

Analysis of Pesticide Residues in Apple Using Agilent SampliQ QuEChERS AOAC Kits by GC/MS

Application Note

Food Safety

Authors

Limian Zhao, David Schultz, and
Joan Stevens
Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19809-1610
USA

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 17 GC-amenable pesticide residues from multiple classes, in apple. 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 utilizing dispersive solid phase extraction (dispersive SPE). The two different dispersive SPE clean-up approaches used either a 1 mL or 8 mL sample volume and were evaluated in parallel after sample extraction. The target pesticides in the apple 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, the pesticide Folpet has an LOQ of 50 ng/g in apple. This application employing SampliQ QuEChERS kits produced results well below the maximum residue limits (MRLs) for all the pesticides screened. The spiked levels for the recovery experiments were 10, 50, and 200 ng/g. Recoveries ranged between 70 and 136% (92.5% on average), with RSD below 15% (5.0% on average).



Agilent Technologies

Introduction

The QuEChERS method for pesticide analysis was first introduced by USDA scientists in 2003. [1] The method was modified to address problematic pesticides by including a buffered extraction system [2]. After a full validation for more than 200 pesticides, this improved method was formalized and adopted as AOAC Official Method 2007.01. [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 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. After mixing and centrifugation, the upper layer is ready for analysis.

Gas Chromatography/Mass Spectrometry (GC/MS) has been widely used in pesticide analysis for many years, because many pesticides are volatile or semi-volatile they are GC-amenable. Previously, we evaluated the performance of a SampliQ AOAC buffered extraction kit and SampliQ AOAC dispersive SPE kits for the analysis of polar pesticides in apple using LC/MS/MS for detection and quantification. [4] In this study, the performance of the SampliQ AOAC Buffered Extraction kit (PN 5982-5755) and SampliQ AOAC dispersive-SPE kits for General Fruits and Vegetables (p/n 5982-5022 and 5982-5058) were 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 non-polar organochlorine pesticides (OCs), certain organophosphorus pesticides (OPs) and organonitrogen pesticides (ONs). The MRLs of these pesticides are a function of both the pesticide class and food matrix and have been set at 10 ng/g or higher. Table 1 shows the chemical and regulatory information for these pesticides in apple.

Experimental

Reagents and Chemicals

All reagents and solvents were HPLC or analytical grade. Acetonitrile (ACN), and methanol (MeOH) were from

Honeywell (Muskegon, MI, USA), and acetic acid (HAc) was 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

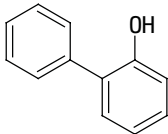
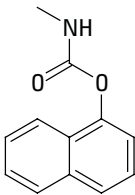
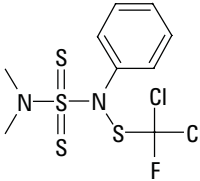
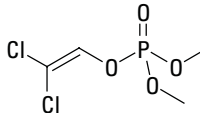
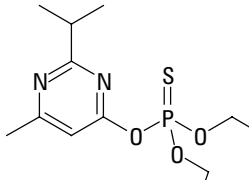
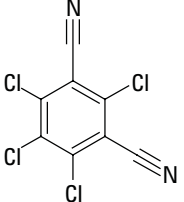
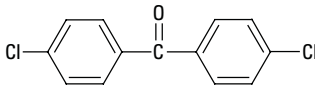
A 1% acetic acid solution in ACN was prepared by adding 10 mL of acetic acid to 1 L of ACN.

Standard and internal standard (IS) stock solutions (2 mg/mL of 11 pesticides) were made in MeOH, respectively, and stored at $-20\text{ }^\circ\text{C}$. A commercially available mix of 6 pesticides, at 20 $\mu\text{g/mL}$ in hexane was used directly. Three QC spiking solutions of 11 pesticides at 1.5, 7.5 and 30 $\mu\text{g/mL}$ were made fresh daily in 1:1 ACN/ H_2O containing 0.1% FA, while the 20 $\mu\text{g/mL}$ of 6 pesticides mix was directly used for QC spike. A 2.5 $\mu\text{g/mL}$ standard solution of 17 pesticides in ACN containing 0.1% FA was used to prepare the calibration curves in the matrix blank extract by appropriate dilution. A 15 $\mu\text{g/mL}$ 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

