Analysis of Pesticide Residues in Spinach Using Agilent Bond Elut QuEChERS AOAC Kit by LC/MS/MS Detection

Application Note

Food Safety

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Abstract

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) AOAC sample preparation approach for the extraction and cleanup of 13 pesticide residues representing various pesticide classes in spinach. The original AOAC method employed involves initial extraction in a buffered aqueous/acetonitrile system, an extraction/partitioning step after the addition of salt, and a cleanup step using dispersive solid-phase extraction (dispersive SPE). In order to address the significant loss of planar pesticides caused by graphitized carbon black (GCB) in dispersive SPE, a modified method with the addition of toluene was employed. The presence of the target pesticides in the spinach extracts were then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode. With the combination of original and modified dispersive SPE, the method was validated in terms of recovery and reproducibility for all of the analytes of interest. The 5 ng/g limit of quantitation (LOQ) for pesticides in spinach shown in this application was well below the maximum residue limits (MRLs). The spiking levels for the recovery experiments were 10, 50, and 200 ng/g. Mean recoveries ranged between 64% and 108% (average of 91.9%), with RSD below 10% (average of 3.3%).

Introduction

The AOAC QuEChERS method has been widely applied in the analysis of pesticides in food since it was introduced by USDA scientists. [1-3] In general, it contains two major steps: extraction and dispersive SPE cleanup. In the extraction step, the method uses a single step buffered 1% acetonitrile (ACN) extraction while simultaneously salting out water from the sample using anhydrous magnesium sulfate ($MgSO_4$) to induce liquid-liquid partitioning. For cleanup, a dispersive solid phase extraction (dispersive SPE) step is employed using a combination of primary secondary amine (PSA) to remove fatty acids as well as other components, and anhydrous $MgSO_4$ to reduce the remaining water in the extract. According to different food matrices, other ingredients may be added in this step, such as graphitized carbon black (GCB) to remove pigments and sterol, or C18 to remove more lipids and waxes.

Spinach is considered to be a highly pigmented vegetable since it contains high levels of chlorophyll. Therefore, the dispersive SPE kits with GCB were selected for further cleanup. In these kits, 50 mg of GCB per mL of ACN extracts are added to 50 mg of PSA and 150 mg of $MgSO_4$. GCB adsorbs planar molecules such as pigments and sterols. Therefore, it is helpful in the cleanup of pigmented matrices such as spinach. However, GCB also adsorbs pesticides with planar structure such as carbendazim, and thiabendazole. As a result, this type of dispersive SPE kit is not recommended for use with planar pesticides. This limitation will have a negative impact on the analysis of planar pesticides from pigmented matrices.

In the previous Application Note, [4] we discussed the impact of toluene addition to the dispersive SPE tube on the analysis of pesticides in pigmented matrix. This Application Note illustrated that this modification can greatly increase the extraction efficiency for problematic pesticides. GCB was employed for the analysis of planar pesticides in pigmented matrices such as spinach with the addition of toluene. In this study, 13 pesticides were used for evaluating the performance of the Agilent AOAC Buffered Extraction kit (p/n 5982-5755) and Bond Elut QuEChERS AOAC Dispersive SPE kits for Pigmented Fruits and Vegetables (p/n 5982-5222 and 5982-5258). With the combination of original and modified dispersive SPE, the method was validated in terms of recovery and reproducibility. Table 1 shows the chemical and regulatory information for these pesticides in spinach.

Experimental

Reagents and Chemicals

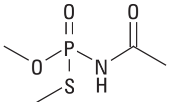
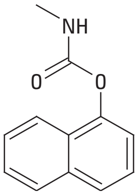
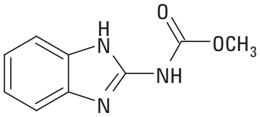
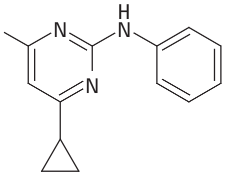
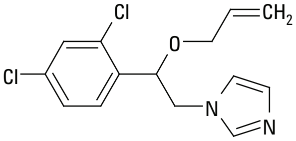
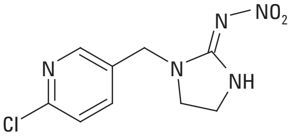
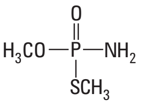
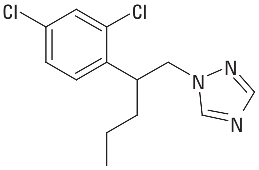
All reagents and solvents were HPLC or analytical grade. Methanol (MeOH), and toluene were from Honeywell (Muskegon, MI, USA). Acetonitrile (ACN), dimethyl sulfoxide (DMSO) and glacial acetic acid (HAc) were from Sigma-Aldrich (St Louis, MO, USA). Ammonium acetate (NH_4OAc) was from Fisher Chemicals (Fair Lawn, NJ, USA). Formic acid (FA) was from Fluka (Sleinheim, Germany). The pesticide standards and internal standard, triphenyl phosphate, (TPP) were purchased from Sigma-Aldrich (St Louis, MO, USA), ChemService (West Chester, PA, USA), Ultra (Kingstown, RI, USA), or AlfaAesar (Ward Hill, MA, USA).

