Sichuan bulb of fritillary, and 58.0% to 100.0% for cassia seeds. DDE-*p,p*' and aldrin in cassia seeds had recovery rates slightly below 60% (average 58% for each component). *Alpha*-BHC had an average of 60% recovery in celosia seed. Other pesticides showed satisfactory recovery results in tested matrices.

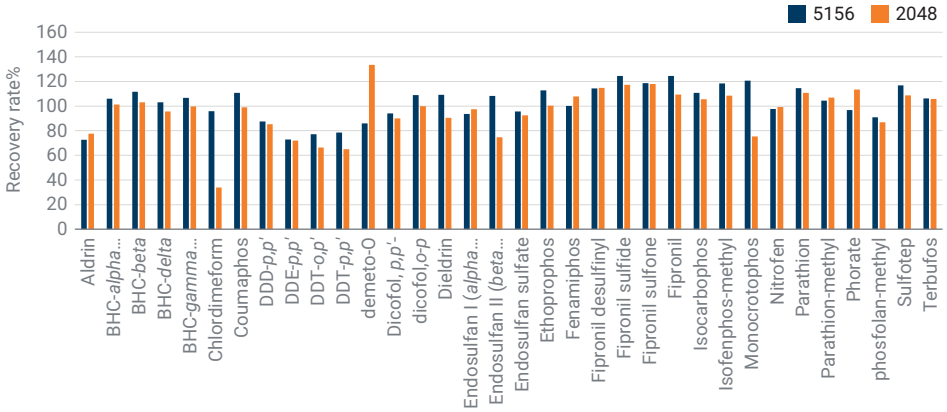

Figure 2. Recovery rate comparison based on two types of dispersive kits (mulberry leaf matrix).

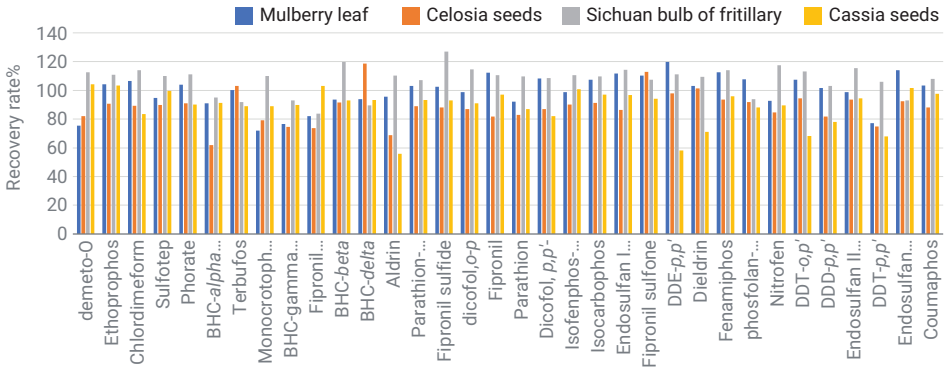

Figure 3A. Recovery rates obtained with dispersive kit 5156 in four types of matrices with components concentration varying from 20 to 50 ng/mL.

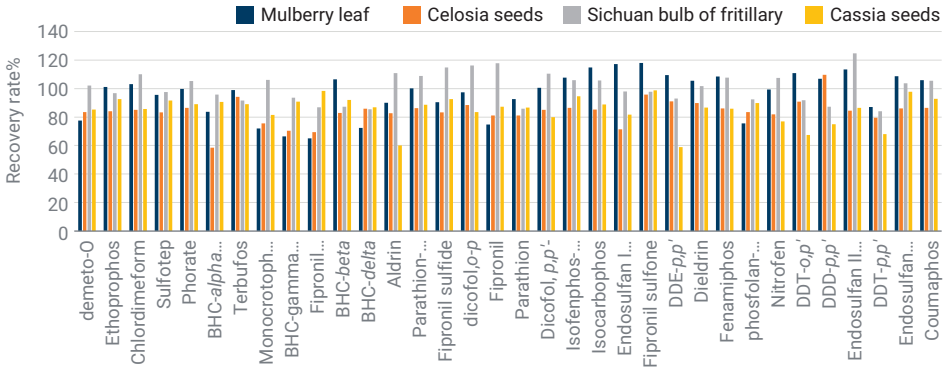

Figure 3B. Recovery rates obtained with dispersive kit 5156 in four types of matrices with components concentration varying from 40 to 100 ng/mL.

