

Innovative Approaches

for today's food analysis challenges

Agilent SampliQ QuEChERS Food Safety Applications Notebook

Volume 2



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Reliable food safety testing begins with reliable Sample Preparation

Dear Valued Customer,

You are committed to producing foods and beverages of consistent quality and uncompromising safety. Your customers demand nothing less.

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From inspection and product development to quality assurance and packaging, Agilent instruments, systems, columns and supplies help your lab meet the toughest standards.

But that's only *part* of the story. Agilent also supports your analytical and business challenges with in-depth experience, broad knowledge, and creative people, plus our keen insight into industry trends and global regulations.

NEW Agilent SampliQ QuEChERS and SPE products: your first step in food safety analysis

High-quality Agilent SampliQ SPE products help you confidently extract and concentrate samples from complex matrices, ensuring fast, accurate, and reproducible results from the very first step. Our family of products includes:

- **Agilent SampliQ QuEChERS kits** enable you to prepare food samples for multi-residue, multi-class pesticide analysis with just a few simple steps.
- **Pre-packed extraction and dispersive SPE kits** are assembled to suit specific food types and screening protocols, eliminating guesswork.
- **Extraction salts** are pre-measured and sealed in anhydrous packets, so you can conveniently add them at precisely the right time.
- **Agilent SampliQ polymers** allow the retention of target molecules over a wide pKa range. And unlike silica-based phases, SampliQ polymers yield the same exacting results if they inadvertently dry out during conditioning.

On the following pages, you'll discover leading-edge techniques and sample prep methods that can dramatically improve the reliability and throughput of your food safety analysis.



Ronald E. Majors, Ph. D., Senior Chemist



What is QuEChERS?

Developed by United States Department of Agriculture in 2003, QuEChERS (pronounced “Catchers”) stands for **Q**uick **E**asy, **C**heap, **E**ffective, **R**ugged and **S**afe – the qualities that describe this sample preparation method for food substances. The technique is very simple, involves a minimum of steps, and is effective for the cleanup of complex samples.

QuEChERS is a technique that was developed for multi-class, multi-residue pesticides analysis in fruits and vegetables but more recently has expanded its scope to other trace contaminants in other non-vegetable foods such as meat and fish. Methods for hundreds of pesticides in a variety of fruits, vegetables, meat, and for dry materials such as beans and nuts have been published. “Official” methods are now available and a standardization of the technique on a worldwide basis is taking place. In the United States, the Association of Official Analytical Chemists (AOAC) has published its 2007.01 Method while the European equivalent, the EN 15662 2007, uses similar methodology.

The practice of QuEChERS involves two steps:

1. An extraction step that is based on partitioning via salting-out extraction involving an equilibrium between an aqueous and an organic layer, and
2. A dispersive solid-phase extraction (SPE) step that involves further cleanup using various combinations of salts and porous sorbents to remove interfering substances.

In the dispersive SPE step, the use of porous sorbents such as a primary-secondary amine (PSA), C18, and graphitized carbon black help to remove a variety of matrix compounds that are co-extracted in step 1. The most popular analytical methodology to measure extracted analytes is either LC/MS or GC/MS or their tandem equivalents.



Original QuEChERS Method



Analysis of Pesticide Residues in Apple by GC/MS using Agilent SampliQ QuEChERS Kits for Pre-injection Cleanup (Publication 5990-4468EN)

Introduction

This application note describes the use QuEChERS, a quick, easy, cheap, effective, rugged, and safe sample preparation approach to investigate the extraction of 15 multi-class pesticides in apples. The pesticides were chosen to represent typical types of volatile/semi-volatile pesticides that might be found in a typical fruit sample at levels normally encountered. The version of the QuEChERS non-buffered extraction method dates back to the original publication in 2003. For analysis, it uses GC/MS with selective ion monitoring (SIM) to measure pesticides down to the 10 ng/g levels.

Instrument conditions

GCMS conditions

Injection source	Manual
Inlet	Splitless
Column	Agilent J&W HP-5ms Ultra Inert GC Capillary Column, 30 m x 0.250 mm, 0.25 µm (Part No. 190915-433UI)
Carrier Gas	Helium in constant flow mode
Oven Temperature Program	70 °C (2 min), 25°C/min to 150 °C (0 min), 3 °C/min to 200 °C (0 min), 8 °C/min to 280 °C (7 min)

Injection volume 1 µL

MS conditions

Tune File	Atune.u
Mode:	SIM
Source, Quad, Transfer line temperature	230 °C, 150 °C, 280 °C respectively
Solvent Delay	4.00 minutes
Multiplier Voltage	Autotune voltage

Ordering information

Agilent SampliQ QuEChERS Non-Buffered Extraction Kit. Part No. 5982-5550.

Agilent SampliQ QuEChERS Dispersive Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 15 mL. Part No. 5982-5058.

Agilent J&W HP-5ms Ultra Inert GC Capillary Column, 30 m x 0.25 mm, 0.250 µm. Part No. 190915-433UI.

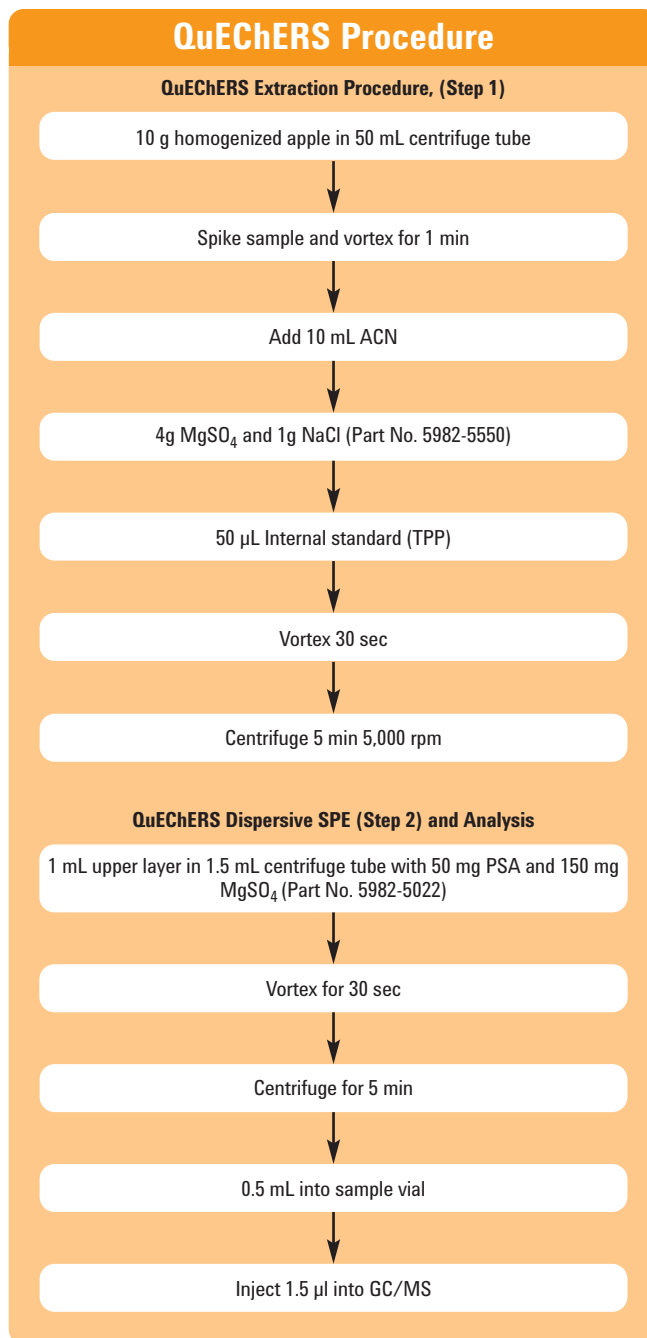


Figure 1: QuEChERS extraction procedure for general fruits and vegetables

Results

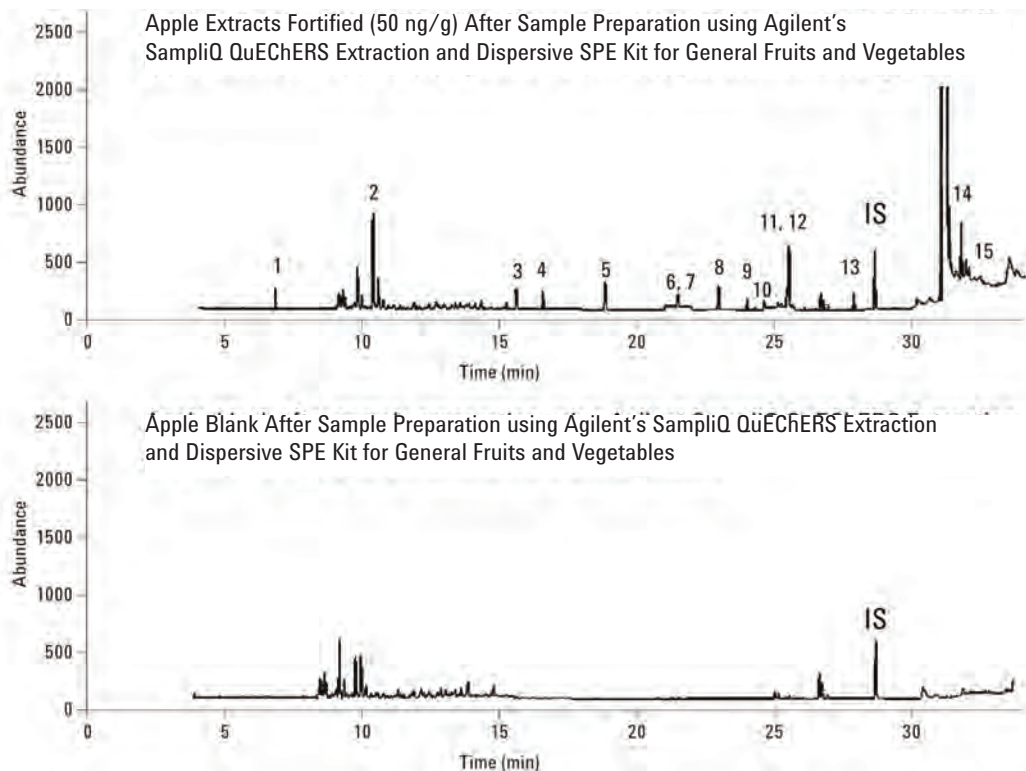


Figure 2: Comparison of blank apple extract to a fortified apple extract

Pesticide	Low-QC 10 ng/g		Mid-QC 50 ng/g		High-QC 200 ng/g	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Dichlorvos	102.8	5.0	96.7	10.8	99.4	2.8
<i>o</i> -phenylphenol	92.0	6.1	79.6	6.8	89.5	6.3
Lindane	97.9	2.0	88.5	9.7	92.6	4.2
Diazinone	90.5	9.1	98.8	5.5	102.1	4.4
Methyl-chlorpyrifos	88.7	7.1	90.0	4.3	98.5	3.1
Chlorpyrifos	93.5	6.5	95.6	4.0	100.2	1.2
Dichlorobenzophenone	90.3	5.0	89.1	6.4	99.4	0.6
Heptachlor-epoxide	87.0	3.2	85.6	5.4	95.4	3.9
γ -chlordane	92.3	3.5	90.0	6.8	95.9	2.0
α -chlordane	95.5	4.7	85.8	6.9	93.5	2.6
Dieldrin	99.4	4.2	93.6	5.3	99.9	1.8
DDE	94.5	4.2	87.1	5.7	92.7	1.9
Endosulfan Sulfate	97.8	2.3	90.8	2.8	99.5	2.3
Permethrin	100.7	4.8	93.0	3.4	97.6	2.1
Coumaphos	72.5	4.5	79.6	3.5	96.6	3.0

Table 1. Recovery and reproducibility of pesticides in apple using the original QuEChERS method (n=4)

To review this Application Note in its entirety, please search for 5990-4468EN at www.agilent.com/chem

EN Methods



Analysis of Pesticide Residues in Apple using Agilent SampliQ QuEChERS European Standard EN Kits by LC/MS/MS Detection (Publication 5990-3938EN)

Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample preparation approach described in the European Committee Standard (EN) for extraction and cleanup of 16 multiple class pesticide residues of interest in apple. The target pesticides in the apple extracts are then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometer (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode.

