Analysis of Pesticide Residues in Spinach Using Agilent Bond Elut QuEChERS AOAC Kits by GC/MS

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 extraction and cleanup of 18 GC-amenable multiple pesticide class residues in spinach. The 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 addition of toluene was employed for the planar pesticides. The target pesticides in the spinach extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode. The method was validated in terms of recovery and reproducibility. The limit of quantitation (LOQ) for most pesticides is 10 ng/g; however folpet has an LOQ of 50 ng/g in spinach. This application, employing Agilent Bond Elut QuEChERS kits, produced results well below the maximum residue limits (MRLs) for all pesticides screened. The spiked levels for the recovery experiments were 10, 50, and 200 ng/g.

Agilent Technologies

Introduction

The AOAC QuEChERS method has been widely applied for the analysis of pesticides in food since it was introduced by USDA scientists. [1-3] In summary, the method uses a single-step buffered acetonitrile (1% HAc) extraction while simultaneously salting out water from the sample using anhydrous magnesium sulfate ($MgSO_4$) to induce liquid-liquid partitioning. For cleanup, a 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. After mixing and centrifugation, the upper layer is ready for analysis. 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 matrix since it contains large amounts of chloryophyll. Therefore, the dispersive SPE kits with GCB were selected for further clean-up. GCB adsorbs planar molecules such as pigments and sterols; therefore it is very helpful in cleaning-up pigmented matrix like spinach. However, GCB also adsorbs pesticides with planar structure, such as carbendazim, chlorothalonil, and coumaphos. As a result, this kind of dispersive SPE kit is not recommended for the analysis of planar pesticides. Previously, we discussed the impact of toluene addition to the dispersive SPE tube on the analysis of pesticides in pigmented matrices [4]. It turned out that this modification can greatly increase the extraction efficiency of those problematic pesticides. With the combination of the original (w/o toluene) and modified (w/toluene) dispersive SPE, the performance of Bond Elut AOAC Buffered Extraction Kits and Bond Elut AOAC Dispersive SPE kits for pigmented produce was demonstrated to be excellent for the analysis of LC amenable pesticides in spinach. [5]

In this study, the performance of the Bond Elut AOAC Buffered Extraction kit (p/n 5982-5755) and Bond Elut AOAC Dispersive-SPE kits for Pigmented Fruits and Vegetables (p/n 5982-5222 and 5982-5258) was evaluated for the extraction of volatile and semi-volatile pesticides. Analysis was performed by GC/MS. Seventeen GC-amenable pesticides were selected which represent multiple classes, including nonpolar organochlorine pesticides (OCs), certain organophosphorus pesticides (OPs) and organonitrogen pesticides (ONs). Table 1 shows the chemical and regulatory information for these pesticides in spinach.

Experimental

Reagents and Chemicals

All reagents and solvents were high-performance liquid chromatography (HPLC) or analytical grade. Methanol (MeOH) and toluene were from Honeywell (Muskegon, MI, USA), acetonitrile (ACN) and glacial acetic acid (HAc) were from Sigma-Aldrich (St Louis, MO, 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), Chem Service (West Chester, PA, USA), or Ultra Scientific (North Kingstown, RI, USA).

Solutions and Standards

A 1% acetic acid in ACN solution was prepared by adding 10 mL of HAc to 1 L of ACN.

Standard and internal standard (IS) stock solutions (2 mg/mL) were made in MeOH, respectively, and stored at –20 °C. 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 2.5 µg/mL standard solution in ACN containing 0.1% FA was used to prepare the calibration curves in the matrix blank extract by appropriate dilution. A 15 µg/mL of TPP spiking solution in 1:1 ACN/H_2O containing 0.1% FA was used as the internal spiking standard (IS).

Equipment and Material

Agilent Gas Chromatograph (Agilent Technologies Inc., Santa Clara, CA, USA).

Agilent 5975C Mass Spectrometer (Agilent Technologies Inc., Santa Clara, CA, USA).