Repeatability test

The system repeatability was evaluated using 50 ng/mL standards in wolfberry, angelica, and licorice blank. The response precision was calculated based on seven injections of each standard and depicted in Figure 4. Twenty-one compounds had response precision between 1% and 5% in three matrices. Monocrotophos, phosfolan-methyl, and coumaphos showed response RSD% bigger than 5% (up to 16.3%) in all three matrices. The other compounds with response precision beyond 5% (5.2% to 10.8%) were matrix-dependent results.

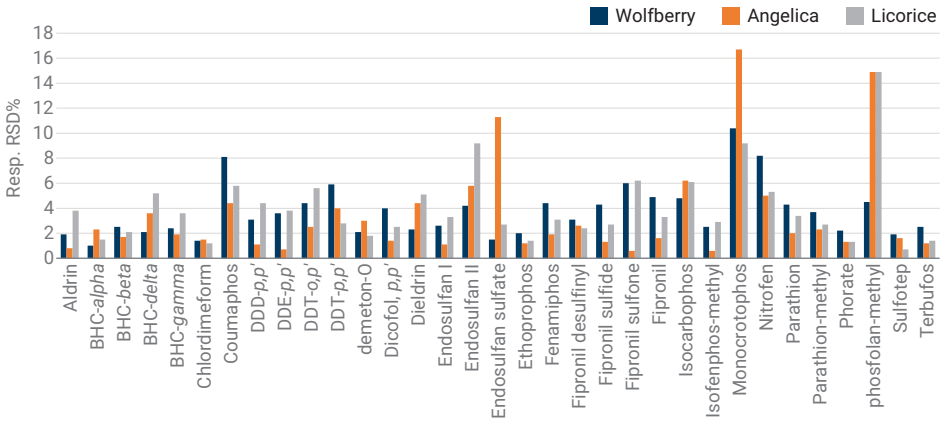

Figure 4. Response precision of 50 ng/mL standards in three matrices (based on seven injections of each standard).

Linearity test

The linearity of matrix-matched calibration standards was evaluated using two sets of standards prepared in seven matrices. Calibration standards set 1 were prepared from pesticide mixture 1 ranging from 10 to 200 ng/mL (approximately 10, 20, 50, 100, 150, and 200 ng/mL) in three matrices: wolfberry, licorice, and angelica. Calibration standards set 2 were prepared from pesticide mixture 2 in four matrices: cassia seeds, celosia seeds, mulberry leaf, and Sichuan bulb of fritillary. The concentration of each pesticide in set 2 calibrants varied at three ranges: 4 to 80 ng/mL, 6 to 120 ng/mL, and 10 to 200 ng/mL. Level 3 of set 2 samples corresponded to each pesticide LOQ level required by method No. 5.

Table 3 shows the pesticide correlation coefficients of their regression formula in different matrices. The average R^2 values ranged from 0.995 to 0.998. Angelica and wolfberry showed the best R^2 values, with an average value of 0.998. The mulberry leaf showed slightly lower R^2 values with an average of 0.995. Among the tested compounds, O-demeton had the worst R^2 in set 2 calibrants, probably because its lowest calibration level started from 4 ng/mL. At this concentration level, the absolute response of O-demeton was small and there were tiny quantities of O-demeton present in cassia seeds and celosia seed extract blanks, which made the O-demeton R^2 worse in these two matrices. Because the O-demeton concentration in set 1 calibrants started from 10 ng/mL, its R^2 was slightly better (>0.995) in set 1 calibrants.

Figure 5 shows the linearity curves of compounds eluting at the early, middle, and late part of the chromatogram (from mulberry leaf).