Instrument conditions

HPLC conditions

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column
Column 3.0 mm x 150 mm, 3.5 μ m
(Part No. 959963-312)

Flow rate: 0.3 mL/min

Column temperature: 30 °C

Injection volume: 10 μ L

Mobile phase: A: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H₂O

B: 5 mM ammonium acetate, pH 5.0 in ACN

Needle wash: 1:1:1 ACN/MeOH/IPA/H₂O (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
	12.0	100	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: 4,000 V

QuEChERS Procedure

Weigh 10 g comminuted sample (\pm 0.05 g) in 50 mL centrifuge tube

Spike samples with 100 μ L of IS solution and vortex for 1 min

Add 10 mL of ACN, and shake 1 min

Add SampliQ EN extraction packet, and shake vigorously by hand for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer 1 mL of upper ACN layer to SampliQ EN dispersive SPE 2 mL tube, or 6 mL to SampliQ EN dispersive SPE 15 mL tube

Vortex 1 min, centrifuge at 13,000 rpm for 2 min for 2 mL tubes or at 4,000 rpm for 5 min for 15 mL tubes

Transfer 200 μ L extract to autosampler vial, add 10 μ L of 1% FA in ACN, and dilute with 800 μ L water

Samples are ready for LC/MS/MS analysis

Figure 1. QuEChERS EN sample preparation procedures flow chart

Results

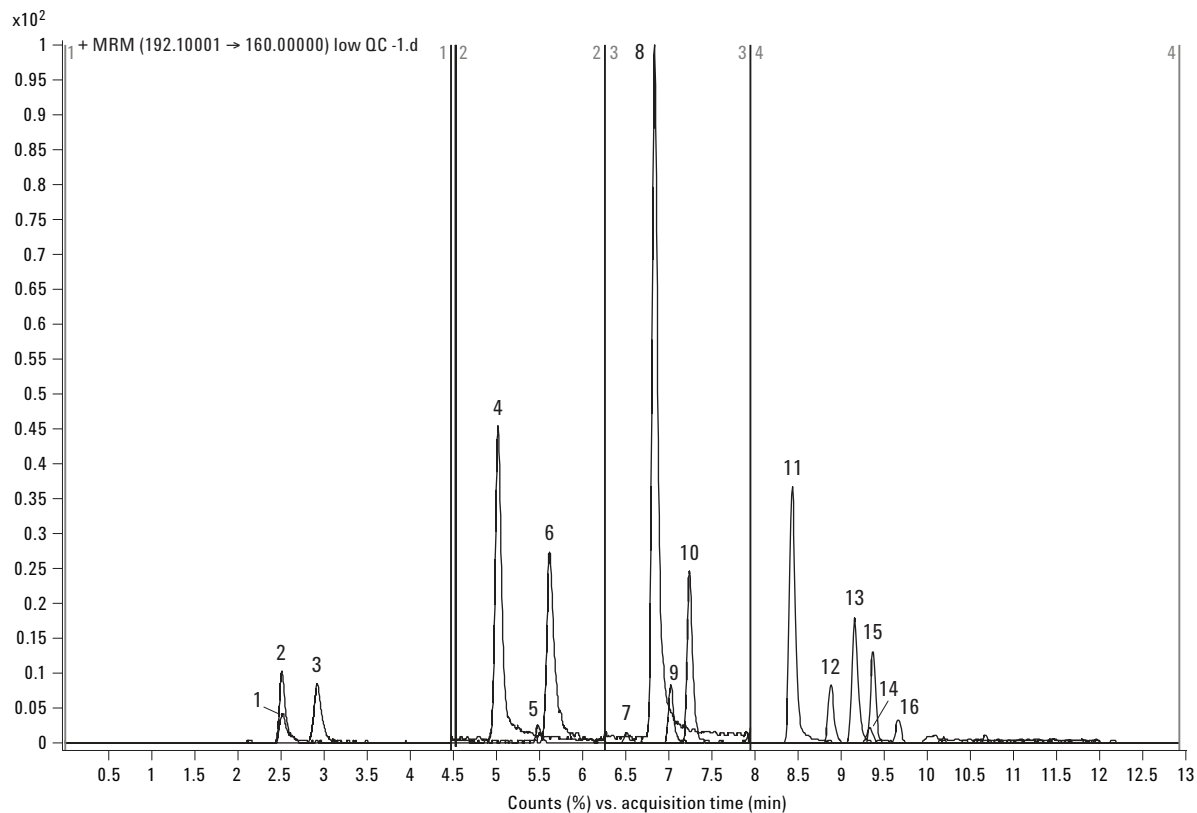


Figure 2. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlofluanid, 15. Kresoxim methyl, 16. Tolyfluanid

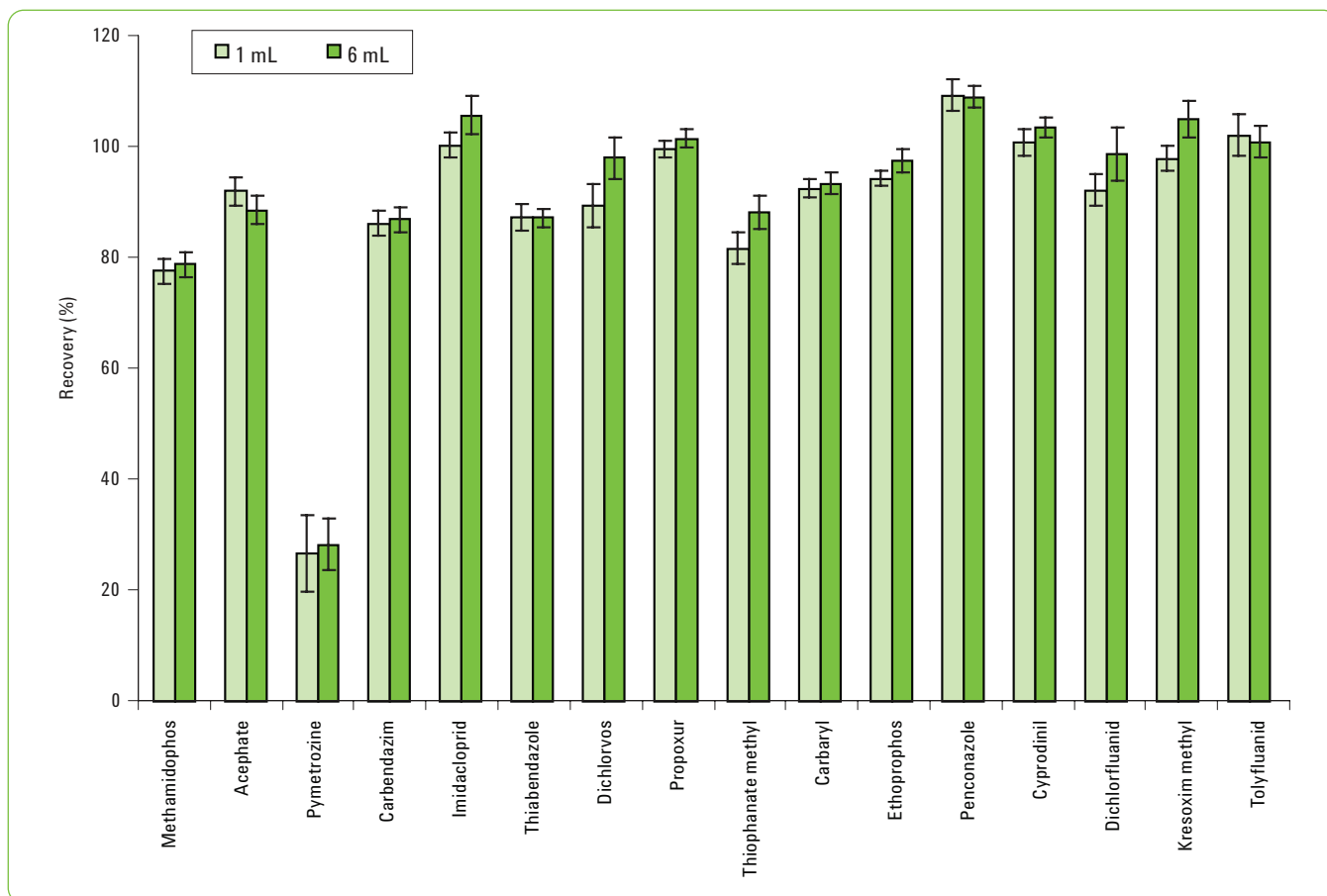


Figure 3. Results comparison of 1 mL dispersive SPE and 6 mL dispersive SPE

Ordering information

Agilent SampliQ QuEChERS EN Method Extraction Kit.
Part No. 5982-5755.

Agilent SampliQ QuEChERS EN Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5021.

Agilent SampliQ QuEChERS EN Dispersive SPE Kit for General Fruits and Vegetables, 15 mL. Part No. 5982-5056.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 mm x 150 mm, 3.5 μ m. Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-3938EN at www.agilent.com/chem

Analysis of Pesticide Residues in Apple Using Agilent SampliQ QuEChERS EN Kits by GC/MS (Publication 5990-4073EN)

Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample preparation approach described in the European Committee (EN) for extraction and cleanup of 17 GC-amenable multiple pesticide class residues in apple. The method involves initial extraction in an aqueous/ acetonitrile system, an extraction/partitioning step after the addition of salt, and a cleanup step using dispersive solid phase extraction (dispersive SPE). The target pesticides in the apple extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

Instrument conditions

GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (Part No. 5188-5398)
Carrier gas:	Helium
Inlet pressure:	20.18 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 (Part No. 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 GC Column, 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (Part No. G3186B)
Restrictor:	65 cm x 0.15 mm x 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 temperatures:	230 °C, 150 °C and 280 °C respectively
Solvent delay:	2.30 min
Multiplier voltage:	Autotune voltage