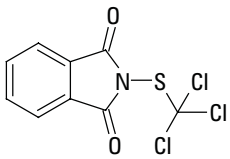
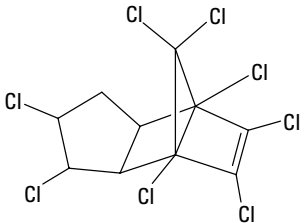
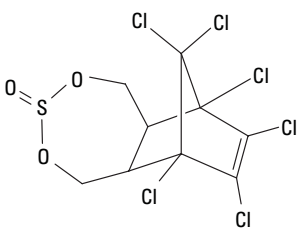
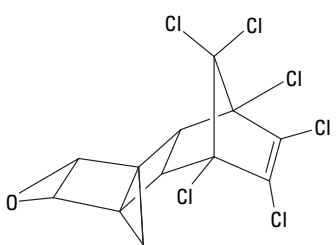
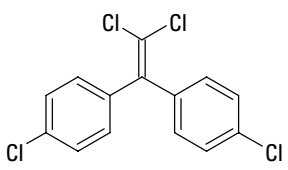
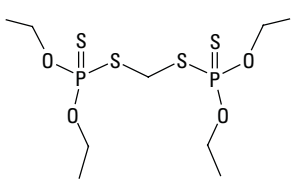
- Agilent Gas Chromatograph (Agilent Technologies Inc., Santa Clara, DE, USA).
- Agilent 5975C Series GC/MSD (Agilent Technologies Inc., Santa Clara, DE, USA).
- Agilent SampliQ QuEChERS AOAC Extraction kits, p/n 5982-5755 (Agilent Technologies Inc., Wilmington, DE, USA).
- Agilent SampliQ QuEChERS AOAC dispersive SPE kits for General Fruits and Vegetables, p/n 5982-5022 and 5982-5058 (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 [5–8]

Name	Category	Log P	pKa	Structure	MRLs in apple (ng/g)*
<i>o</i> -Phenylphenol	Phenol	3.18	9.4		20
Carbaryl	Carbamate	2.36	10.4		50
Dichlofluanid	Sulphamide	3.7	NA		5000
Dichlorvos	Organophosphate	1.9	NA		10
Diazinon	Organophosphate	3.69	2.6		100
Chlorothalonil	Chloronitrile	2.94	NA		10
Dichlorobenzo phenone	Organochlorine	4.44	NA		500

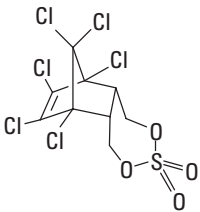
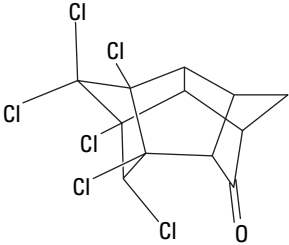
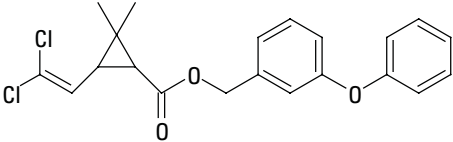
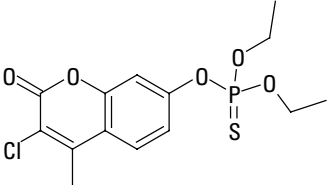
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Table 1. Pesticides Chemical and Regulatory Information [5–8]

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Folpet	Phthalimide	3.02	NA		3000
Chlordane	Cyclodiene organochlorine	2.78	NA		20
Endosulfan	Organochlorine	3.13	NA		50
Dieldrin	Chlorinated hydrocarbon	3.7	NA		10
DDE	Organochlorine	6.55	NA		50
Ethion	Organophosphate	5.07	NA		300

(Continued)

Table 1. Pesticides Chemical and Regulatory Information [5–8]

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Endosulfan sulfate	Organochlorine	3.13	NA		50
Endrin ketone	Organochlorine	4.99	NA		10
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. [9]