The oven program used in this work took 26.75 minutes (26.75 minutes for separation and 3.25 minutes for concurrent backflush) and was run under constant column flow rate mode. The method recommended by method No. 5 took approximately 53 minutes and was run under constant pressure mode. The short and long methods were compared based on the identification of pesticides in cassia seeds. The quantifier and qualifier ions of three compounds are shown in Figures 6A to 6C. The left chromatograms for the quantifier MRM transition displayed the compound absolute response and signal-to-noise ratio (S/N). The right MRM chromatograms were for qualifier transitions.

Table 3. Correlation coefficients R^2 in seven matrices.

Peak No.	Compound Name	RT (min)*	Angelica	Licorice	Wolfberry	Mulberry Leaf	Cassia Seeds	Sichuan Bulb of Fritillary	Celosia Seeds
1	O-Demeton	6.72	0.9997	0.9963	0.9980	0.9930	0.9729	0.9891	0.9672
2	Ethoprophos	7.26	0.9997	0.9982	0.9982	0.9983	0.9970	0.9977	0.9978
3	Chlordimeform	7.40	0.9997	0.9972	0.9995	0.9950	0.9992	0.9966	0.9965
4	Sulfotep	7.51	0.9997	0.9984	0.9992	0.9971	0.9992	0.9993	0.9977
5	Phorate	7.73	0.9991	0.9951	0.9984	0.9973	0.9980	0.9994	0.9962
6	*alpha*-BHC (benzene hexachloride)	8.13	0.9999	0.9970	0.9996	0.9985	0.9996	0.9989	0.9993
7	Terbufos	8.33	0.9995	0.9979	0.9996	0.9972	0.9993	0.9984	0.9988
8	Monocrotophos	9.18	0.9998	0.9932	0.9992	0.9832	0.9993	0.9811	0.9911
9	*gamma*-BHC (Lindane, *gamma*-HCH)	9.22	0.9763	0.9959	0.9931	0.9974	0.9992	0.9982	0.9975
10	Fipronil desulfinyl	9.43	0.9990	0.9975	0.9982	0.9964	0.9949	0.9972	0.9995
11	*beta*-BHC	9.85	0.9997	0.9972	0.9989	0.9965	0.9995	0.9991	0.9979
12	*delta*-BHC	10.94	0.9987	0.9966	0.9947	0.9994	0.9994	0.9952	0.9996
13	Aldrin	11.32	0.9981	0.9984	0.9979	0.9970	0.9990	0.9950	0.9980
14	Parathion-methyl	11.76	0.9999	0.9983	0.9981	0.9966	0.9970	0.9843	0.9745
15	Fipronil sulfide	12.42	0.9992	0.9970	0.9950	0.9966	0.9990	0.9985	0.9997
16	Dicofol-*o,p*	12.44	0.9961	0.9967	0.9972	0.9949	0.9982	0.9968	0.9987
17	Fipronil	12.55	0.9976	0.9947	0.9952	0.9934	0.9979	0.9954	0.9935
18	Parathion	12.96	0.9994	0.9973	0.9985	0.9914	0.9952	0.9943	0.9943
19	Dicofol-*p,p'*	13.52	NA	NA	NA	0.9952	0.9994	0.9997	0.9967
20	Isofenphos-methyl	14.01	0.9994	0.9934	0.9966	0.9964	0.9995	0.9988	0.9971
21	Isocarbophos	14.97	0.9941	0.9976	0.9977	0.9986	0.9970	0.9973	0.9970
22	Endosulfan I (*alpha* isomer)	15.75	0.9989	0.9976	0.9987	0.9891	0.9960	0.9960	0.9975
23	Fipronil sulfone	16.15	0.9996	0.9947	0.9993	0.9987	0.9974	0.9957	0.9990
24	DDE-*p,p'*	17.14	0.9994	0.9968	0.9997	0.9980	0.9996	0.9998	0.9986
25	Dieldrin	17.47	0.9991	0.9967	0.9996	0.9966	0.9962	0.9945	0.9963
26	Fenamiphos	18.32	0.9996	0.9971	0.9992	0.9959	0.9987	0.9996	0.9885
27	Phosfolan-methyl	19.33	0.9911	0.9985	0.9982	0.9977	0.9993	0.9952	0.9990
28	Nitrofen	19.93	0.9967	0.9935	0.9933	0.9997	0.9947	0.9981	0.9989
29	DDT-*o,p'*	20.02	0.9997	0.9962	0.9981	0.9993	0.9974	0.9991	0.9984
30	DDD-*p,p'*	20.25	0.9999	0.9984	0.9990	0.9999	0.9995	0.9995	0.9979
31	Endosulfan II (*beta* isomer)	20.41	0.9999	0.9964	0.9982	0.9972	0.9977	0.9965	0.9990
32	DDT-*p,p'*	20.88	0.9999	0.9948	0.9954	0.9990	0.9984	0.9979	0.9987
33	Endosulfan sulfate	21.36	0.9895	0.9972	0.9976	0.9980	0.9994	0.9935	0.9985
34	Coumaphos	24.75	0.9992	0.9942	0.9891	0.9994	0.9993	0.9978	0.9926