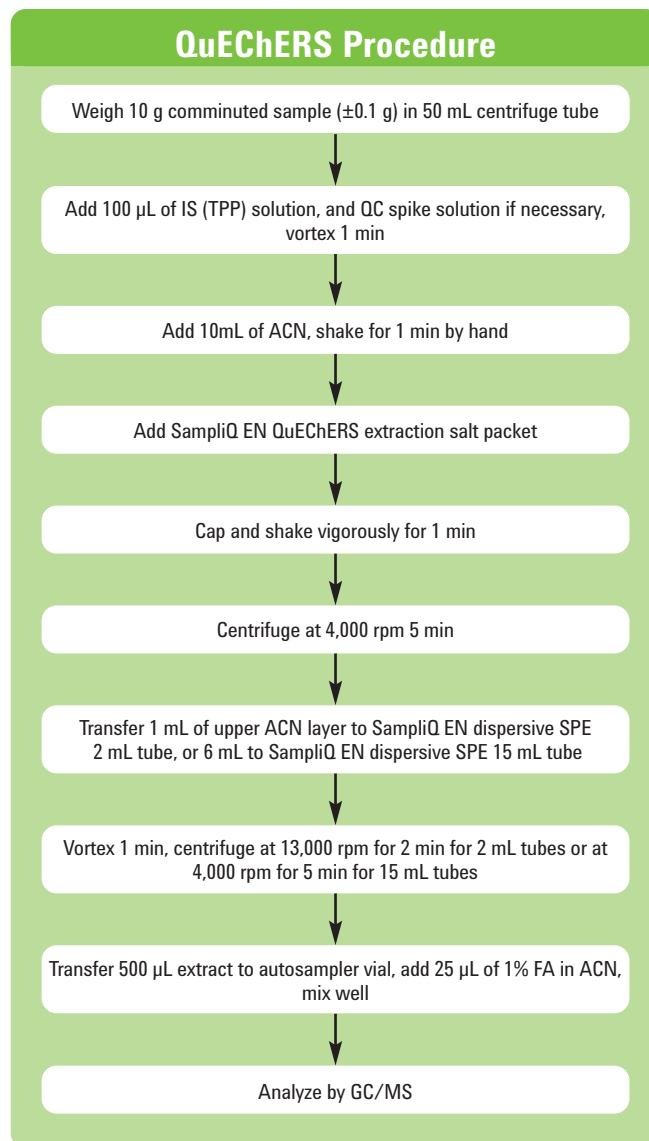


Figure 1. Flow chart of the Agilent SampliQ QuEChERS EN extraction procedure

Results

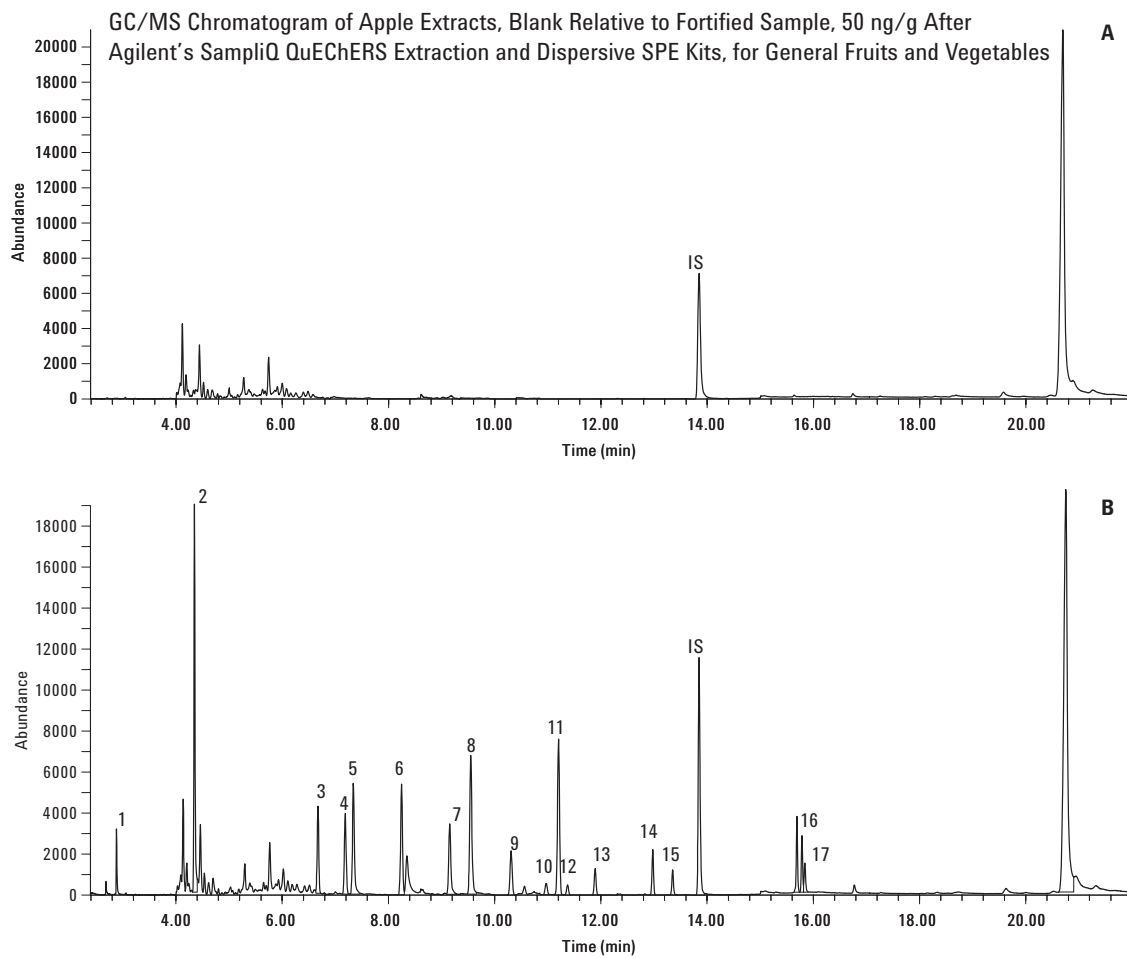


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50 ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2. *o*-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil, 6. Chlorpyrifos-methyl, 7. Dichlofluanid, 8. Dichlorobenzophenone, 9. Heptachlor epoxide, 10. γ -Chlordane, 11. DDE, 12. α -Chlordane, 13. Dieldrin, 14. Ethion, 15. Endosulfan sulfate, 16. Permethrin, 17. Coumaphos. IS: Triphenyl phosphate (TPP)

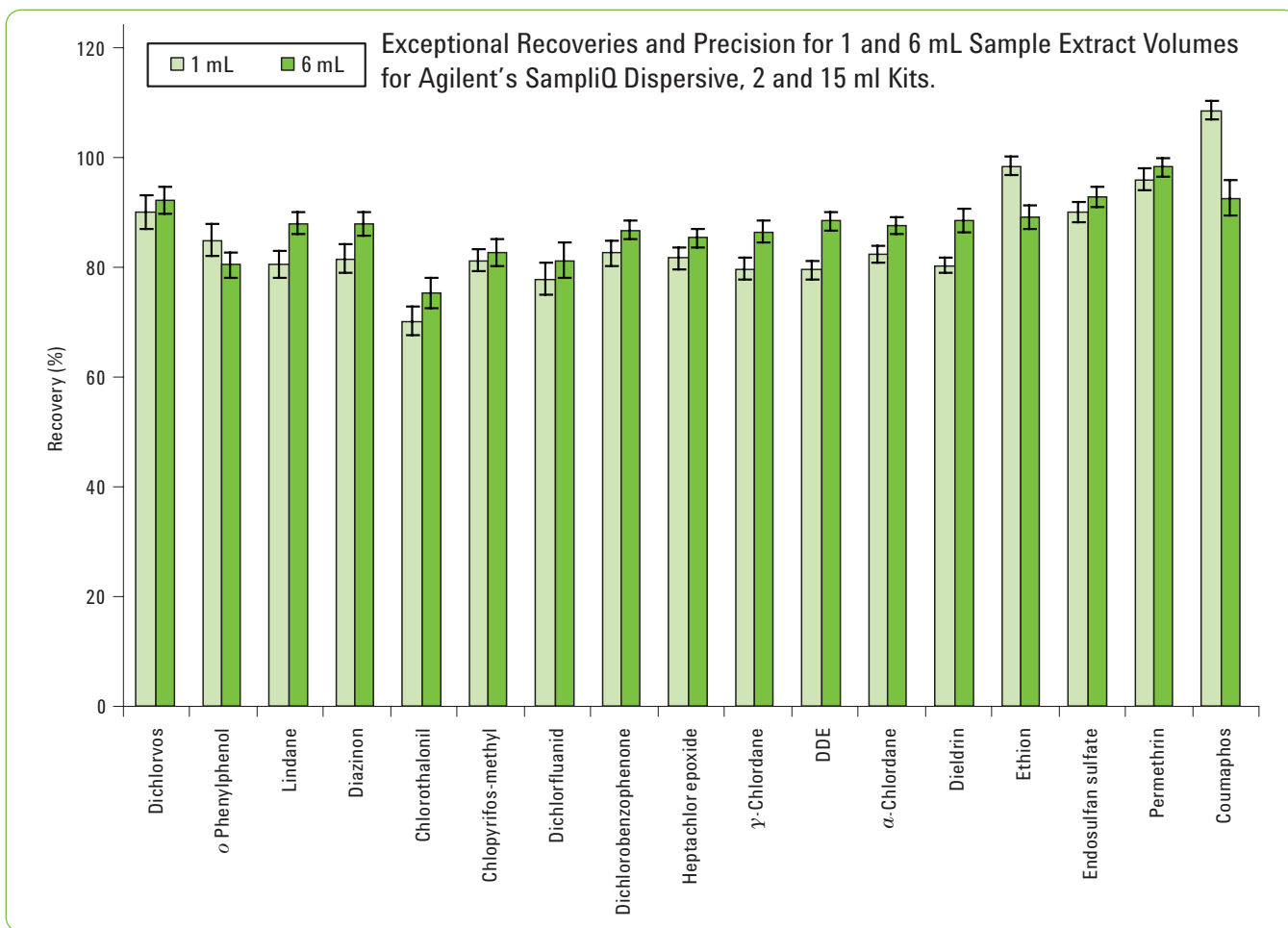


Figure 3. The recovery and precision results of 1 and 6 mL sample volumes employing Agilent's SampliQ Dispersive SPE, 2 and 15 mL Kits, respectively

Ordering information

Agilent SampliQ QuEChERS EN Method Extraction Kit.
Part No. 5982-5650.

Agilent SampliQ QuEChERS EN Method Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5021,
15 mL. Part No. 5982-5056.

Agilent J&W HP-5ms Ultra Inert GC Column,
15 m x 0.25 mm, 0.25 μm. Part No. 19091S-431UI.

To review this Application Note in its entirety, please search for 5990-4073EN at www.agilent.com/chem

Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS EN Kit by LC/MS/MS Detection (Publication 5990-4395EN)

Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) EN sample preparation approach for extraction and cleanup of 13 pesticide residues representing various classes in spinach. Because spinach is considered a highly pigmented matrix, the EN dispersive SPE kit for highly pigmented fruits and vegetables is selected. Graphitized carbon black (GCB) in the amount of 7.5 mg/mL of ACN extract is added to the kit. The target pesticides in the spinach extracts are 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.

Instrument conditions

HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 μ m (Part No. 959963-312)		
Flow rate:	0.3 mL/min		
Column Temperature:	30 °C		
Injection volume:	10 μ L		
Mobile Phase:	A, 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H ₂ O B, 5 mM ammonium acetate, pH 5.0 in ACN		
Needle wash:	1:1:1 ACN/MeOH/IPA/H ₂ O w/0.2% FA.		
Gradient:	Time	% Acetonitrile	Flow rate (mL/min)
	0	20	0.3
	0.5	20	0.3
	8.0	100	0.3
	10.0	100	0.3
	13.0	STOP	
Post run:	4 min		
Total cycle time:	17 min		

MS conditions

Positive mode	
Gas temp.:	350 °C
Gas flow:	10 L/min
Nebulizer:	40 Psi
Capillary:	4,000 V

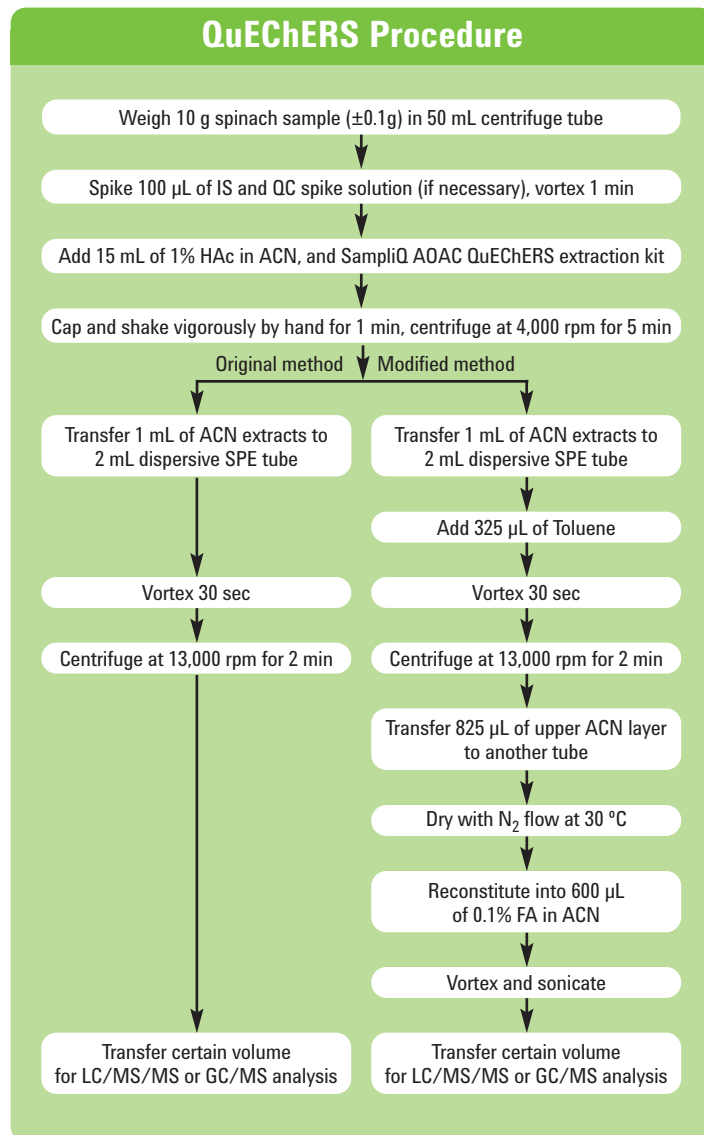


Figure 1. Flow chart of the QuEChERS EN procedure (original and modified dispersive SPE, 2 mL size)