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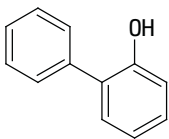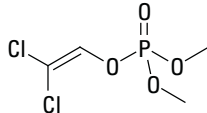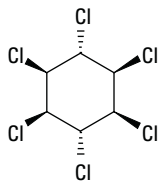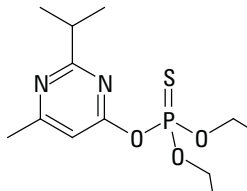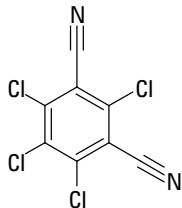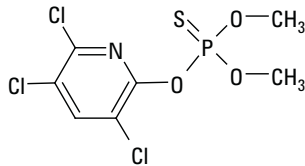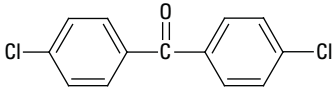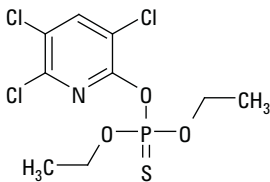
Agilent Bond Elut QuEChERS AOAC dispersive SPE kits for Pigmented Fruits and Vegetables, p/n 5982-5222 and 5982-5258 (Agilent Technologies Inc., Wilmington, DE, USA).

CentraCL3R Centrifuge (Thermo IEC, MA, USA)

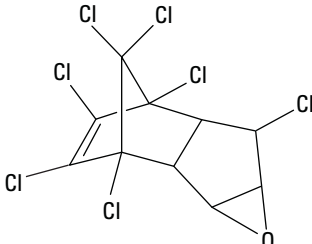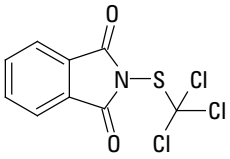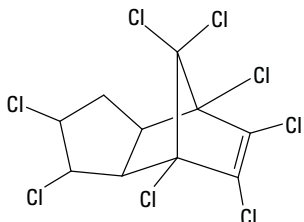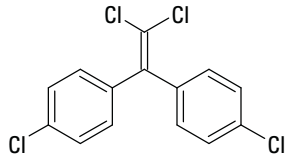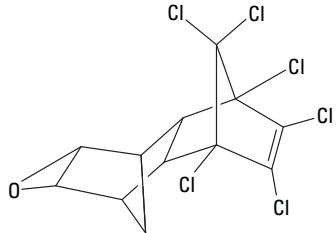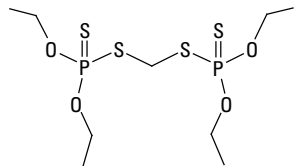Bottle top dispenser (VWR, So Painfield, NJ, USA)

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

Table 1.   Pesticides Chemical and Regulatory Information [6–9]

Name	Category	Log P	pKa	Structure	MRLs in spinach (ng/g)*
σ-Phenylphenol	Phenol	3.18	9.4		2000
Dichlorvos	Organophosphate	1.9	NA		10
Lindane	Organochlorine	3.69	NA		10
Diazinon	Organophosphate	3.69	2.6		100
Chlorothalonil	Chloronitrile	2.94	NA		4000
Chlorpyrifosmethyl	Organophosphate	4.00	NA		30
Dichlorobenzophenone	Organochlorine	4.44	NA		500
Chlorpyrifos	Organophosphate	4.7	NA		10

(Continued)

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Table 1. Pesticides Chemical and Regulatory Information [6–9]

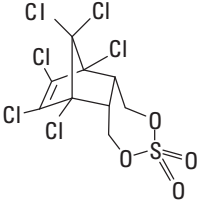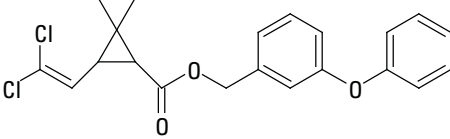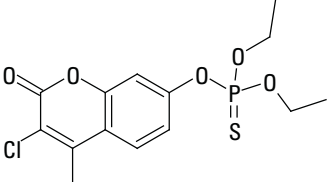| Name	Category	Log P	pKa	Structure	MRLs in spinach (ng/g)*
Heptachlor epoxide	Organochlorine	5.83	NA		30
Folpet	Phthalimide	3.02	NA		2000
Chlordane	Cyclodiene organochlorine	2.78	NA		20
DDE	Organochlorine	6.55	NA		50
Dieldrin	Chlorinated hydrocarbon	3.7	NA		10
Ethion	Organophosphate	5.07	NA		300

(Continued)

4

Table 1.   Pesticides Chemical and Regulatory Information [6–9]

Name	Category	Log P	pKa	Structure	MRLs in spinach (ng/g)*
Endosulfan sulfate	Organochlorine	3.13	NA		50
Permethrins	Pyrethroid	6.1	NA		50
Coumaphos	Organothio phosphate	3.86	NA		100

*The MRLs numbers list in the table are for apple or lowest level in other fruit and vegetables. They could be higher in different commodities.