Solutions and Standards

The 1M NH_4OAc pH 5 stock solution was made by dissolving 19.27 g NH_4OAc powder in 250 mL Milli-Q water. The pH was adjusted to 5 with HAc monitored with a pH meter. The solution was stored at 4 ºC. A 5 mM NH_4OAc solution in 20:80 $MeOH/H_2O$, pH 5, was made by combining 200 mL MeOH and 800 mL Milli-Q water, adding 5 mL of 1 M NH_4OAc pH 5 stock solution and mixing well. A 5 mM NH_4OAc in ACN solution was prepared by adding 5 mL of 1 M NH_4OAc pH 5 stock solution to 1 L ACN, mixing well and sonicating 5 min. A 1% HAc in ACN solution was prepared by adding 10 mL of HAc to 1 L of ACN, and mixing well.

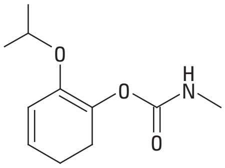
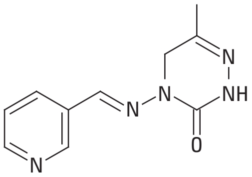
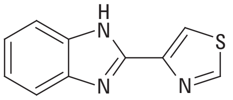
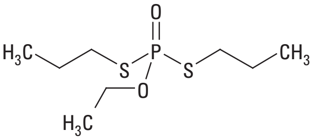
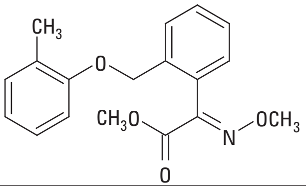
Standard and internal standard (IS) stock solutions (2.0 mg/mL for all, except 0.5 mg/mL for carbendazim) were made in MeOH, 0.1% FA in ACN, or DMSO, respectively, and stored at –20 ºC. Three QC spiking solutions of 1.5, 7.5 and 30 µg/mL were made fresh daily in 1:1 ACN/H_2O containing 0.1% FA. A 10 µg/mL standard spiking solution in 1:1 ACN/H_2O containing 0.1% FA was made for the preparation of calibration curves in the matrix blank extract by appropriate dilution. A 15 µg/mL IS spiking standard of TPP was made in 1:1 ACN/H2O containing 0.1% FA.

Table 1. Pesticides Chemical and Regulatory Information [5–7]

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Acephate	Organophosphate	–0.89	8.35		20
Carbaryl	Carbamate	2.36	10.4		50
Carbendazim	Benzimidazole	1.48	4.2		100
Cyprodinil	Anilinopyrimidine	4	4.44		500
Imazalil	Imidazole	3.82	6.53		20
Imidacloprid	Neonicotinoid	0.57	NA		1000
Methamidophos	Organophosphate	–0.79	NA	 $H_3CO{-}P(=O)(SCH_3){-}NH_2$	10
Penconazole	Triazole	3.72	1.51		50

(Continued)

Table 1. Pesticides Chemical and Regulatory Information [5–7]

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Propoxur	Carbamate	0.14	NA		2000
Pymetrozine	Pyridine	–0.19	4.06		600
Thiabendazole	Benzimidazole	2.39	4.73 12.00 0		50
Ethoprophos	Organophosphate	2.99	NA		5
Kresoxim-methyl	Strobilurin	3.4	NA		50

*The MRLs numbers listed in the table are for spinach or other vegetables. They could be different in different commodities.

Equipment and Material

Agilent 1200 Series HPLC with Diode Array Detector (Agilent Technologies Inc., CA, USA).

Agilent 6410 triple quadrupole LC/MS/MS system with Electrospray Ionization (Agilent Technologies Inc., CA, USA).

Agilent Bond Elut QuEChERS AOAC Extraction kits, p/n 5982-5755, and Bond Elut QuEChERS AOAC dispersive SPE kits for Pigmented Fruits and Vegetables, p/n 5982-5222 and 5982-5258 (Agilent Technologies Inc., DE, USA).

CentraCL3R Centrifuge (Thermo IEC, MA, USA)

Bottle top dispenser (VWR, So. Painfield, NJ, USA)

Eppendorf microcentrifuge (Brinkmann Instruments, Westbury, NY, USA)

Instrument Condition

The previous LC/MS/MS method was directly used. [8]

HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm (p/n 959963-312)
Flow rate:	0.3 mL/min
Column Temperature:	30 °C
Injection volume:	10 µL
Mobile Phase:	A: 5 mM NH_4OAc, pH 5.0 in 20:80 MeOH/H_2O B: 5 mM NH_4OAc, pH 5.0 in ACN
Needle wash:	1:1:1:1 ACN/MeOH/isopropyl alcohol (IPA)/H_2O w/0.2% FA.