* The compound retention times are impacted by the matrices. For a given component, it is possible that its RT shifted as far as 0.5 minutes in two different matrices. The RT values listed here are from one test matrix, just for reference.

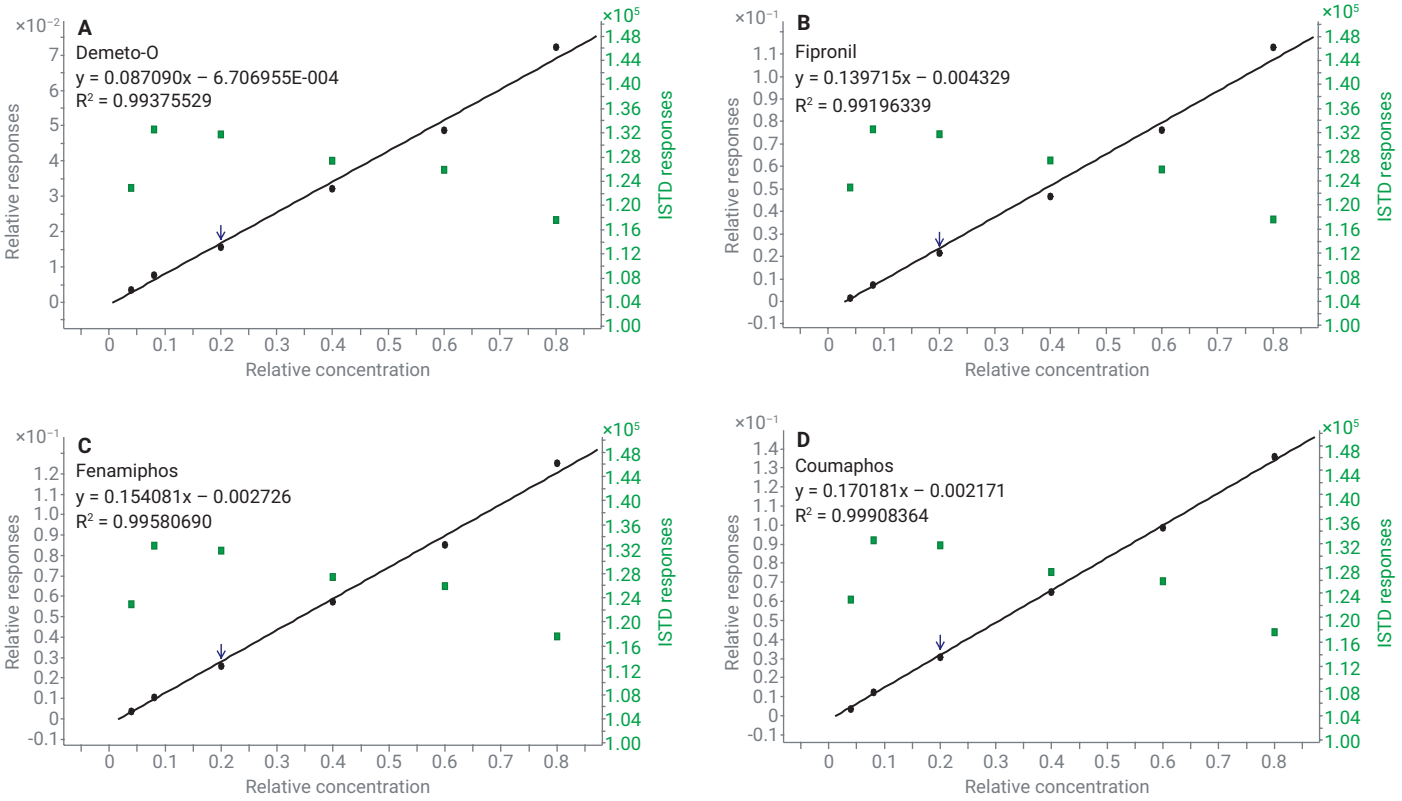

Figure 5. Linear regression curve of representative compounds eluting at