Instrument Condition

An Agilent GC/MS method for pesticides analysis was used for this study. [10]

GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (p/n 5188-5398)
Carrier gas:	Helium
Inlet pressure:	19.6 psi (constant pressure mode) during run 1.0 psi during backflush
Inlet temperature:	250 °C
Injection volume:	1.0 µL
Purge flow to split vent:	30 mL/min at 0.75 min
Oven temperature program:	70 °C (1 min), 50 °C/min to 150 °C (0 min), 6 °C/min to 200 °C (0 min), 16 °C/min to 280 °C (6 min)
Post run:	3 min
Capillary flow technology:	Purged Ultimate Union (p/n G3186B) - used for backflushing the analytical column and inlet.
	Aux EPC gas: Helium plumbed to Purged Ultimate Union
Aux EPC pressure:	4.0 psi during run, 80.0 psi during backflush
Column:	Agilent J&W HP-5MS Ultra Inert 15 m × 0.25 mm, 0.25 µm (p/n 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (p/n: G3186B)
Restrictor:	65 cm x 0.15 mm, 0.15 µm DB-5MS Ultra Inert
Connections:	Between the Purged Ultimate Union and the MSD

MS conditions

Tune file	Atune.u
Mode	SIM (refer to Table 2 for settings in detail)
Source, quad, transfer line temperature	230 °C, 150 °C and 280 °C respectively
Solvent delay	2.30 min
Multiplier voltage	Autotune voltage

Sample Preparation

The sample preparation procedure includes sample comminution, extraction and partitioning and dispersive SPE clean-up. This process has been described in detail in previous application notes. [8] The procedure used for spinach was similar with the exception of the dispersive SPE clean-up step which includes toluene addition.

The frozen chopped organic spinach was homogenized thoroughly. A 15 g (±0.1 g) amount of homogenized sample was placed into a 50 mL centrifuge tube. Samples were fortified with appropriate QC spiking solutions (100 µL) when necessary, then fortified with 100 µL of IS spiking solution (15 µg/mL of TPP). After vortexing the sample for 30s, 15 mL of 1% HAc 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.

Next, the ACN extracts were separated into two parts for both original and modified dispersive SPE methods. The modified dispersive SPE method has a different procedure; therefore, it is described below in detail. The volume of ACN extracts (about 14 mL) will be enough for simultaneously processing samples with original and modified dispersive SPE when using the 2 mL size dispersive SPE tube. If you are using the 15 mL size tube, then 14 mL of ACN extracts from the one sample will not be enough for processing dispersive SPE by the two methods (since 8 mL are required for each dispersive SPE method). Therefore, another sample must be extracted from the beginning.

Table 2.   Instrument Acquisition Data Used for the Analysis of 18 Pesticides by GC/MS

Analyte	SIM	Collection window (min)	RT (min)
(1) Dichlorvos	184.9	2.3 − 4.0	2.88
(2) o-Phenylphenol	170.1, 169.1	4.0 − 5.0	4.35
(3) Lindane	180.9, 182.9	5.0 − 6.9	6.67
(4) Diazinon	137.1, 179.1	6.9 − 7.7	7.19
(5) Chlorothalonil	265.8, 263.8	6.9 − 7.7	7.34
(6) Chlorpyrifos-methyl	285.9, 287.9	7.7 − 8.6	8.25
(7) Dichlorobenzophenone	250.0,139.0	8.6 − 10.0	9.55
(8) Chlorpyrifos	196.8, 198.8	8.6 − 10.0	9.57
(9) Heptachlor epoxide	352.8, 354.8	10.0 − 10.4	10.31
(10) Folpet	259.9, 261.9	10.4 − 10.85	10.75
(11) γ-Chlordane	372.8, 374.8	10.85 − 11.6	10.97
(12) DDE	245.9, 317.9	10.85 − 11.6	11.21
(13) α-Chlordane	372.8, 374.8	10.85 − 11.6	11.50
(14) Dieldrin	262.9, 264.9	11.0 − 12.3	11.89
(15) Ethion	230.9	12.3 − 13.6	12.97
(16) Endosulfan sulfate	273.8	12.3 − 13.6	13.35
TPP (IS)	325.1, 326.1	13.6 − 15.0	13.84
(17) Permethrin	183.1	15.0 − 23.0	15.69, 15.79
(18) Coumaphos	362.0, 225.9	15.0 − 23.0	15.83

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 an 8 mL aliquot was transferred 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, and 3 mL of toluene were added to the 15 mL tube. The tubes were tightly capped and vortexed for 1 min. We suggest vortexing the tubes for a few seconds before adding the sample, to prevent possible agglomerates. The 2 mL tubes were centrifuged with a micro-centrifuge at 13,000 rpm for 2 min, and the 15 mL tubes were centrifuged in a standard centrifuge at 4000 rpm for 5 min. An 825 µL amount 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, the reconstituted samples were transferred directly into autosampler vials for GC/MS injection. The reconstituted blank samples were directly used to prepare the calibration curve.