Gradient:

Time	% B	Flow rate (mL/min)
0	20	0.3
0.5	20	0.3
8.0	100	0.3
10.0	100	0.3
10.01	20	0.5
13.0	STOP	

Post run:	4 min
Total cycle time:	17 min

MS conditions

Positive mode

Gas temperature:	350 °C
Gas flow:	10 L/min
Nebulizer:	40 psi
Capillary:	4000 V

Other conditions relating to the analytes are listed in Table 2.

Sample Preparation

The sample preparation procedure includes sample comminution, extraction/partitioning and dispersive SPE cleanup. It has been described in detail in previous Application Notes [8]. The procedure used in spinach was similar except for the addition of toluene to the dispersive SPE cleanup step.

Frozen chopped organic spinach was homogenized thoroughly. A 15 g (± 0.1g) amount of homogenized sample was placed into a 50 mL centrifuge tube. Samples were fortified with appropriate QC spiking solutions (100 µL) when necessary, and then 100 µL of IS spiking solution (15 µg/mL of TPP) were added. After vortexing sample for 30 s, 15 mL of 1% HOAc in ACN was added to each tube using the dispenser. To each tube, an Agilent Bond Elut QuEChERS AOAC extraction salt packet (p/n 5982-5755) was added directly. Sample tubes were capped tightly, and hand-shaken vigorously for 1 min. Tubes were centrifuged at 4000 rpm for 5 min.

The ACN extracts were separated into two samples for both original and modified dispersive SPE methods. The modified dispersive SPE method follows a different procedure, therefore it is described below in detail. The volume of ACN extracts (~14 mL) was enough for simultaneously processing samples with original and modified dispersive SPE when using 2 mL size dispersive SPE tubes. When 15 mL size tubes were used, 14 mL of ACN extracts from one sample was not enough for processing dispersive SPE by two methods simultaneously. Therefore, another sample was extracted from the beginning.

A 1 mL aliquot of the upper ACN layer was transferred into an Agilent Bond Elut QuEChERS dispersive SPE 2 mL tube (p/n 5982-5222); or 8 mL aliquot into an Agilent Bond Elut QuEChERS dispersive SPE 15 mL tube (p/n 5982-5258). The 2 mL tube contained 50 mg of PSA, 50 mg of GCB and 150 mg of anhydrous $MgSO_4$; while the 15 mL tube contained 400 mg of PSA, 400 mg of GCB and 1200 mg of anhydrous $MgSO_4$. Next, 375 µL of toluene were added to the 2 mL tube, or 3 mL of toluene was added to 15 mL tube. The tubes were tightly capped and vortexed for 1 minute. The tubes were vortexed for a few seconds before sample addition, to prevent agglomerates. The 2 mL tubes were centrifuged with a microcentrifuge at 13,000 rpm for 2 min, and the 15 mL tubes in a standard centrifuge at 4000 rpm for 5 min. An 825 µL amount

Table 2. Instrument Acquisition Data for the Analysis of 13 Pesticides by LC/MS/MS

Analyte	MRM channels (*m/z*)	Fragmentor (V)	CE (V)	RT (min)
Acephate	1) 184.0 > 94.9 2) 184.0 > 111.0	60	3 15	2.55
Methamidophos	1) 142.0 > 94.0 2) 142.0 > 124.9	60	8 8	2.54
Pymetrozine	1) 218.1 > 105.0 2) 218.1 > 78.0	115	20 50	2.97
Carbendazim	1) 192.1 > 160.0 2) 192.1 > 105.0	95	18 40	5.07
Imidacloprid	1) 256.1 > 209.1 2) 256.1 > 175.0	60	12 18	5.53
Thiabendazole	1) 202.1 > 175.0 2) 202.1 > 131.0	110	27 38	5.65
Propoxur	1) 210.1 > 111.0 2) 210.1 > 92.9	50	12 15	6.89
Carbaryl	1) 202.0 > 145.0 2) 202.0 > 115.0	50	3 40	7.30
Ethoprophos	1) 243.1 > 130.9 2) 243.1 > 172.9	80	15 15	8.50
Imazalil	1) 297.1 > 158.9 2) 297.1 > 200.9	80	22 15	8.52
Penconazole	1) 284.1 > 158.9 2) 284.1 > 172.9	80	32 32	8.95
Cyprodinil	1) 226.1 > 93.0 2) 226.1 > 108.0	120	35 35	9.23
Kresoxim methyl	1) 314.0 > 222.1 2) 314.0 > 235.0	70	10 10	9.44
TPP (IS)	1) 327.1 > 77.0 2) 327.1 > 151.9	70	45 45	9.49

1) Quantifier transition channel
2) Qualifier transition channel

of extract was then transferred into another tube, and dried by N_2 flow. Samples were reconstituted into 600 µL of ACN containing 0.1%FA. After vortexing and sonicating, 200 µL of extract were transferred into an autosampler vial, and then 800 µL of water or other appropriate standard solution (prepared in water) were added. The samples were capped and vortexed thoroughly for LC/MS/MS analysis.