different time windows (mulberry leaf). The green spots represented ISTD compounds and their absolute responses are referred to the right Y-axis. (A) O-demeton; (B) fipronil; (C) fenamiphos; (D) coumaphos.

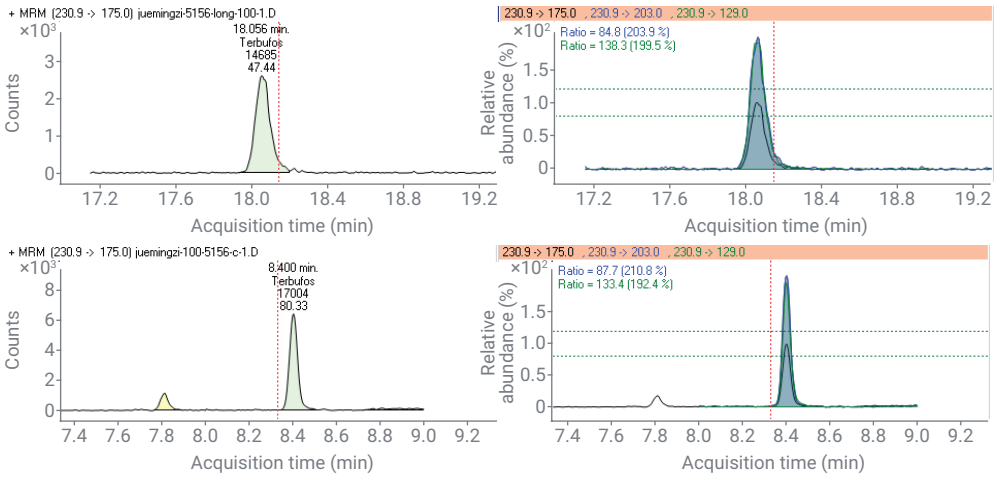

Figure 6A. Quantifier and qualifier transitions of terbufos using the long (top) and short (bottom) method.