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, 2 mL size) for spinach samples.

Weigh 15 g spinach sample (± 0.1 g) in 50 mL centrifuge tube

↓

Spike 100 µL of IS and QC spike solution (if necessary), vortex 1 min.

↓

Add 15 mL of 1% HAc in ACN, and Bond Elut QuEChERS AOAC extraction kit

↓

Cap and shake vigorously by hand for 1 min, centrifuge at 4000 rpm for 5 min

Original method

Transfer 1 mL of ACN extracts to
2 mL dispersive SPE tube

↓

Vortex 30 sec

↓

Centrifuge at 13,000 rpm for 2 min

↓

Transfer certain volume for
GC/MS

Modified method

Transfer 1 mL of ACN extracts to
2 mL dispersive SPE tube

↓

Add 375 µL of Toluene

↓

Vortex 30 sec

↓

Centrifuge at 13,000 rpm for 2 min

↓

Transfer 825 µL of upper ACN layer to another tube

↓

Dry with N_2 flow at 30°C

↓

Reconstitute into 600 µL of 0.1%FA in ACN

↓

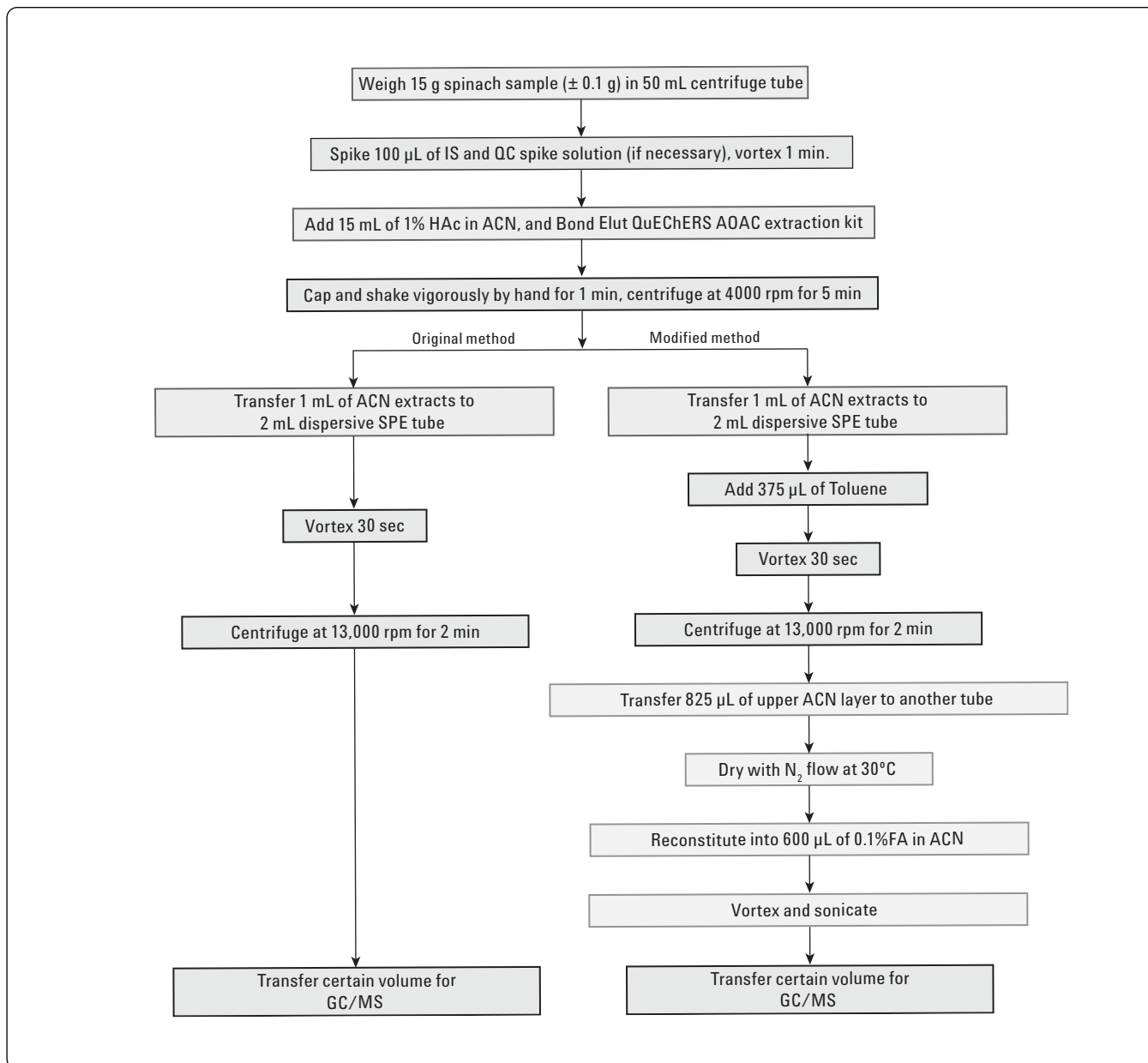Vortex and sonicate

↓

Transfer certain volume for
GC/MS

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

Results and Discussion

The 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 greatly remove pigments and sterols. In order to address the significant loss of planar pesticides, toluene was added to increase the extraction efficiency of those pesticides. Previously we discussed that the addition of toluene retained more matrix impurities in the final sample. [4] In the application using LC/MS/MS, there's no chromatographic differences between the samples processed by the original and modified methods due to the powerful selectivity of LC/MS/MS. The selectivity of GC/MS (SIM mode) is not as powerful as that of

LC/MS/MS (MRM mode). In GC/MS there are interference peaks apparent in the blank chromatogram. Fortunately most of the pesticides tested are free of co-eluting interferences. There was also an interference eluting at a retention time very close to that of o-phenylphenol, and this cannot be differentiated for quantitation. The response of this interferent within the blank was integrated to be less than 20% of the response of the o-phenylphenol peak at the LOQ (10 ng/g) sample. Therefore, it was considered selectivity-acceptable for this compound. Finally, the GC/MS blank chromatograms showed minor differences from samples processed by the original and modified methods, but these differences did not affect the analysis of the target analytes. Figure 2 and Figure 3 show the GC/MS chromatograms of matrix blank (IS spiked) and 50 ng/g fortified spinach extract processed by the original and modified dispersive SPE methods.