GC conditions

Auto-sampler:	Agilent 7683 automatic liquid sampler
Inlet:	Splitless
Column:	Agilent 30 m × 0.25 mm × 0.25 µm HP-5MS Ultra Inert (p/n 19091S-433UI)
Carrier gas:	Helium in the constant pressure mode
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

Sample preparation

Sample comminution

Organically grown, pesticide-free apples were purchased from a local grocery store. Approximately three pounds of apples were chopped into small, bean sized cubes. Skin was included, but the seeds were discarded. The chopped apple cubes were then placed into a clean plastic bag and frozen at –20 °C overnight. The bag was massaged occasionally to make sure the cubes remained separate. The following day, only the required amount of frozen apple cubes was removed and thoroughly blended. Dry ice was added while comminuting, when possible. Samples were comminuted thoroughly to get the best sample homogeneity, ensuring there were no pieces of apple visible in the final sample.

Extraction/Partitioning

A 15 g (± 0.1g) amount of previously homogenized sample was placed into a 50 mL centrifuge tube (from the SampliQ QuEChERS extraction kit). QC samples were fortified with 100 µL of appropriate QC spiking solution (11 pesticides) and 7.5, 37.5, and 150 µL of 20 µg/mL stock solution (6 pesticides mixture), respectively, yielding QC samples with concentra-

Table 2. Instrument Acquisition Data Used for the Analysis of 17 Pesticides by GC/MS.

Analyte	SIM	Collection window (min)	RT (min)
(1) Dichlorvos	184.9	3.0 – 6.5	5.8
(2) σ -Phenylphenol	170.1, 169.1	6.5 – 9.5	8.8
(3) Diazinon	137.1, 179.1	13.5 – 14.65	14.5
(4) Chlorothalonil	265.9, 263.9	14.65 – 16.0	14.8
(5) Carbaryl	144	16.0 – 17.5	16.8
(6) Dichlofluanid	123, 167.1	17.5 – 18.8	18.4
(7) Dichlorobenzophenone	139, 249.9	18.8 – 20.5	19.2
(8) Folpet	259.9, 261.9	21.35 – 21.8	21.6
(9) γ -Chlordane	372.9, 374.9	21.8 – 22.3	22.0
(10) Endosulfan	240.8, 238.8	22.3 – 23.2	22.6
(11) Dieldrin	262.8	23.2 – 25.0	23.9
(12) DDE	245.9, 317.9	23.2 – 25.0	24.0
(13) Ethion	230.9	25.0 – 26.4	26.0
(14) Endosulfan sulfate	273.8	26.4 – 27.2	26.8
TPP (IS)	325.1, 326.1	27.2 – 28.0	27.7
(15) Endrin ketone	316.9	28.0 – 28.5	28.2
(16) Permethrin	183.1	30.0 – 32.5	31.4, 31.6
(17) Coumaphos	362.0	30.0 – 32.5	31.7

tions of 10, 50 and 200 ng/g. A 100 μL amount of internal standard spiking solution (15 $\mu\text{g}/\text{mL}$ of TPP) was added to all samples except the control blank to yield a 100 ng/g concentration in each sample. Tubes were capped and vortexed for 1 min. A 15 mL amount of 1% HAc in ACN was added to each tube using the dispenser. An Agilent SampliQ QuEChERS extraction salt packet from the kit (PN 5982-5755) containing 6 g of anhydrous MgSO_4 , and 1.5 g of anhydrous NaOAc was added directly to the tubes. The salt bag was massaged carefully to break up any salt clumps before pouring. The tubes were examined to ensure that no powder was left in the threads or rims of the tubes. Sample tubes were sealed tightly and shaken vigorously for 1 min by hand to ensure that the solvent interacted with the entire sample and crystalline agglomerates were dispersed. Sample tubes were centrifuged at 4000 rpm for 5 min.