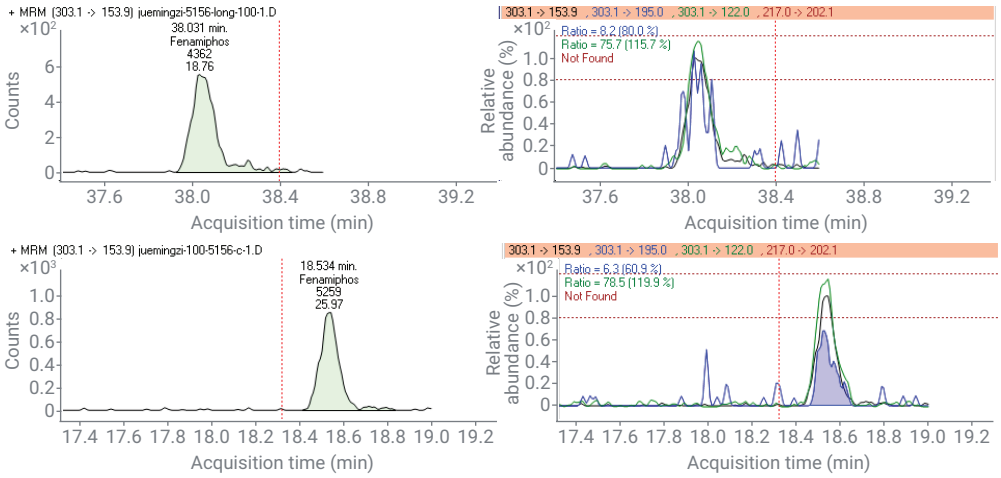

Figure 6B. Quantifier and qualifier transitions of fenamiphos using the long (top) and short (bottom) method.

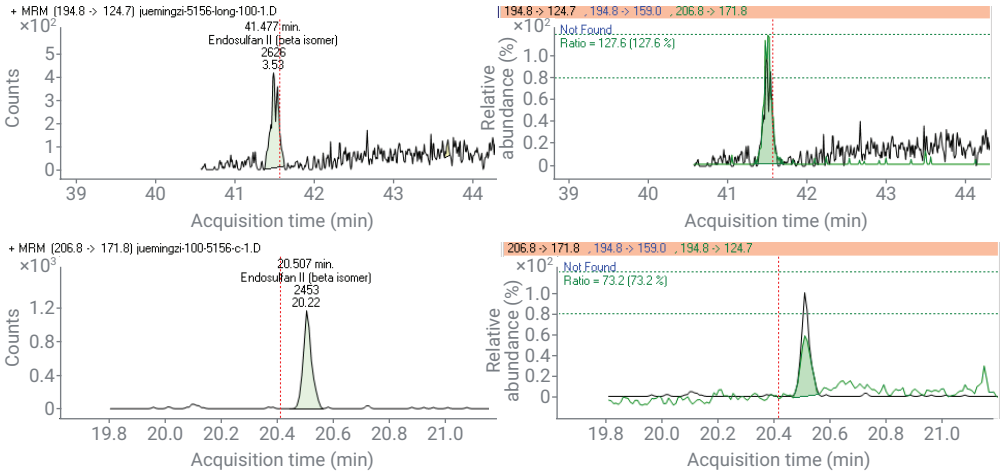

Figure 6C. Quantifier and qualifier transitions of endosulfan II (beta isomer) on long (top) and short (bottom) method.

It was observed that the S/N and peak shapes are better in the short program. With the improved peak shape, S/N ratio, and the verified system precision and linearity performance, it is fair to say that the short oven ramp program under constant flow mode can provide reliable and fast screening for pesticides residue. If an unknown sample result exceeds the regulation limit in the short method, the longer method can easily be applied to the Intuvo system, to provide a confirmational analysis without configurational change. The analysis time saved using a screening approach can improve lab throughput significantly. The calibration curve based on the long method should be ready before the confirmation analysis is made.

Pesticides residue analysis of real sample was performed on cassia seeds, celosia seeds, mulberry leaf, and Sichuan bulb of fritillary. Besides the O-demeton present in cassia seeds and celosia seeds matrices (whose concentration was estimated to be lower than the LOQ level), no pesticides were identified. Figure 7 shows the MRM TIC chromatograms for the four matrices. The peak at approximately 22 minutes was TPP (IS).

Conclusion

The Agilent Intuvo 9000 GC and Agilent 7000 triple quadrupole GC/MS were applied for pesticides residue screening in plant-origin TCM matrices. The dispersive kit used for sample purification was optimized to obtain satisfied recovery rate for tested compounds. The optimized oven program and the use of constant column flow mode accelerated analysis speed and saved 40% analysis time compared to the original method. The system repeatability and linearity were evaluated based on matrix-matched calibration standards with satisfactory results. The combined Intuvo 9000 GC and 7000 triple quadrupole GC/MS systems were demonstrated to be a good choice for pesticides residue screening in TCM.