Another aliquot of ACN extracts was processed following the original dispersive SPE clean-up procedure. Figure 1 shows the flow chart of the whole extraction procedure (original and modified dispersive SPE) for a spinach sample.

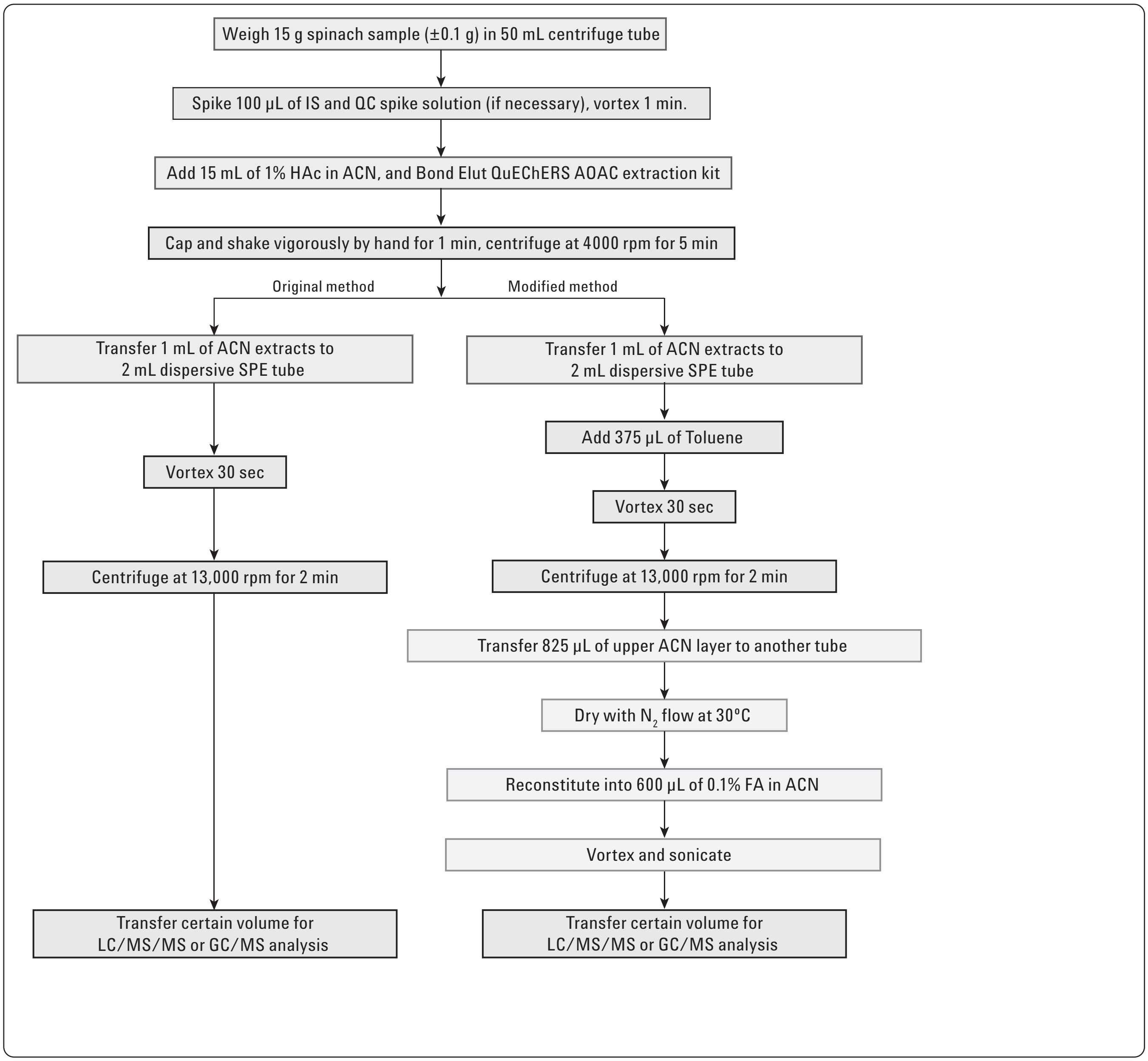


Figure 1. Flow chart of the QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for a spinach sample.

Results and Discussion

QuEChERS method for pesticide residues analysis provides high-quality results in a fast, easy, inexpensive approach. For the pigmented fruits and vegetables, the addition of GCB in the dispersive SPE tube can improve the removal of pigments and sterols. Toluene was added to increase the extraction efficiency of planar pesticides. Previously it was established that the addition of toluene produces a yellow final sample, indicating that matrix impurities are retained. [4] However, with the powerful selectivity provided by LC/MS/MS, there have not been any chromatographic differences found between the samples processed with the original and modified methods. Figures 2 and 3 show the LC/MS/MS chromatograms of matrix blank (IS spiked) and 50 ng/g fortified spinach extract processed by original and modified dispersive SPE method.