GC/MS Chromatograms of Spinach Matrix Blank with Minors Differences Detected
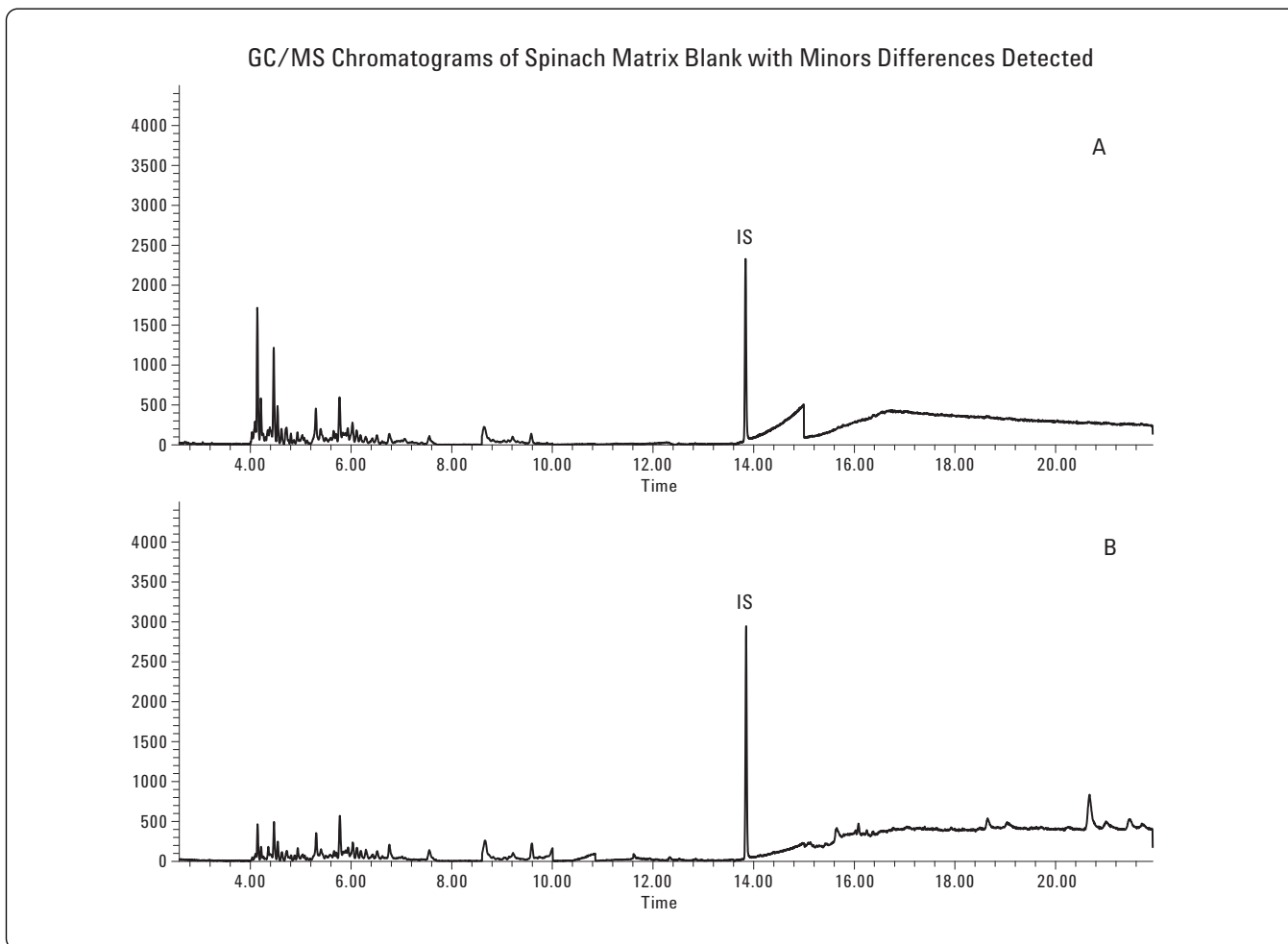
Figure 2.   GC/MS chromatograms of spinach matrix blank processed by original dispersive SPE (A) and