Reference

1. Method No. 5, Multi-residue determination method for banned pesticides in medicinal materials and decoction pieces (plants), *the Chinese Pharmacopoeia* **2020**.

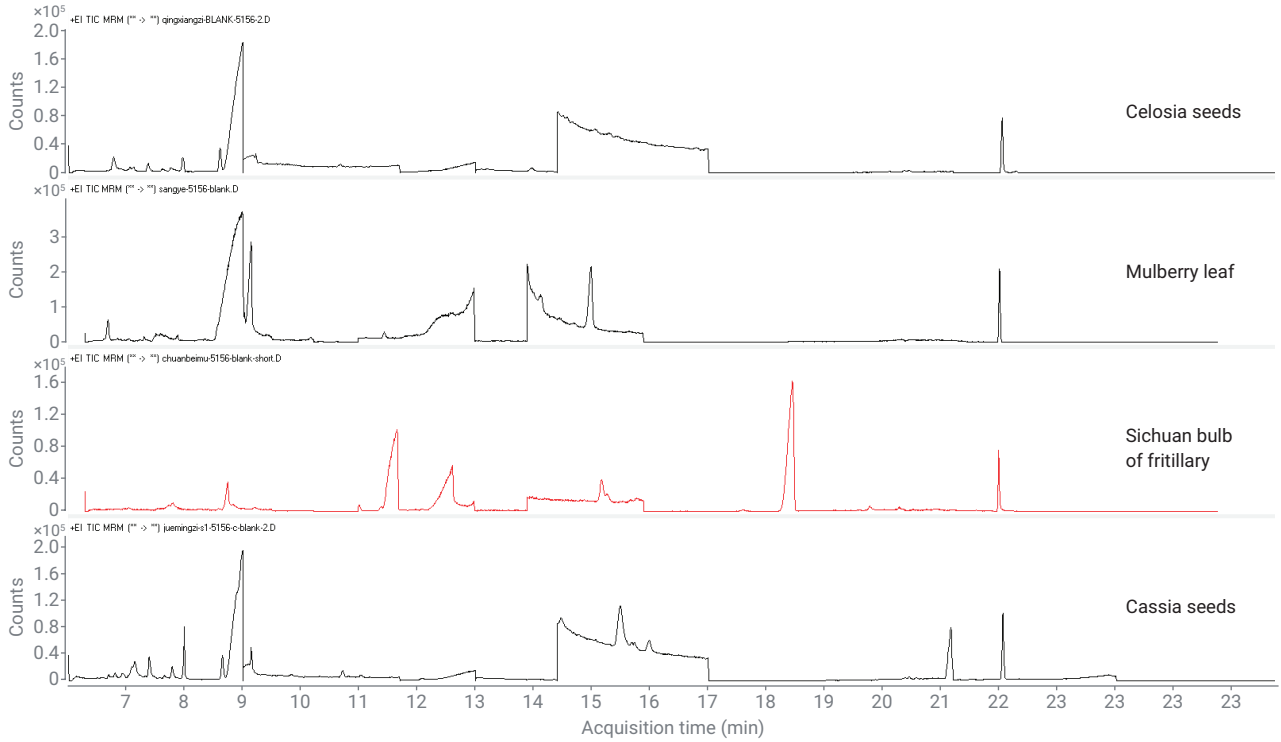

Figure 7. MRM TICs of real TCM samples including celosia seeds, mulberry leaf, Sichuan bulb of fritillary, and cassia seeds.

Appendix

Target pesticides list and MRM conditions (The transitions underlined were used for quantitation in this work. They were selected based on the tested matrices and probably not the best choices for other untested matrices. The selection of quantitation and qualification transitions can be optimized based on matrices type according to method No. 5 in the China Pharmacopoeia).