Results

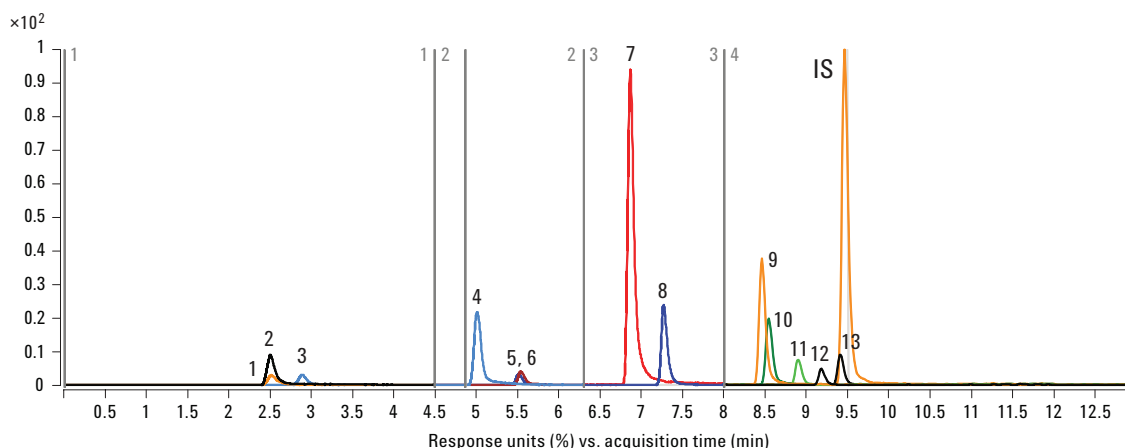


Figure 2. MRM chromatogram of 50 ng/g fortified sample processed by EN method. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos, 10. Imazalil, 11. Penconazole, 12. Cyprodinil, 13. Kresoxim methyl, IS: Internal Standard, TPP

Analytes	10 ng/g fortified QC		50 ng/g fortified QC		200 ng/g fortified QC	
	Recovery	RSD (n=6)	Recovery	RSD (n=6)	Recovery	RSD (n=6)
Methamidophos	85.0	8.3	87.7	2.7	95.0	9.4
Acephate	88.6	5.1	84.6	3.1	94.6	9.3
Pymetrozine*	68.7	3.7	65.7	1.5	71.9	10.8
Carbendazim*	94.0	5.4	91.4	2.7	53.5	.3
Imidacloprid	102.0	8.9	85.4	6.1	100.1	7.7
Thiabendazole*	77.2	4.4	77.6	2.4	79.2	9.7
Propoxur	98.2	5.7	96.3	1.8	93.9	7.2
Carbaryl	98.5	3.6	94.0	1.7	97.4	7.2
Ethoprophos	102.3	6.0	95.3	1.7	91.0	6.8
Imazalil	88.8	6.4	86.8	2.8	93.5	7.7
Penconazole	104.5	2.5	96.4	2.0	84.6	5.5
Cyprodinil*	101.5	4.2	92.2	2.4	86.8	7.6
Kresoxim methyl	99.7	6.1	97.4	1.6	95.3	6.9

* Pesticides with planar structure.

Table 1. Recovery and reproducibility of pesticides in fortified spinach with 6 mL dispersive SPE tube (Part No. 5982-5356)

Ordering information

Agilent SampliQ QuEChERS EN Extraction Kits.

Part No. 5982-5650.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kits for Pigmented Fruits and Vegetables. Part Nos. 5982-5321 and 5982-5356.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm. Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-4395EN at www.agilent.com/chem

AOAC Methods



Analysis of Pesticide Residues in Apples using Agilent SampliQ QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-3937EN)

Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS), Association of Analytical Communities (AOAC) Official Method 2007.01; sample preparation approach for extraction and cleanup of 16 pesticide residues in apple.

The 5 ng/g limit of quantitation (LOQ) for pesticides in apple 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 76 and 117% (95.4% on average), with RSD below 15% (4.3% on average).

Instrument conditions

HPLC conditions

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column
3.0 mm x 150 mm, 3.5 μ m (Part No. 959963-312)

Flow rate: 0.3 mL/min

Column Temperature: 30 °C

Injection volume: 10 μ L

Mobile Phase: A: 5mM NH₄OAc, pH 5.0 in 20:80 MeOH/H₂O

B: 5 mM NH₄OAc, pH 5.0 in ACN

Needle wash: 1:1:1 ACN/MeOH/IPA/H₂O (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
	12.0	100	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: 4,000 V

QuEChERS Procedure

Accurately weigh 15 g homogenized sample (\pm 0.05 g) in 50 mL centrifuge tubes

Spike samples with 100 μ L of IS solution and vortex for 1 min

Add 15 mL of 1% acetic acid in ACN, shake vigorously for 1 min

Add SampliQ QuEChERS AOAC salt packet, cap tubes and shake vigorously for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer upper ACN layer to SampliQ QuEChERS dispersive-SPE tube, 1 mL/2 mL tube or 8 mL/15 mL tube

Vortex 1 min then centrifuge

Transfer 200 μ L extract to autosampler vial, dilute with 800 μ L appropriate solution if necessary

Samples are ready for LC/MS/MS analysis

Figure 1. QuEChERS AOAC sample preparation procedures flow chart

Ordering information

Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.
Part No. 5982-5755.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 15 mL. Part No. 5982-5058.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 mm x 150 mm, 3.5 μ m. Part No. 959963-312.

Results

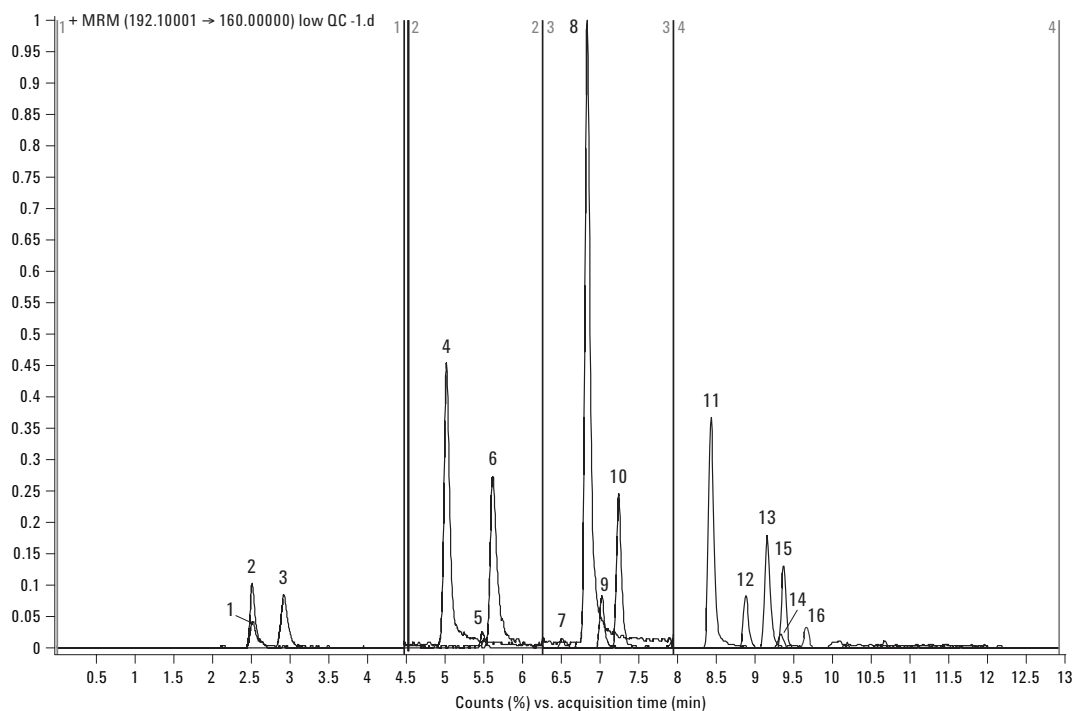


Figure 2. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlorfluandil, 15. Kresoxim methyl, 16. Tolyfluandil

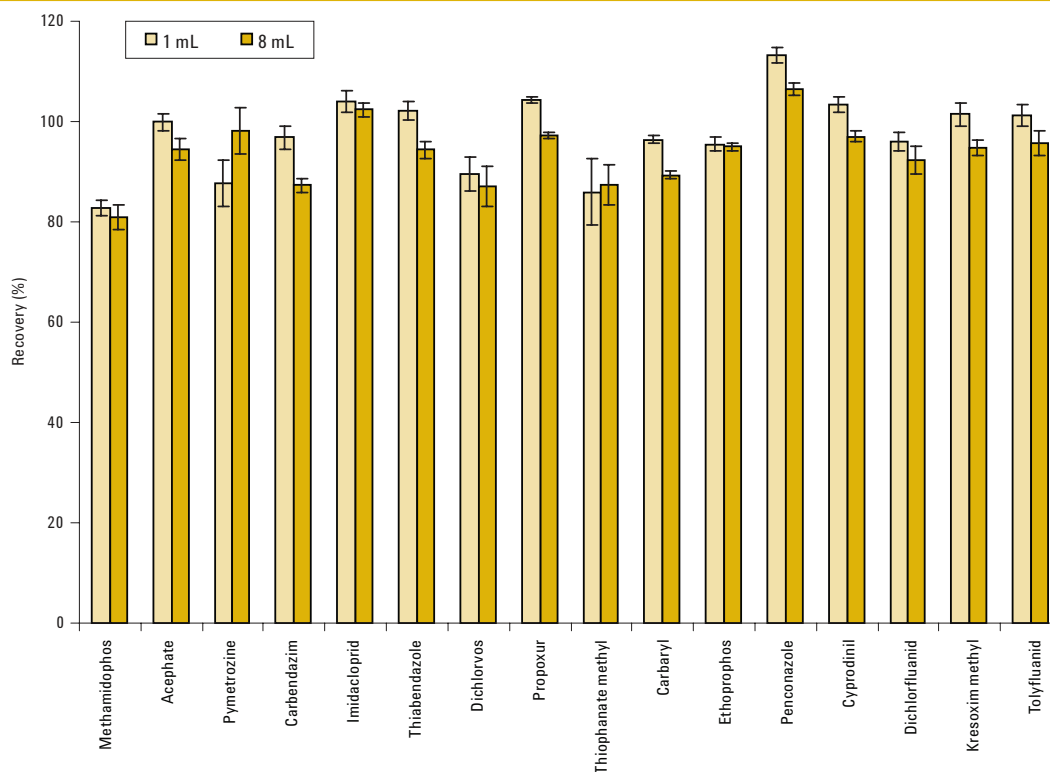


Table 1. Results comparison of 1 mL and 8 mL dispersive SPE sample volume

To review this Application Note in its entirety, please search for 5990-3937EN at www.agilent.com/chem