modified dispersive SPE (B). IS: Internal Standard TPP.
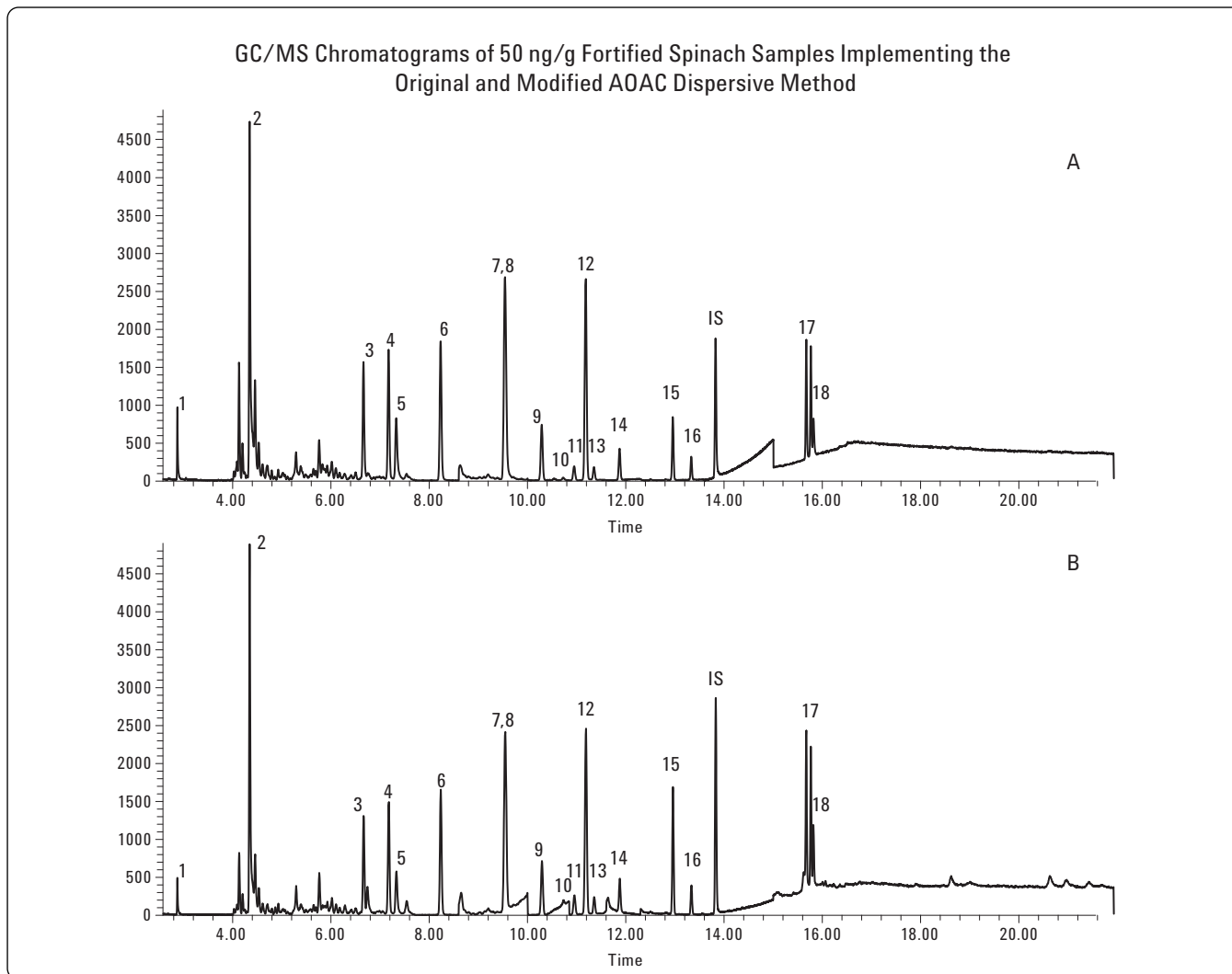
GC/MS Chromatograms of 50 ng/g Fortified Spinach Samples Implementing the Original and Modified AOAC Dispersive Method