Segment	Compound Name	RT	Precursor Ion	Product Ion	Collision Energy (CE)
1	O-Demeton	6.793	88	60	4
			88	59	20
			88	45	25
1	Ethoprophos	7.171	199.7	157.8	5
			199.7	114	5
			157.8	113.8	15
			157.8	96.7	20
1	Chlordimeform	7.516	196	181	5
			152	117	15
			117	90	20
1	Sulfotep	7.565	322	174	15
			321.8	294	10
			321.8	201.9	20
1	Phorate	7.805	260	75	5
			230.8	175	10
			230.8	128.6	25
2	*alpha*-BHC (benzene hexachloride)	8.228	218.9	147	10
			218.9	111	10
			218.7	182.9	5
			181	145	15
2	Terbufos	8.33	230.9	203	5
			230.9	175	13
			230.9	129	25
3	Monocrotophos	9.18	192	127.1	10
			127	109	12
			127	95	16
			127	79	20
3	*gamma*-BHC *beta*-BHC *delta*-BHC	9.357 10.105 11.196	218.9 218.9 218.7 181	147 111 182.9 145	10 10 5 15
3	Fipronil desulfinyl	9.703	388	333	20
			388	281	35
3	Aldrin	11.405	262.7	202.7	20
			262.7	192.7	30
			254.9	220	20

Segment	Compound Name	RT	Precursor Ion	Product Ion	Collision Energy (CE)
4	Parathion-methyl	11.940	263.1	136	5
			263.1	109	13
			263.1	79	35
4	Fipronil sulfide	12.823	420	351	12
			420	255	20
4	Dicofol-o,p	12.597	250	215	5
			250	139	15
			139	111	15
4/5	Fipronil	13.065	367	332	15
			367	255	25
			367	213	35
			351	255	20
4	Parathion	13.152	291	109	25
			291	81	30
			139	109	10
5	Dicofol-*p,p'*	13.763	250	215	5
			250	139	15
			139	111	15
5	Isofenphos-methyl	14.120	241	199	5
			241	166.7	10
			241	120.8	20
			199	121	15
			199	65	40
6	Isocarbophos	15.042	229.7	211.7	10
			135.7	108	15
			121	93	15
			120.7	65	20
6	Endosulfan I (*alpha* isomer)	15.909	240.8	205.6	15
			240.8	170	25
			194.9	159	10
6/7	Fipronil sulfone	16.866	452	383	8
			383	255	20
			383	213	32
7	DDE-*p,p'*	17.316	316	246	25
			246	220	15
			246	210	28
			246	176	30
7	Dieldrin	17.615	276.8	240.7	10
			276.8	172	35
			276.8	169.7	35
			262.9	193	35
7	Fenamiphos	18.542	303.1	195	25
			303.1	153.9	30
			303.1	122	20
			217	202.1	10

Segment	Compound Name	RT	Precursor Ion	Product Ion	Collision Energy (CE)
8	Phosfolan-methyl	19.605	227	167.8	10
			227	92	10
			227	60	30
			167.8	109	10
			91.9	63.8	15
9	Nitrofen	20.080	284.8	254.9	10
			282.8	253	10
			282.8	201.8	15
			201.8	138.7	28
9	DDT-*o,p'*	20.102	246	176	15
			237	165	25
			235	199	15
			235	165	25
9	DDD-*p,p'*	20.367	237	165	25
			235	199	18
			235	165	25
9	Endosulfan II (beta isomer)	20.515	206.8	171.8	15
			194.8	159	10
			194.8	124.7	30
9	DDT-*p,p'*	20.947	237	165	25
			235	199	18
			235	165	25
10	Endosulfan sulfate	21.36	273.8	238.9	15
			271.8	236.7	15
			271.8	141	40
			271.8	117	40
10	Triphenylphosphsate (TPP)	22.043	326	233	18
			326	215	25
			326	169	5
11	Coumaphos	24.896	361.8	225.8	15
			361.8	109	15
			361.8	81	5

www.agilent.com/chem

DE44371.5111458333

Printed in the USA, September 14, 2021
5994-4044EN

Agilent
Trusted Answers