Analysis of Pesticide Residues in Apple Using Agilent SampliQ QuEChERS AOAC Kits by GC/MS (Publication 5990-4068EN)

Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample preparation approach for extraction and cleanup of 17 GC-amenable pesticide residues from multiple classes, in apple. The target pesticides in the apple extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

Instrument conditions

GC conditions

Auto-sampler:	Agilent 7683 automatic liquid
Inlet:	Splitless
Column:	Agilent J&W HP-5ms Ultra Inert GC Column 30 m x 0.25 mm, 0.25 µm (Part No. 19091S-433UI)
Carrier gas:	Helium in the constant pressure
Retention time locking:	Chlorpyrifos-methyl locked to 16.596 min (nominal Column head pressure=22.0 psi)
Oven temperature: program:	70 °C (2 min), 25 °C/min to 150 °C (0 min), 3 °C /min to 200 °C (0 min), 8 °C/min to 280 °C (11.5 min)
Injection volume:	1.0 µL

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:	3.00 min
Multiplier voltage:	Autotune voltage

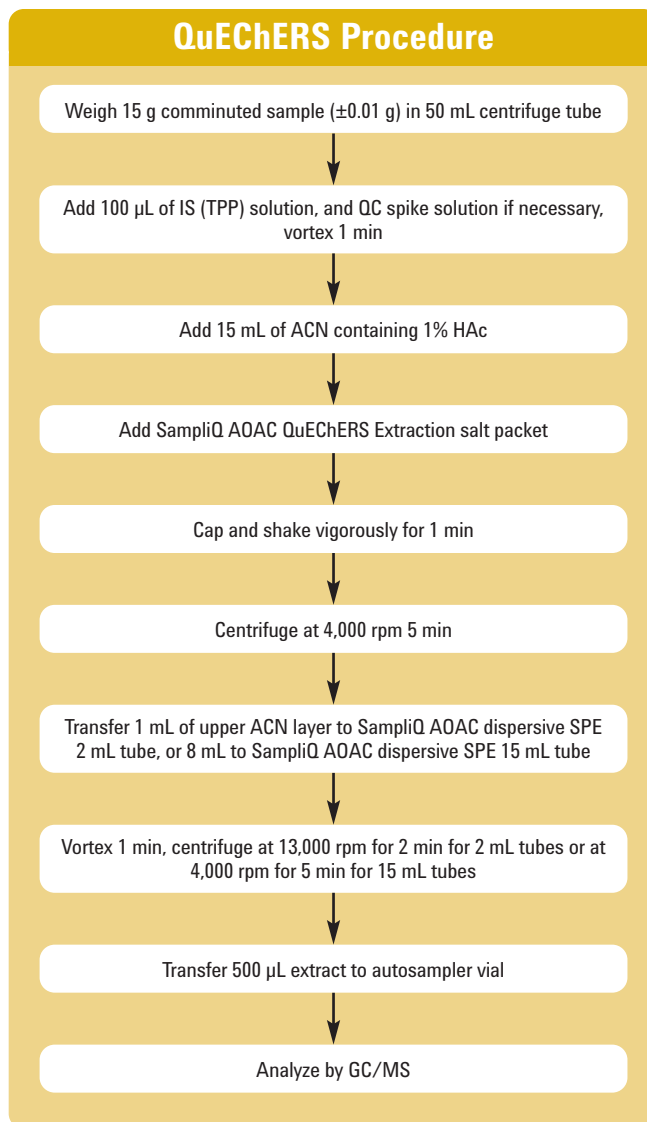


Figure 1. Flow chart of the Agilent SampliQ QuEChERS AOAC extraction procedure

Ordering information

Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.
Part No. 5982-5755.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 15 mL. Part No. 5982-5058.

Agilent J&W HP-5ms Ultra Inert GC Column, 30 m x 0.25 mm, 0.25 µm. Part No. 19091S-433UI.

Results

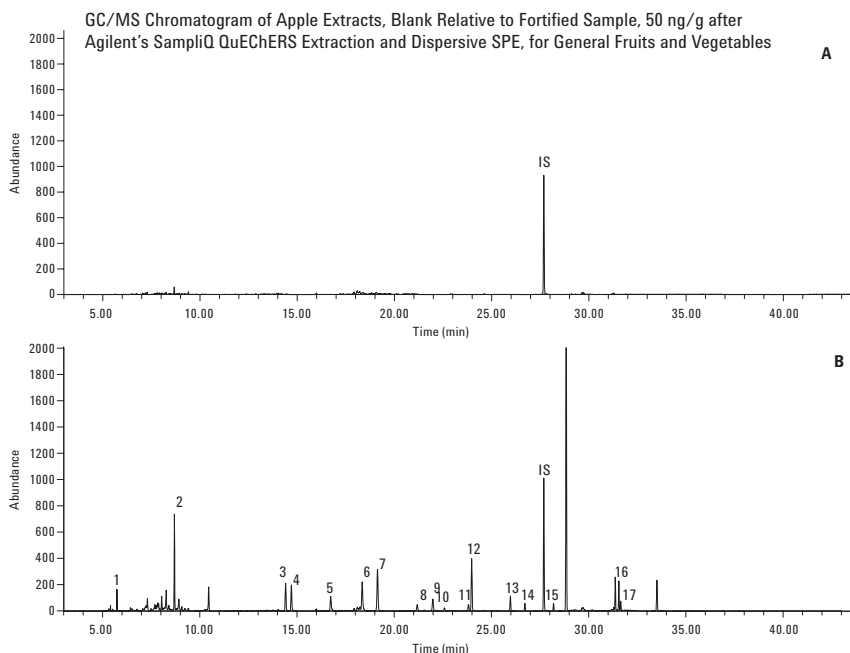


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50 ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2. σ -Phenylphenol, 3. Diazinon, 4. Chlorothalonil, 5. Carbaryl, 6. Dichlorfuanid, 7. Dichlorobenzophenone, 8. Folpet, 9. α -Chlordane, 10. Endosulfan, 11. Dieldrin, 12. DDE, 13. Ethion, 14. Endosulfan sulfate, 15. Endrin ketone, 16. Permethrin, 17. Coumaphos. IS: Triphenyl phosphate (TPP)

Exceptional Recoveries and Precision for 1 and 8 mL Volumes for Agilent SampliQ Dispersive SPE, 2 and 15 mL Kits

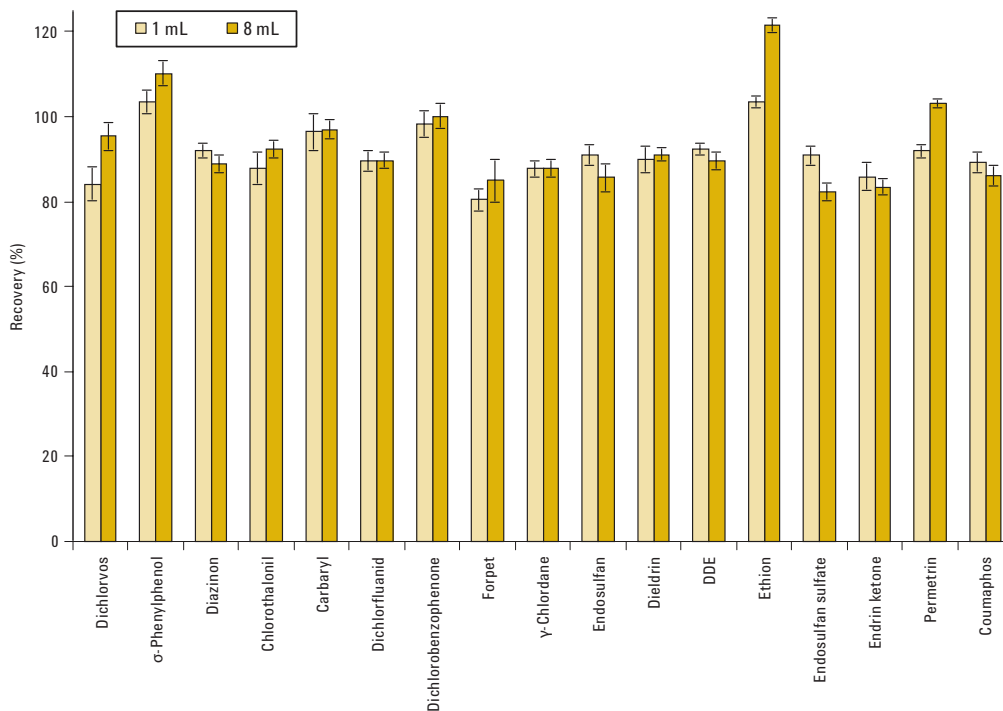


Figure 3. Recoveries and precision for 1 and 8 mL sample volumes employing Agilent SampliQ dispersive SPE, 2 and 15 mL kits, respectively

To review this Application Note in its entirety, please search for 5990-4068EN at www.agilent.com/chem

Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS AOAC Kits by GC/MS (Publication 5990-4305EN)

Introduction

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. 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.

Instrument conditions

GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (Part No. 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 (Part No. 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 GC Column 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (Part No. 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
Source, quad, transfer line temperature	230 °C, 150 °C and 280 °C respectively
Solvent delay	2.30 min
Multiplier voltage	Autotune voltage

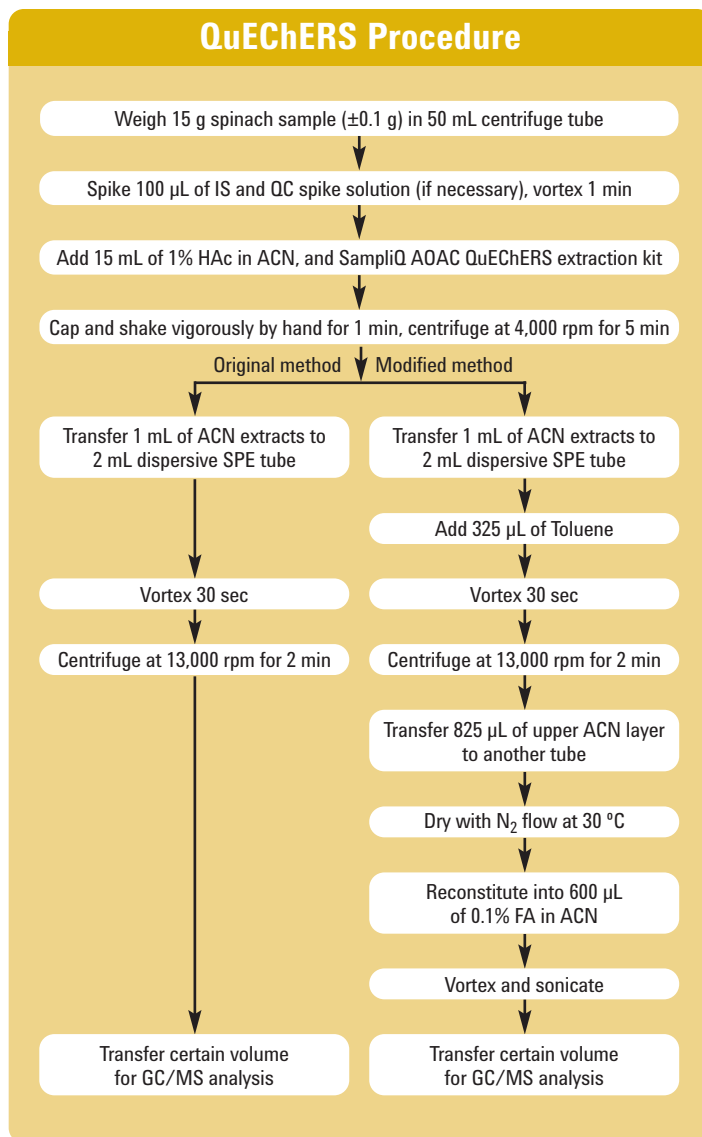


Figure 1. Flow chart of the QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for spinach sample