Four pesticides with planar structure showed significant loss by the original dispersive SPE method. The modified method with toluene addition increased the recovery of those four pesticides by two to three times, from 20% to 40% and 60% to 100%. In addition, the repeatability improved from >15% to <5% RSD. The addition of toluene had no affect on the quantitation results of other pesticides. Therefore, the results from the original method for high recovered pesticides were combined with the results from modified method for planar pesticides. The method was validated in terms of recovery and reproducibility, and the quantitation results are discussed.

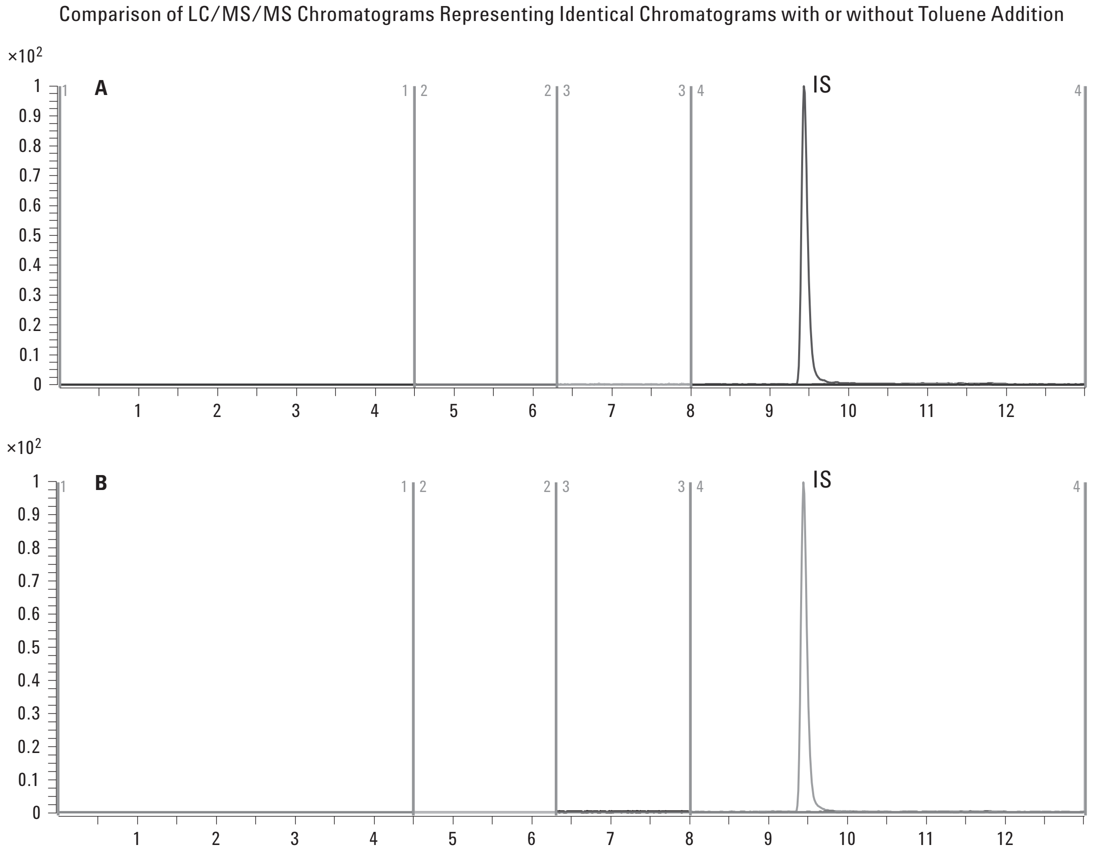


Figure 2. LC/MS/MS chromatograms of spinach matrix blank processed by original dispersive SPE (A) and modified dispersive SPE (B). IS: Internal Standard TPP.