Dispersive SPE Cleanup

A 1 mL aliquot of the upper ACN layer was transferred to an Agilent SampliQ QuEChERS dispersive SPE 2 mL tube (p/n 5982-5022). An 8 mL aliquot was transferred to an Agilent SampliQ QuEChERS dispersive SPE 15 mL tube (p/n 5982-5058). The 2 mL tube contained 50 mg of PSA and 150 mg of anhydrous MgSO_4 ; while the 15 mL tube contained 400 mg of PSA and 1200 mg of anhydrous MgSO_4 . The tubes were tightly capped and vortexed for 1 min. The 2 mL tubes were centrifuged with a micro-centrifuge at 13,000 rpm for 2 min, and 15 mL tubes in a standard centrifuge at 4000 rpm for 5 min. An aliquot from the extract, 500 μL was transferred into an autosampler vial, and analyzed by GC/MS.

Figure 1 shows the flow chart for the QuEChERS AOAC sample extraction procedure.

Results and Discussion

Using the SampliQ QuEChERS kits, the entire procedure is faster, easier, offers time and labor savings, while ensuring consistency. An analyst can process 40–50 samples in just a few hours. The addition of a food sample with a high content of water directly to the salts creates an exothermic reaction, which can affect analyte recoveries, especially for volatile pesticides. Agilent's SampliQ extraction salts are uniquely prepared in an anhydrous package. The unique SampliQ anhydrous salts packet allows addition after adding organic solvent to the sample, as specified in the original QuEChERS method.

In our previous study, the new design of SampliQ QuEChERS AOAC kits demonstrated excellent recovery and precision for a broad variety of semi-polar to polar pesticides using

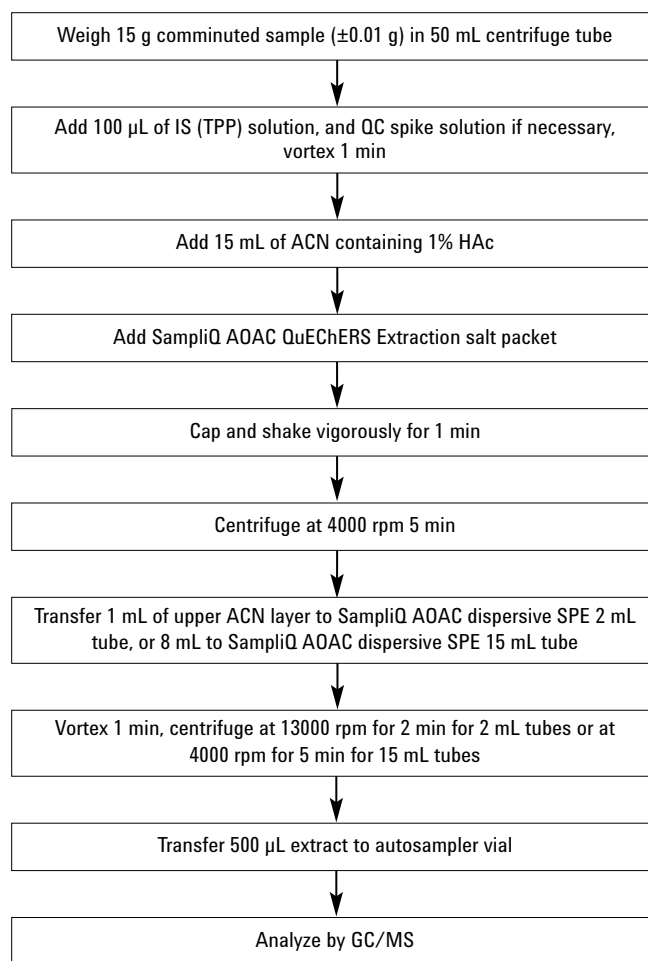


Figure 1. Flow chart of the Agilent SampliQ QuEChERS AOAC extraction procedure.