Results

GC/MS Chromatograms of 50 ng/g Fortified Spinach Samples Implementing the Original and Modified AOAC Dispersive Method

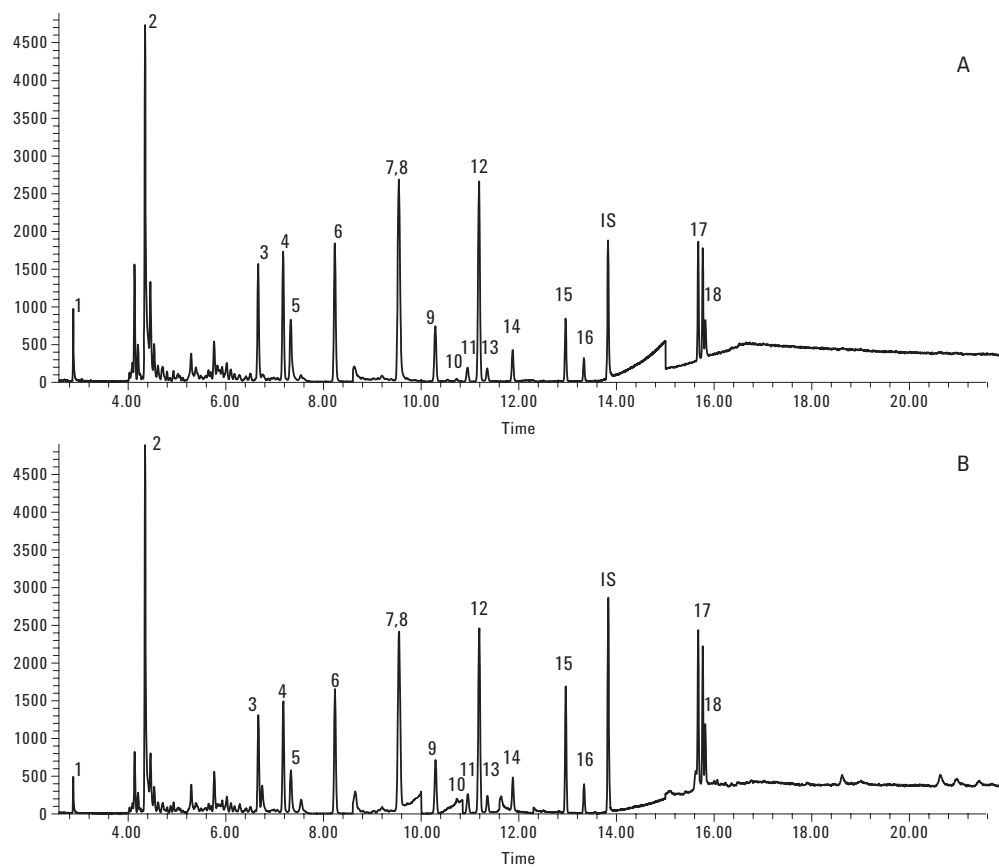


Figure 2. GC/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Diachlorvos, 2. o-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil 6. Chlorpyrifos methyl 7. Dichlorobenzophenone, 8. Chlorpyrifos,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

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
<i>o</i> -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 1. Spinach AOAC dispersive, 1 mL sample volume, 2 mL tube, LC/MS/MS results

Ordering information

Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.
Part No. 5982-5755.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL. Part No. 5982-5222.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 15 mL. Part No. 5982-5258.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 19091S-431UI.

Agilent Ultimate Union. Part No. G3186B.

To review this Application Note in its entirety, please search for 5990-4305EN at www.agilent.com/chem

Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-4248EN)

Introduction

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. 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. 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.

Instrument conditions

HPLC conditions

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column 3.0 mm x 150 mm, 3.5 μ m (Part No. 959963-312)

Flow rate: 0.3 mL/min

Column Temperature: 30 °C

Injection volume: 10 μ L

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: 4,000 V

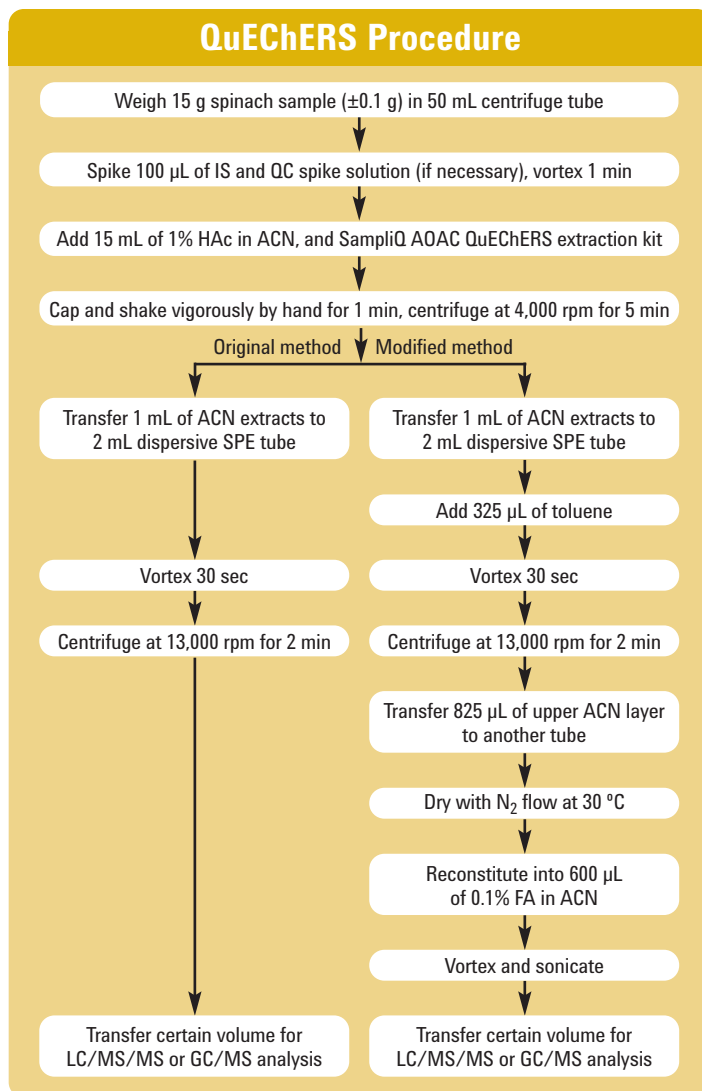


Figure 1. Flow chart of the QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for a spinach sample

Results

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

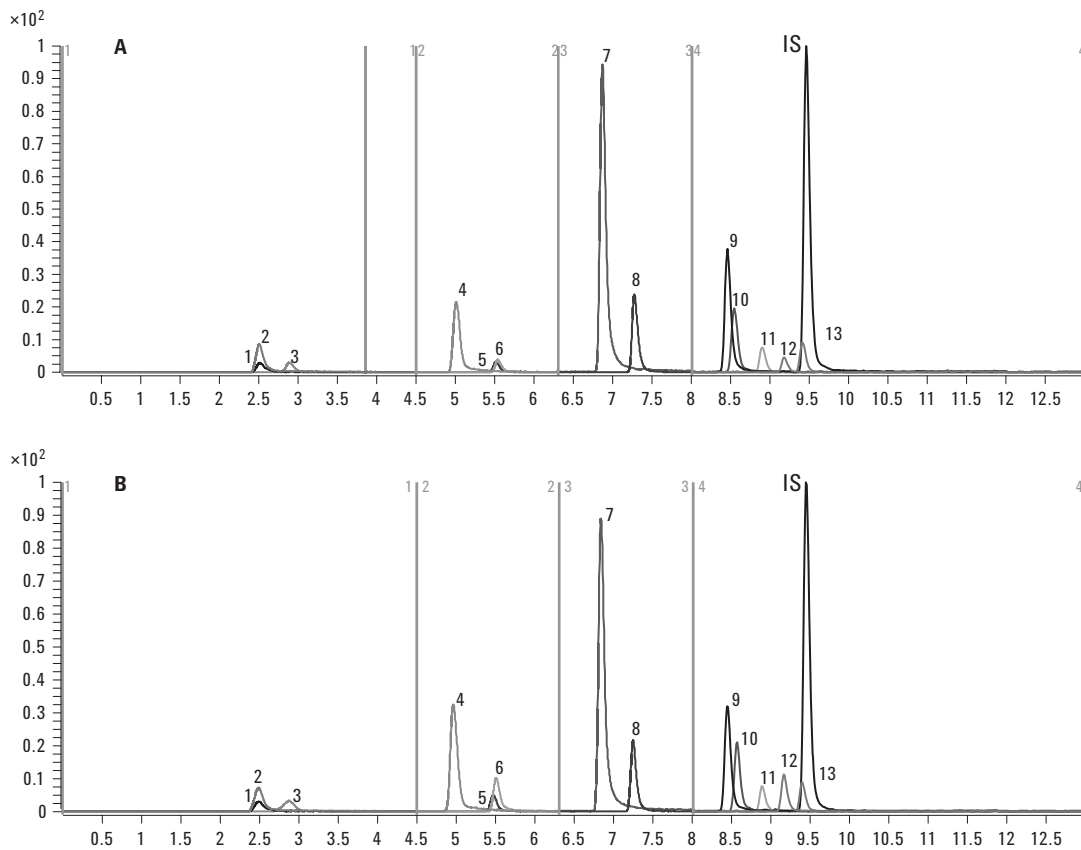


Figure 2. LC/MS/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos,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

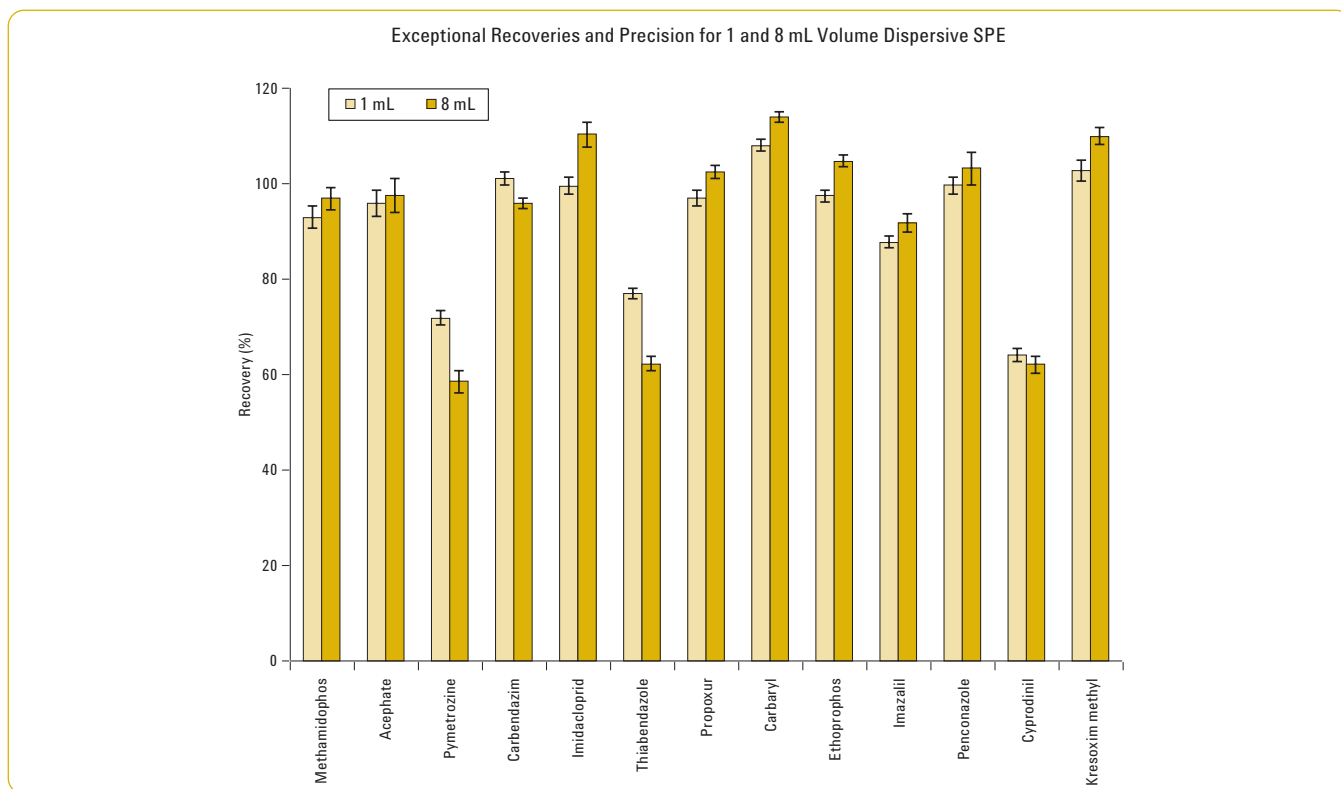


Figure 3. The recovery and precision results for 1 mL dispersive SPE and 8 mL dispersive SPE

Ordering information

Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.
Part No. 5982-5755.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL. Part No. 5982-5222.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 15 mL. Part No. 5982-5258.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 mm x 150 mm, 3.5 μ m.
Part No. 959963-312.