Figure 3.   *GC/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Diachlorvos, 2. o-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil 6. Chloropyrifos methyl 7. Dichlorobenzophenone, 8. Chlorpyrifos, 9. Heptachlor epoxide, 10. Folpet, 11. γ-Chlordane, 12. DDE, 13. α-Chlordane, 14. Dieldrin, 15. Ethion, 16. Endosulfan sulfate, 17. Permethrin, 18. Coumaphos. IS: Internal Standard, TPP.*

Linearity and Limit of Quantification (LOQ)

The linear calibration range for all of the pesticides was 10–400 ng/g, except folpet, which was 50–400 ng/g. For the sample processed by original and modified methods, the corresponding matrix blank was used to prepare the calibration curves respectively. Calibration curves, spiked in matrix blanks, were made at levels of 10, 20, 50, 100, 250, and 400 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 10 ng/g quantification limits LOQ (10 ppb) and 50 ng/g LOQ for folpet (50 ppb) established for the pesticides are substantially lower than many MRLs of those pesticides in fruit and vegetables. The regression fit used for the calibration curves was the average response factor. 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 17 Pesticides in Spinach Extract

Pesticide	1 mL dispersive SPE		8 mL dispersive SPE	
	Linear Term	RF Rel Std Dev (%)	Linear Term	RF Rel Std Dev (%)
Dichlorvos	5.55e-001	9.1	4.71e-001	6.8
o-Phenylphenol	2.93e+000	7.9	2.30e+000	9.3
Lindane	8.34e-001	9.5	6.98e-001	8.1
Diazinon	1.03e+000	8.7	9.25e-001	11.2
Chlorothalonil *	7.67e-001	14.2	7.83e-001	13.7
Chlorpyrifos methyl	1.26e+000	12.5	1.20e+000	10.3
Dichlorobenzophenone *	3.03e+000	9.8	2.61e+000	12.2
Chlorpyrifos	6.46e-001	6.9	5.99e-001	11.5
Folpet *,**	4.36e-002	10.2	3.33e-002	11.4
γ-Chlordane	1.73e-001	6.2	1.38e-001	6.4
DDE	2.98e+000	7.4	2.49e+000	6.2
α-Chlordane	1.37e-001	8.1	1.07e-001	8.1
Dieldrin	3.41e-001	5.1	2.92e-001	10.6
Ethion	1.07e+000	15.6	1.42e+000	12.2
Endosulfan sulfate	3.06e-001	4.6	2.42e-001	4.2
Permethrin	1.14e+000	6.4	1.22e+000	9.7
Coumaphos *	3.45e-001	6.4	2.64e-001	11.3

* Results from modified dispersive SPE
** Calibration curve range: 50 – 400 ng/g.

Recovery and Reproducibility

The recovery and reproducibility were evaluated by spiking pesticides standards in comminuted 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 in replicates of six (n = 6) at each level. The recovery and reproducibility (shown as % RSD) data for 1 mL and 8 mL dispersive SPE are shown in Table 4 and Table 5, respectively. Since it was demonstrated that the dispersive SPE size (1 mL and 8 mL) didn't affect the results, the 8 mL size modified dispersive SPE test was not performed due to sample volume limitation. In the 18 GC-amenable pesticides we screened, four pesticides, chlorothalonil, dichlorobenzophenone, folpet, and coumaphos, were found to be adversely affected by the GCB in the dispersive SPE step. With the addition of toluene, the recoveries of those pesticides were increased from 50% to 200% with better precision. However, the modified method also reduced the recovery of certain pesticides that had generated good results originally.