LC/MS/MS. [4] There are many semi-volatile and volatile pesticides, so the use of GC/MS is applicable for the performance evaluation of the AOAC kits for the analysis of these groups of pesticides. The selectivity of GC/MS (SIM mode) is not as effective as that of LC/MS/MS (MRM mode). Furthermore, the final QuEChERS samples still contained food matrix impurities, which can be observed in the GC/MS chromatogram of blank apple extract. Therefore, it is important to carefully choose the monitored ions of each compound when setting up the SIM method. In general, the most abundant ions were selected in order to achieve the best sensitivity; however in a few instances the sensitivity was compromised to obtain better selectivity by using more unique but less abundant ions. As shown in Figure 2a, there are interference peaks apparent in the blank chromatogram; fortunately most pesticides are free of co-eluting interferences. There was an interference eluting at a retention time very close to that of σ -phenylphenol, and can not be differentiated for quantitation.

The response of this interferent within the blank was integrated to be less than 20% response of *o*-phenylphenol peak at the LOQ (10 ng/g) sample. Therefore, the selectivity was considered acceptable for this compound. Figure 2 (a, b) shows the chromatograms of a blank apple extract and 50 ng/g fortified apple extract.

Linearity and Limit of Quantification (LOQ)

The linear calibration range for all of the pesticides was 0–400 ng/g; excluding Folpet at 50–400 ng/g due to poor sensitivity. Two different dispersive SPE volumes (1 mL and 8 mL) were used for evaluation and comparison; therefore, two calibration curves were generated from matrix blanks prepared

from each size. Each calibration curve was made at levels of 10, 20, 50, 100, 250, and 400 ng/g. The TPP was the internal standard (IS) at 100 ng/g in all cases. 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). Table 1 shows that the 10 ng/g quantification limits LOQ (10 ppb) and 50 ng/g LOQ for Folpet (50 ppb) established for pesticides are substantially lower than many MRLs for the pesticides in fruit and vegetables. The regression fit used for the calibration curves was the average response factor. Table 3 shows the linear term and RF relative standard deviation (%) for both 1 mL and 8 mL dispersive SPE.