Optimizing Recoveries of Planar Pesticides in Spinach Using Toluene and Agilent SampliQ AOAC QuEChERS Kits with Graphitized Carbon (Publication 5990-4247EN)

Introduction

This application note describes the impact of toluene addition in the dispersive solid phase extraction (SPE) step on the analysis of pesticides in spinach using Agilent SampliQ QuEChERS AOAC kits for highly pigmented fruits and vegetables. With the modified AOAC method, the eight problematic pesticides generated substantially improved recoveries, 50% to 300%, and < 10% RSD.

Instrument conditions

HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312)		
Flow rate:	0.3 mL/min		
Column temperature:	30 °C		
Injection volume:	10 µL		
Mobile phase:	A: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H ₂ O; B: 5 mM ammonium acetate, pH 5.0 in ACN		
Needle wash:	1:1:1 ACN/MeOH/IPA/H ₂ O 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.		

GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (Part No. 5188-5398)
Carrier gas:	Helium
Inlet pressure:	19.6 psi (constant pressure mode) during run 1.0 psi during back flush
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 (Part No. 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 GC Column 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (Part No. G3186B)
Restrictor:	65 cm x 0.15 mm, 0.15 µm DB-5 ms Ultra Inert.
Connections:	Between the Purged Ultimate Union and the MSD.

QuEChERS Procedure

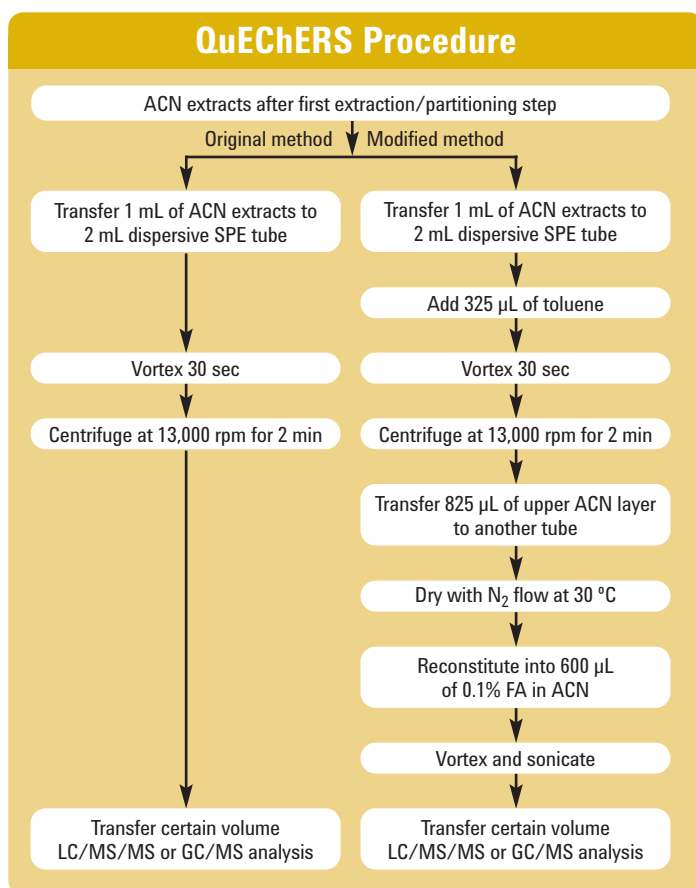


Figure 1. Dispersive SPE procedures of original method (w/o toluene) and modified method (w/toluene)

Ordering information

Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL. Part No. 5982-5222.

Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 15 mL. Part No. 5982-5258.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm x 0.25 µm. Part No. 19091S-431UI.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 15 m x 0.25 mm, 0.25 µm. Part No. 959963-312.

Results

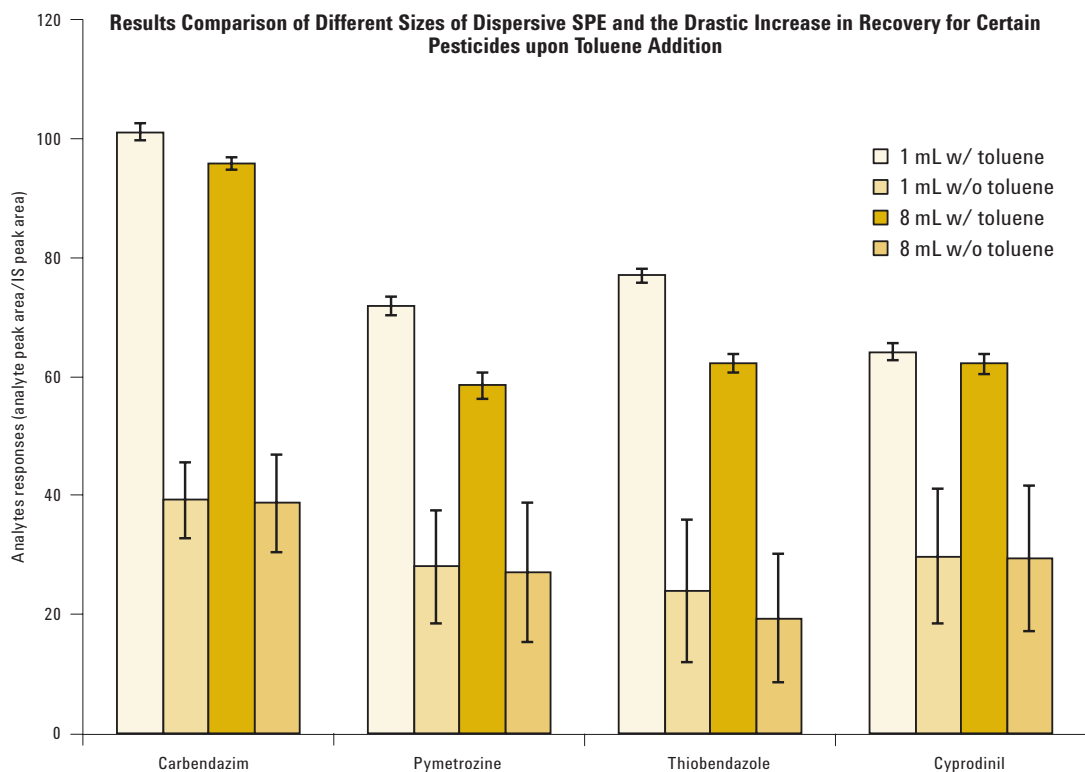


Figure 2. Results comparison of 1 mL and 8 mL dispersive SPE with the modified method (w/ toluene) and the original method (w/o toluene)

Analytes	Original method (w/o toluene)		Modified method (w/ toluene)		Impact with modified method	Detection method
	Recovery	RSD (n=6)	Recovery	RSD (n=6)		
Carbendazim	38.9	14.6	98.5	2.5	Positive	LC/MS/MS
Thiabendazole	21.8	19.7	69.7	2.7	Positive	LC/MS/MS
Pymetrozine	27.6	21.2	65.2	3.7	Positive	LC/MS/MS
Cyprodinil	29.6	23.4	63.1	3.2	Positive	LC/MS/MS
Chlorthalonil	21.1	16.4	47.3	5.9	Positive	GC/MS
Coumaphos	30.1	24.0	87.9	6.1	Positive	GC/MS
Dichlorobenzophenone	53.7	4.5	77.7	6.1	Positive	GC/MS
Folpet	62.0	14.6	88.2	6.3	Positive	GC/MS
Dichlorvos	88.8	6.0	20.4	89.8	Greatly negative	GC/MS
o-Phenylphenol	88.6	4.6	73.7	7.4	Slightly negative	GC/MS
Diazinon	94.9	5.9	81.3	4.0	Slightly negative	GC/MS
Chlordane	103.9	4.5	101.3	4.5	None	GC/MS
Permethrin	81.4	7.2	83.3	5.1	None	GC/MS
Acephate	95.5	5.6	99.8	4.7	None	LC/MS/MS
Carbaryl	108.0	2.5	109.1	1.9	None	LC/MS/MS
Propoxur	97.0	3.19	6.7	2.5	None	LC/MS/MS

Table 1. The impact on certain pesticides by the modified dispersive SPE with addition of toluene

To review this Application Note in its entirety, please search for 5990-4247EN at www.agilent.com/chem

Other Food Methods



Determination of Quinolone Residues in Bovine Liver Using Agilent SampliQ QuEChERS Kit by LC/MS/MS (Publication 5990-4974EN)

Introduction

A method for the determination of 11 Quinolone antibiotics in bovine liver has been established:

- Analytes were extracted and cleaned up from bovine liver with Agilent SampliQ QuEChERS kits
- Extraction was performed using SampliQ EN extraction kits and 5%FA in Acetonitrile
- Clean up was performed using SampliQ dispersive SPE kits Part no. 5982-4921 (25 mg C18 and 150 mg MgSO₄)
- Extracted samples were then analyzed by LC/MS/MS
- Limits of Quantitation (LOQ) were 5.0 ng/g
- Calibration curves were linear over the range of 5.0 to 400 ng/g
- The sample pre-fortified recoveries were between 62.0% and 113.1% with RSD (n=6) values between 2.2% and 13.4%

Instrument conditions

HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 150 mm x 3.0 mm, 3.5 μm (Part No. 959963-312)		
Flow rate:	0.3 ml/min		
Column Temperature:	30° C		
Injection volume:	10 μl		
Mobile phase:	A) 5 mM ammonium acetate in H ₂ O, pH3.0, B) 1:1 methanol/acetonitrile.		
Post Time:	4min		
Gradient:	time (minutes)	% B	Flow Rate (mL/min)
	0	15	0.3
	0.2	15	0.3
	8.0	75	0.3
	9.0	100	0.3
	11.5	Stop	

MS conditions

Polarity:	Positive
Gas Temperature:	325° C
Gas Flow:	8 L/min
Nebulizer:	50 psi
Capillary :	4,000 V

QuEChERS extraction procedure

Weigh 2 g homogenized liver sample (±0.05g) in 50 mL centrifuge tube

Spike 100 μL of IS spike solution, 50 μL of QC spike solution if necessary vortex 30 s

Add 8 mL of 30 mM KH₂PO₄, pH 7.0 buffer, vortex

Add 10 mL of 5% FA in ACN, and shake vigorously for 30 s

Add SampliQ EN QuEChERS extraction kit, and shake vigorously for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer 1 mL of upper ACN layer to SampliQ QuEChERS dispersive SPE 2 mL tube