Therefore, the quantitation results shown here are the combination of 14 pesticides from original dispersive SPE and four pesticides from the modified method. It can be seen from the results that the 14 pesticides processed by the original method give out good recoveries (average of 88.8% for 1 mL and 86.3% for 8 mL) and precision (average of 5.4% RSD for 1 mL and 4.8% RSD for 8 mL). Although the four pesticides processed by the modified method give lower recovery (average of 75.3% for 1 mL) but great precision (average of 6.1% RSD for 1 mL), the results were much better than the results obtained by original methods (average recovery of 41.7% with 14.9% average RSD). Please refer to the previous application note [4] for discussions in more detail. Folpet was quantified, but the LOQ was found to be 50 ng/g due to poor sensitivity.

Table 4.   Spinach AOAC Dispersive, 1 mL Sample Volume, 2 mL Tube, LC/MS/MS Results

Pesticide	Low QC (10 ng/g)		Mid QC (50 ng/g)		High QC (200 ng/g)	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Dichlorvos	94.0	3.0	91.7	10.5	80.9	4.6
o-Phenylphenol	95.0	2.2	92.0	7.9	78.7	3.8
Lindane	83.7	3.1	93.9	12.2	91.8	3.3
Diazinon	97.3	4.3	95.6	9.9	91.8	3.3
Chlorothalonil *	47.5	6.8	44.9	6.6	49.4	4.3
Chlorpyrifos methyl	74.1	4.6	71.7	4.5	72.2	5.8
Dichlorobenzo Phenone *	97.5	7.6	66.8	3.9	68.8	6.8
Chlorpyrifos	88.3	3.0	79.6	3.5	77.0	3.5
Heptachlor epoxide	74.9	1.9	81.6	11.7	78.2	3.9
Folpet *	NA	NA	98.8	6.0	77.7	6.7
γ-Chlordane	106.0	4.9	112.2	3.3	93.6	5.3
DDE	80.3	2.2	86.8	9.6	75.4	3.5
α-Chlordane	107.6	4.2	108.4	3.5	91.6	3.7
Dieldrin	99.7	2.6	93.7	9.6	78.9	3.4
Ethion	91.4	3.4	100.0	5.0	107.4	7.6
Endosulfan sulfate	93.7	4.8	97.3	8.8	89.8	4.3
Permethrin	84.7	5.7	74.8	9.9	84.6	6.0
Coumaphos *	98.4	5.5	84.2	9.5	81.2	3.2

* Results from modified dispersive SPE method.

Table 5. Spinach AOAC Dispersive 8 mL Volume, 15 mL Tube, Results by GC/MS

Pesticide	Low QC (10 ng/g) Recovery	RSD	Mid QC (50 ng/g) Recovery	RSD	High QC (200 ng/g) Recovery	RSD
Dichlorvos	93.7	2.6	92.5	4.2	86.2	5.9
o-Phenylphenol	87.9	5.1	92.5	6.6	95.2	6.3
Lindane	83.1	5.1	85.4	2.9	84.5	5.2
Diazinon	85.8	6.9	85.2	2.9	87.3	5.5
Chlorothalonil*	21.1	49.7	23.6	14.3	23.2	14.0
Chlorpyrifos methyl	76.4	2.4	73.9	2.7	73.8	3.6
Dichlorobenzophenone*	93.3	4.1	56.6	2.0	61.4	4.5
Chlorpyrifos	77.8	3.6	70.2	4.6	69.0	3.1
Heptachlor epoxide	78.6	4.6	79.6	2.6	85.3	5.1
Folpet*	NA	NA	60.3	17.4	53.7	10.7
γ-Chlordane	106.8	5.7	110.7	3.5	100.4	4.9
DDE	80.8	4.2	81.8	2.6	81.3	4.9
α-Chlordane	104.2	6.2	103.6	3.3	95.8	5.3
Dieldrin	96.4	6.2	93.0	1.2	79.3	5.3
Ethion	83.8	4.0	82.8	2.3	85.3	4.9
Endosulfan sulfate	90.5	8.8	87.5	7.3	84.5	6.4
Permethrin	84.0	4.9	78.4	6.8	79.5	10.7
Coumaphos*	61.2	22.2	42.6	28.8	35.3	20.6

*Poor results caused by GCB added in dispersive SPE, can be improved by addition of toluene in the 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 volatile to semi-volatile pesticides in spinach. The modified dispersive SPE method with the addition of toluene provides a very useful option to improve the loss of planar pesticides caused by GCB in dispersive SPE

tubes. The recovery and reproducibility, based on matrix spiked standards, were acceptable for multiclass, multi-residue pesticide determination in spinach. The impurities and matrix effects from spinach did not interfere with the quantitation of target compounds. 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 similar pigmented matricies.

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