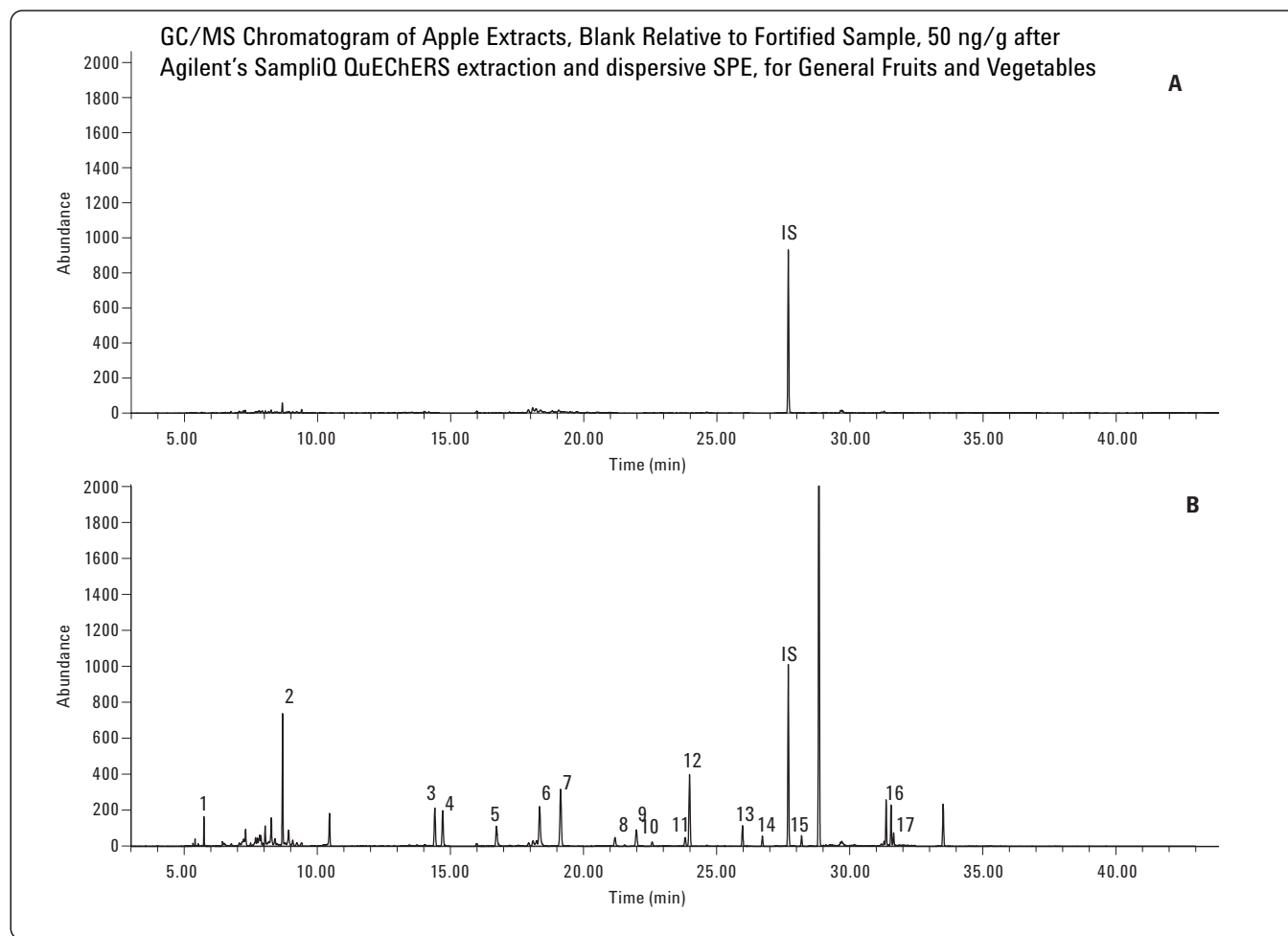


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50-ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2. *O*-Phenylphenol, 3. Diazinon, 4. Chlorothalonil, 5. Carbaryl, 6. Dichlofluanid, 7. Dichlorobenzophenone, 8. Folpet, 9. γ -Chlordane, 10. Endosulfan, 11.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

Table 3. Linearity of 17 Pesticides in Apple Extract

Analytes	1 mL dispersive SPE		8 mL dispersive SPE	
	Linear Term	RF Rel Std Dev (%)	Linear Term	RF Rel Std Dev (%)
Dichlorvos	3.47e-001	11.4	3.87e-001	4.6
σ -Phenylphenol	1.37e-000	10.7	1.50e-000	11.4
Diazinon	7.04e-001	10.9	7.39e-001	6.5
Chlorothalonil	6.84e-001	13.7	8.02e-001	8.9
Carbaryl	8.07e-001	14.1	1.01e-000	10.8
Dichlofluanid	1.04e-000	12.8	1.08e-000	8.6
Dichlorobenzophenone	4.55e-001	11.4	4.60e-001	8.2
Folpet	3.88e-002	19.5	4.52e-002	20.1
γ -Chlordane	3.23e-001	10.4	3.31e-001	9.2
Endosulfan	8.56e-002	15.2	8.26e-002	8.8
Dieldrin	2.71e-001	6.2	2.59e-001	5.9
DDE	1.43e-000	8.4	1.39e-000	7.5
Ethion	5.87e-001	19.7	5.63e-001	17.0
Endosulfan sulfate	2.72e-001	9.6	2.74e-001	9.5
Endrin ketone	2.75e-001	10.1	2.75e-001	7.8
Permethrin	9.71e-001	9.4	9.29e-001	8.0
Coumaphos	2.70e-001	15.6	2.72e-001	15.7

Recovery and Reproducibility

The recovery and reproducibility were evaluated by spiking pesticides standards in comminuted apple 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 volume dispersive SPE are shown in Table 4 and Table 5, respectively. It can be seen from the results that all of the pesticides give excellent recoveries (average of 90.8% for 1 mL and 94.2% for 8 mL) and precision (average of 5.7% RSD for 1 mL and 4.3% RSD for 8 mL). As mentioned above, an

interferent was eluted very closely with σ -phenylphenol. The selectivity was still acceptable because the interferent contributed less than 20% of LOQ; however, the contribution of the interference peak resulted in the higher recovery of this compound at low levels. Folpet is a notoriously unstable pesticide, and the main problems dealing with degradation and instability come from the N-trihalomethylthio functional group. [3, 10] Folpet was quantified, but the LOQ was found to be 50 ng/g due to poor sensitivity, however, recovery and reproducibility at 50 ng/g and above were acceptable (average recovery 85.5%, average reproducibility 10%).