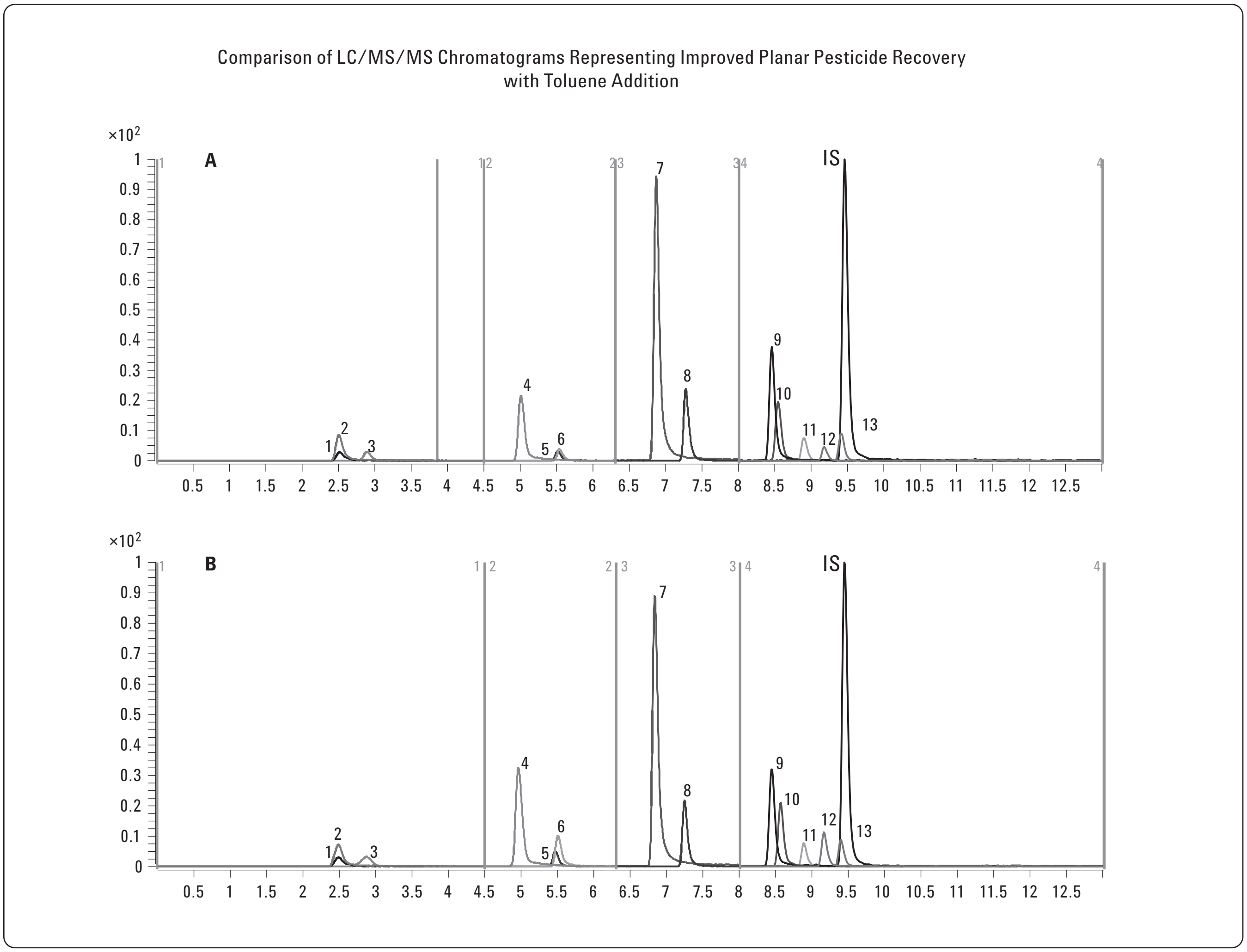

Comparison of LC/MS/MS Chromatograms Representing Improved Planar Pesticide Recovery with Toluene Addition

Figure 3. LC/MS/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos, 10. Imazalil, 11. Penconazole, 12. Cyprodinil, 13. Kresoxim methyl IS: Internal Standard, TPP.

Linearity and Limit of Quantification (LOQ)

The linear calibration range for all of the pesticides was 5–250 ng/g. For samples processed by original and modified methods, the corresponding matrix blanks were used to prepare the calibration curves respectively. Calibration curves, spiked in matrix blanks, were made at levels of 5, 10, 50, 100, 200, and 250 ng/g. The TPP was used as an internal standard at 100 ng/g. The calibration curves were generated by plotting the relative responses of analytes (peak area of analyte / peak area of IS) to the relative concentration of analytes (concentration of analyte / concentration of IS). The 5 ng/g quantification limits LOQ (5 ppb) established for all of the pesticides was lower than or equal to the MRLs of these pesticides in fruits and vegetables. Table 3 shows the linear regression equation and correlation coefficient (R^2) for both 1 mL and 8 mL dispersive SPE.