Vortex 1 min, centrifuge at 13,000 rpm for 3 min with micro-centrifuge

Transfer 800 μL extract to another tube, blow down at 40° C with N₂

Reconstitute into 800 μL 1:9 MeOH/H₂O w/ 0.1% FA, vortex and sonicate

Filter samples w/ 0.22 μm cellulose acetate spin filter

Sample are ready for LC/MS/MS analysis

Figure 1: Agilent's QuEChERS flow chart procedure for antibiotics

Results

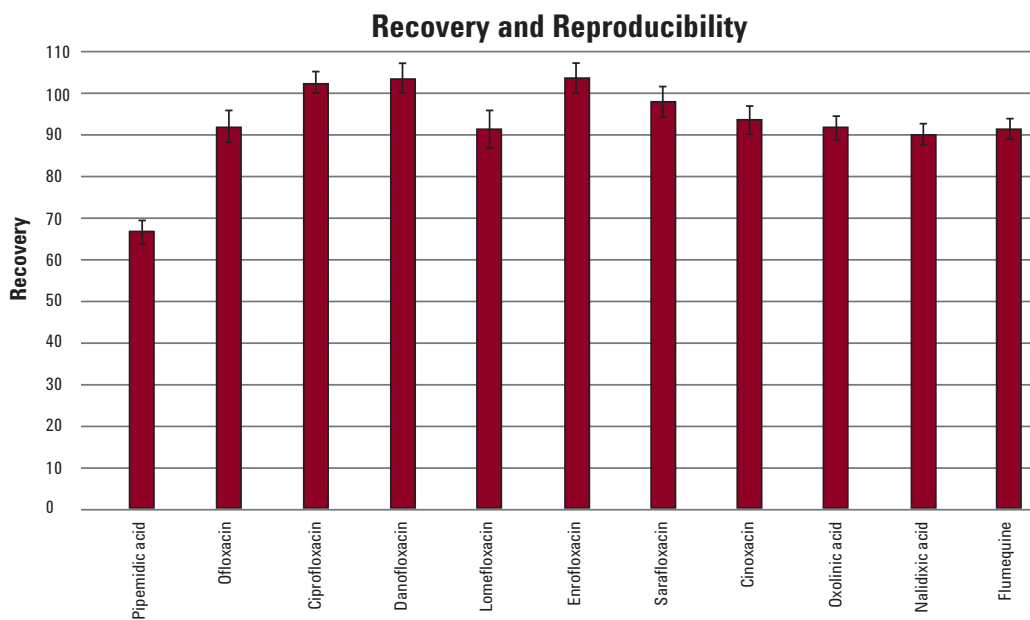


Figure 2: Recovery for 11 quinolone antibiotics in bovine liver

Ordering information

Agilent SampliQ QuEChERS EN Extraction Kit. Part No. 5982-5650.

Agilent SampliQ QuEChERS Dispersive SPE Kit. Part Nos. 5982-4921, 5982-4956.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 150 mm x 3.0 mm, 3.5 μ m. Part No. 959963-312.

Agilent Spin Filters, 0.22 μ m Cellulose Acetate. Part No. 5185-5990.

Determination of Sulfonamide Residues in Bovine Liver Using SampliQ QuEChERS EN Kit by LC/MS/MS (Publication 5990-4975EN)

Introduction

A method for the determination of 9 Sulfonamide antibiotics in bovine liver has been established:

- Analytes were extracted and cleaned up from bovine liver with Agilent SampliQ QuEChERS kits
- Extraction was performed using SampliQ EN extraction kits and 1% AA in Acetonitrile
- Clean up was performed using SampliQ EN fatty dispersive SPE kits, 6 mL (150 mg PSA, 150 mg C18 and 900 mg MgSO₄)
- Extracted samples were then analyzed by LC/MS/MS
- Limits of Quantitation (LOQ) were 2.0 ng/g
- Calibration curves were linear over the range of 2.0 to 400 ng/g
- The sample pre-fortified recoveries were between 53.0% and 92.8% with RSD (n=6) values between 2.1% and 16.8%

Instrument conditions

HPLC conditions

Column:	Agilent ZORBAX Eclipse Rapid Resolution HT Plus C18 LC Column, 50 X 3.0 mm, 1.8 µm (Part No. 959941-302)		
Flow rate:	0.3 ml/min		
Column Temperature:	30° C		
Injection volume:	10 µl		
Mobile phase:	A) 5 mM ammonium acetate in H ₂ O, pH 3.0, B) 1:1 methanol/acetonitrile		
Post Time:	3.5min		
Gradient:	time (minutes)	% B	Flow Rate (mL/min)
	0	15	0.3
	0.2	15	0.3
	6.0	60	0.3
	6.01	100	0.3
	7.0	Stop	

MS conditions

Polarity:	Positive
Gas Temperature:	325° C
Gas Flow:	8 L/min
Nebulizer:	50 psi
Capillary :	4,000 V

QuEChERS extraction procedure

Weigh 2 g homogenized liver sample (±0.05g) in 50 mL centrifuge tube

Spike 50 µL of IS spike solution, 50 µL of QC spike solution if necessary vortex 30 s

Add 8 mL of water, vortex

Add 10 mL of 1% AA in ACN, and shake vigorously for 30 s

Add SampliQ EN QuEChERS extraction kit, and shake vigorously for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer 6 mL of upper ACN layer to SampliQ EN QuEChERS fatty dispersive SPE 2 mL tube

Vortex 2 min, centrifuge at 4,000 rpm for 5 min

Transfer 4 mL extract to another tube, blow down at 40° C with N₂

Reconstitute into 800 µL 1:9 MeOH/H₂O w/ 0.1% FA, vortex and sonicate

Filter samples w/ 0.22 µm cellulose acetate spin filter

Sample are ready for LC/MS/MS analysis

Figure 1: Flow chart for Agilent's QuEChERS procedure

Ordering information

Agilent SampliQ QuEChERS EN Extraction Kit. Part No. 5982-5650.

Agilent SampliQ QuEChERS EN Fatty Dispersive SPE Kit. Part No. 5982-5156.

Agilent ZORBAX Eclipse Rapid Resolution HT Plus C18 LC Column, 50 X 3.0 mm, 1.8 µm. Part No. 959941-302.

Agilent Spin Filters, 0.22 µm Cellulose Acetate. Part No. 5185-5990.

Results

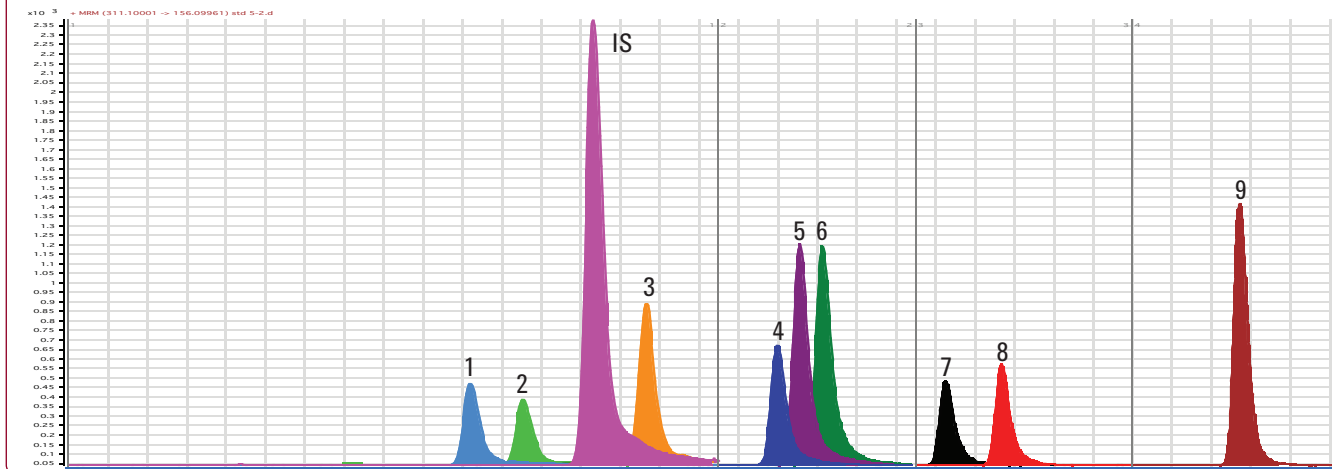


Figure 2. LC/MS/MS Chromatogram of 100 ng/g fortified liver extract. Peaks identification: 1. Sulfadizine, 2. Sulfathiazole, 3. Sulfamerazine, 4. Sulfamethizole, 5. Sulfamethazine, 6. Sulfamethoxyipyridazine, 7. Sulfachloropyridazine, 8. Sulfamethoxazole, 9. Sulfadimethoxin, IS (internal standard)

Compound	Low QC (5 ng/g)		Mid QC (100 ng/g)		High QC (400 ng/g)	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Sulfadizine	73.9	15.6	90.0	13.7	81.9	5.3
Sulfathiazole	62.9	16.8	75.3	8.4	67.9	5.8
Sulfamerazine	77.6	11.5	92.8	6.6	82.0	4.2
Sulfamethizole	62.8	4.7	60.7	6.5	53.0	2.1
Sulfamethazine	87.4	6.9	90.0	10.7	83.4	3.4
Sulfamethoxyipyridazine	81.8	9.4	84.8	8.1	76.4	2.9
Sulfachloropyridazine	84.2	10.0	78.6	6.3	73.8	3.6
Sulfamethoxazole	85.9	7.6	82.3	5.9	78.1	3.3
Sulfadimethoxin	77.8	8.4	80.9	4.9	75.6	3.3

Table 1. Quantitation results – recovery and reproducibility (n=6)

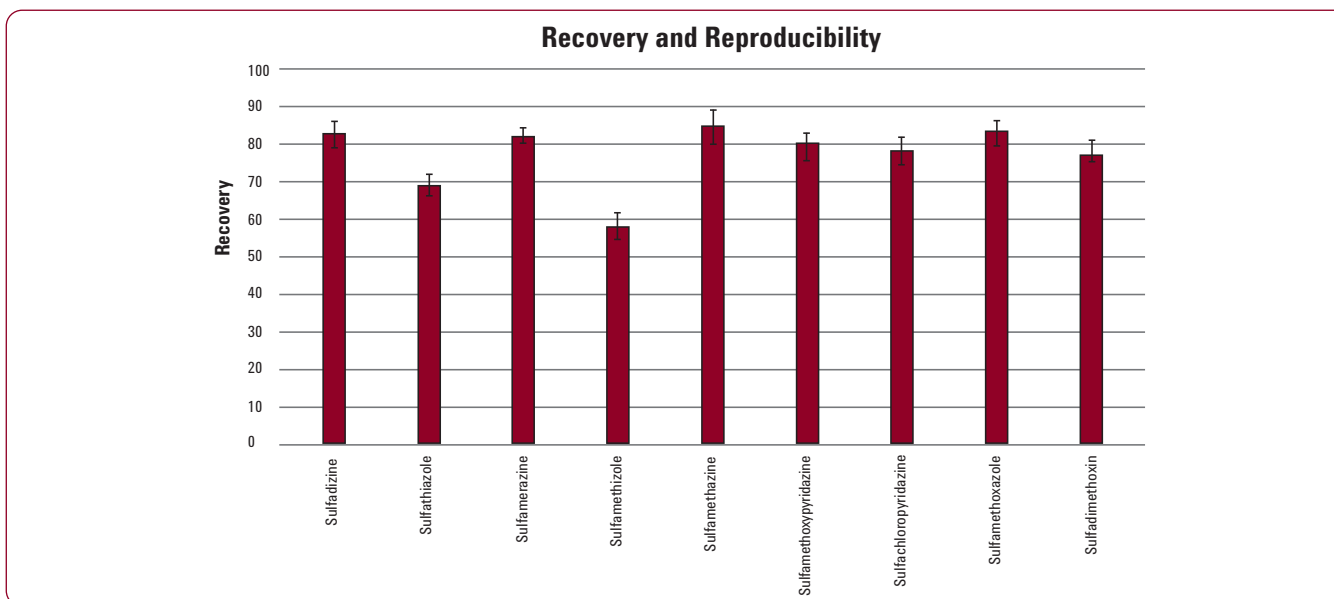


Figure 3. Recovery and reproducibility for 9 sulfonamides in bovine liver

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