Table 4. Recovery and Repeatability of Pesticides in Fortified Apple With Agilent SampliQ 2 mL Dispersive SPE Tube (p/n 5982-5022); Recovery 90.8%, RSD 5.7% (avg)

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)
Dichlorvos	86.8	7.0	83.9	11.6	81.5	5.5
<i>o</i> -Phenylphenol	113.4	6.3	96.3	6.5	100.5	3.6
Diazinon	98.6	2.3	87.3	2.8	90.4	4.9
Chlorothalonil	86.1	10.0	84.4	5.3	93.2	7.6
Carbaryl	96.1	9.0	93.8	8.3	99.1	8.2
Dichlofluanid	90.0	7.0	84.6	2.9	94.6	5.0
Dichlorobenzo phenone	97.8	7.6	95.0	6.2	102.2	4.3
Folpet	–	–	74.4	9.1	95.7	11.0
γ -Chlordane	79.6	4.4	88.9	4.3	95.3	4.4
Endosulfan	69.8	9.2	91.2	5.3	96.2	5.2
Dieldrin	90.6	10.9	86.6	3.2	92.8	4.8
DDE	84.0	4.8	89.4	3.8	95.4	4.5
Ethion	90.9	1.8	103.5	1.4	116.5	5.0
Endosulfan sulfate	79.8	1.9	80.4	4.6	86.8	5.6
Endrin ketone	85.2	12.0	80.7	3.6	91.8	4.5
Permethrin	87.9	2.8	93.8	2.0	94.0	4.4
Coumaphos	87.8	5.1	89.7	3.0	90.0	6.4

Table 5. Recovery and Repeatability of Pesticides in Fortified Apple With Agilent SampliQ 15 mL Dispersive SPE Tube (p/n 5982-5058); Recovery 94.2%, RSD 4.3% (avg)

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)
Dichlorvos	103.4	4.2	85.6	8.1	97.2	7.2
σ-Phenylphenol	125.8	8.7	99.2	4.4	105.4	5.0
Diazinon	96.0	4.5	82.3	2.1	88.4	6.3
Chlorothalonil	96.5	3.0	82.8	5.2	97.7	4.5
Carbaryl	97.7	3.9	91.4	4.4	101.9	5.0
Dichlofluanid	91.7	5.1	83.7	1.0	93.7	5.1
Dichlorobenzo phenone	98.8	9.3	96.2	4.7	105.3	4.3
Folpet	–	–	88.4	4.0	72.5	6.0
γ-Chlordane	80.9	3.5	87.5	3.3	94.8	5.0
Endosulfan	80.3	7.3	84.1	3.6	98.6	3.0
Dieldrin	81.2	3.4	93.1	2.0	98.7	3.9
DDE	86.1	1.8	92.4	3.4	98.9	3.9
Ethion	106.5	3.6	122.2	2.0	136.3	4.2
Endosulfan sulfate	91.6	4.6	87.7	4.0	93.0	4.1
Endrin ketone	76.2	3.3	82.4	3.9	91.8	4.1
Permethrin	97.9	1.6	104.7	1.1	106.6	4.2
Coumaphos	82.3	6.7	86.5	2.5	89.3	5.1

Figure 3 shows the recovery and precision results for 1 mL dispersive SPE and 8 mL dispersive SPE. The two different dispersive SPE clean-ups were performed by transferring 1 mL or 8 mL of ACN extract from the same sample following the extraction step. In order to simplify the comparison, the average recovery and precision of three fortification concentrations were used for all pesticides. The results of each dispersive SPE clean-up appeared to be independent of volume used. Both approaches provided efficient and similar sample clean-up, and thus generated relatively equivalent results.