Table 3. Linearity of Pesticides in Spinach Extract

Analytes	1 mL dispersive-SPE Regression equation	R^2	8 mL dispersive-SPE Regression equation	R^2
Methamidophos	Y = 0.2358X – 0.0008	0.9976	Y = 0.2164X – 0.0014	0.9983
Acephate	Y = 0.0862X – 0.0003	0.9975	Y = 0.0804X – 0.0006	0.9942
Pymetrozine *	Y = 0.2073X – 0.0002	0.9995	Y = 0.2034X – 0.0013	0.9978
Carbendazim *	Y = 0.8375X + 0.0032	0.9915	Y = 0.8383X + 0.0002	0.9982
Imidacloprid	Y = 0.0652X – 0.0007	0.9905	Y = 0.0620X – 0.0011	0.9742
Thiabendazole *	Y = 0.4081X - 0.0008	0.9995	Y = 0.4102X – 0.0011	0.9975
Propoxur	Y = 1.9253X – 0.0042	0.9995	Y = 1.8253X – 0.0037	0.9996
Carbaryl	Y = 0.4243X – 0.0013	0.9979	Y = 0.3993X – 0.0019	0.9946
Ethoprophos	Y = 0.7859X – 0.0012	0.9983	Y = 0.7420X – 0.0012	0.9985
Imazalil	Y = 0.4586X + 0.0002	0.9954	Y = 0.4229X + 0.0005	0.9903
Penconazole	Y = 0.1643X – 0.0014	0.9923	Y = 0.1468X – 0.0003	0.9944
Cyprodinil *	Y = 0.3274X – 0.0024	0.9904	Y = 0.3067X – 0.0013	0.9978
Kresoxim methyl	Y = 0.1809X – 0.0015	0.9975	Y = 0.1659X – 0.0008	0.9928

* Results from modified dispersive SPE method.

Recovery and Reproducibility

The recovery and reproducibility were evaluated by spiking pesticides standards in communited spinach sample at levels of 10, 50 and 200 ng/g. These QC samples were quantitated against the matrix spiked calibration curve. The analysis was performed six times at each level. The recovery and reproducibility (shown as RSD) data of 1 mL and 8 mL volume dispersive SPE are shown in Tables 4 and 5, respectively. The results show that the nine pesticides processed with the original method resulted in excellent recoveries (average of 97.8% for 1 mL and 103.4% for 8 mL) and precision (average of 3.6% RSD for 1 mL and 4.3% RSD for 8 mL). Although the four pesticides processed with the modified method resulted in lower recoveries (average of 78.5% for 1 mL and 69.7% for 8 mL) but high precision (average of 2.7% RSD for 1 mL and 3.3% RSD for 8 mL). The results from the modified method were much better than the results obtained by original methods. Please refer to previous Application Note [4] for a detailed discussion.

Table 4. Excellent Recovery and Reproducibility of Pesticides in Fortified Spinach with a 1 mL volume, 2 mL Dispersive SPE Tube (p/n 5982-5222)

Analytes	10 ng/g fortified QC Recovery	 RSD (n=6)	50 ng/g fortified QC Recovery	 RSD (n=6)	200 ng/g fortified QC Recovery	 RSD (n=6)
Methamidophos	91.8	4.2	93.3	3.7	93.8	5.7
Acephate	93.4	3.3	91.3	5.6	101.9	7.8
Pymetrozine *	74.0	2.9	71.1	3.2	70.3	2.9
Carbendazim *	105.3	4.0	109.1	2.5	88.9	1.7
Imidacloprid	98.2	4.5	100.4	3.7	100.0	2.7
Thiabendazole *	79.0	2.7	76.6	2.3	75.5	1.8
Propoxur	100.0	1.7	98.1	3.5	93.0	4.0
Carbaryl	110.8	3.2	108.1	1.0	105.1	3.2
Ethoprophos	98.8	1.6	98.2	3.3	95.1	3.1
Imazalil	84.0	3.8	89.6	2.5	89.8	1.7
Penconazole	103.1	5.4	98.4	3.5	97.2	1.9
Cyprodinil *	69.1	4.7	62.0	2.9	61.3	1.1
Kresoxim methyl	104.4	4.8	101.2	5.0	102.6	3.0

* Results from modified dispersive SPE method.

Table 5. Excellent Recovery and Reproducibility of Pesticides in Fortified Spinach with a 8 mL volume, 15 mL Dispersive SPE Tube (p/n 5982-5258)

Analytes	10 ng/g fortified QC Recovery	 RSD (n=6)	50 ng/g fortified QC Recovery	 RSD (n=6)	200 ng/g fortified QC Recovery	 RSD (n=6)
Methamidophos	98.6	3.8	94.2	7.1	97.8	2.9
Acephate	95.5	8.9	91.5	6.3	105.6	5.7
Pymetrozine *	62.4	4.3	53.9	3.4	59.3	5.4
Carbendazim *	95.7	1.6	98.6	1.9	93.3	2.9
Imidacloprid	112.7	4.2	107.6	7.7	110.4	3.7
Thiabendazole *	58.0	3.5	62.1	3.3	66.8	2.8
Propoxur	104.9	1.4	103.3	3.7	99.0	3.3
Carbaryl	116.9	2.2	114.6	2.4	110.8	2.1
Ethoprophos	105.3	2.5	105.7	2.8	103.0	2.3
Imazalil	86.3	3.9	94.9	4.3	93.9	3.4
Penconazole	103.5	10.4	106.9	3.6	99.2	6.4
Cyprodinil *	63.1	2.8	60.6	4.8	62.7	2.9
Kresoxim methyl	111.2	4.5	106.6	3.2	112.0	3.0

* Results from modified dispersive SPE method.

Figure 4 shows the recovery and precision results obtained by 1 mL and 8 mL volume dispersive SPE. To simplify the comparison, the average recovery and precision of three fortification concentrations were used for all pesticides. The results of two dispersive SPE cleanup approaches appeared to be independent of volume used. Apparently, both approaches provided efficient and similar sample cleanup, and thus generated relatively equivalent results. However, if 8 mL size dispersive SPE volume is used, two duplicated extractions must be performed initially to complete both original and modified dispersive SPE. If 1 mL size dispersive SPE is used, only one extraction is needed to provide enough volume to perform both original and modified dispersive SPE simultaneously. This is more cost effective saving time, sample amount, and labor. The extractions can be performed according to the user's requirements and regulations.

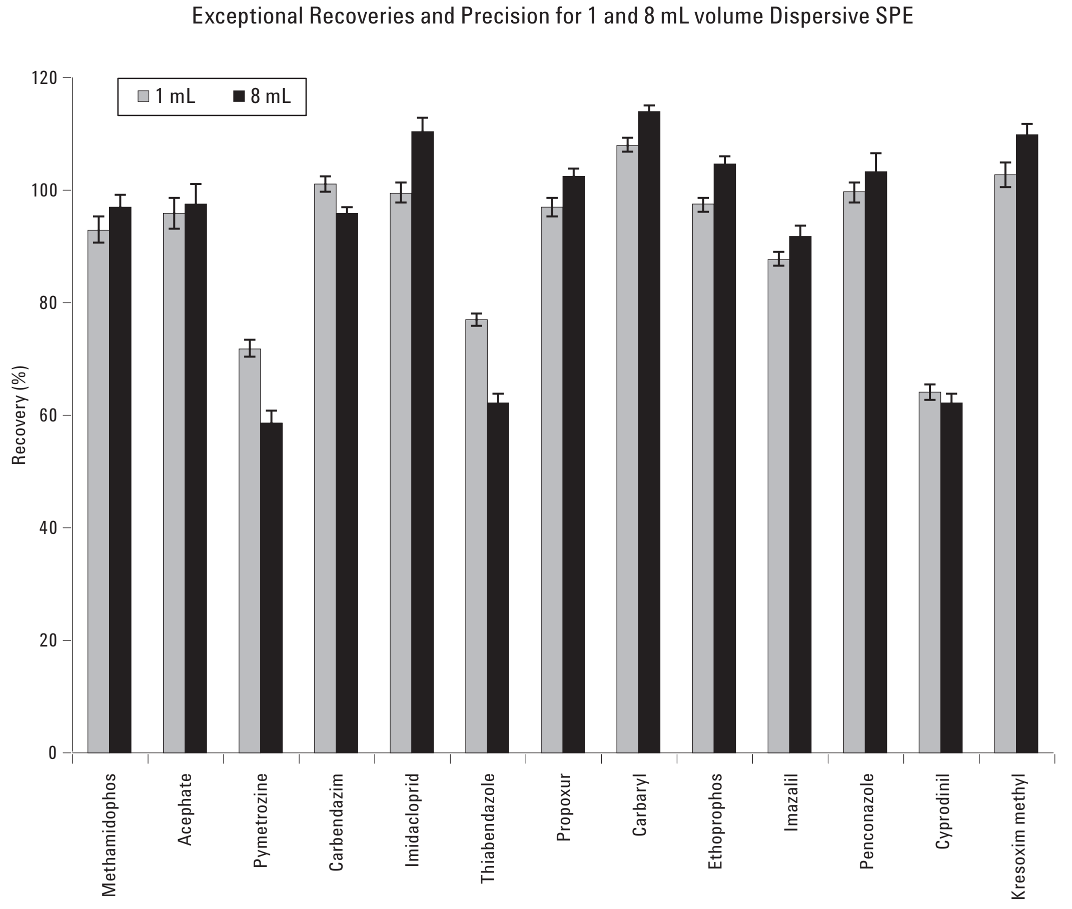


Figure 4. The recovery and precision results for 1 mL dispersive SPE and 8 mL dispersive SPE.

Conclusions

Agilent Bond Elut QuEChERS AOAC buffered extraction kits and dispersive SPE kits for pigmented fruits and vegetables provide a simple, fast and effective method for the purification of representative pesticides in spinach. The modified dispersive SPE method with the addition of toluene provides a very useful way to limit the loss of planar pesticides caused by GCB in dispersive SPE. The recovery and reproducibility of this method, based on matrix spiked standards, were acceptable for multiclass, multi-residue pesticide determination in spinach. The impurities and matrix effects from spinach were minimal and did not interfere with the quantitation of any target compound. As the selected pesticides represented a broad variety of different classes and properties, the Agilent Bond Elut QuEChERS AOAC Buffered Extraction and Dispersive kits for Pigmented Fruits and Vegetables can be used for other pesticides in other similar pigmented matrices.

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