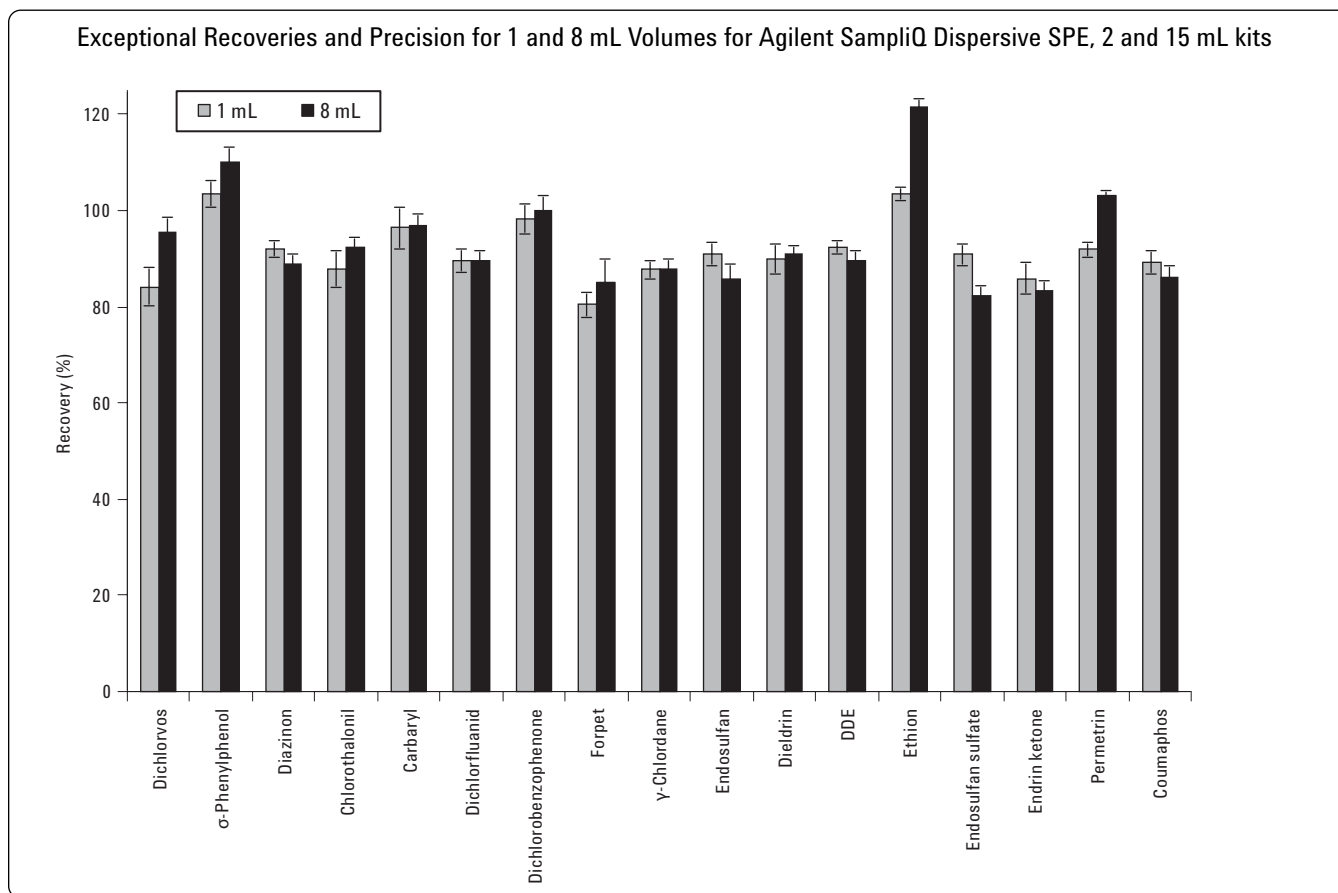


Figure 3. Recoveries and precision for 1 and 8 mL sample volumes employing Agilent SampliQ Dispersive SPE, 2 and 15 mL kits, respectively.

Conclusions

Agilent SampliQ QuEChERS AOAC method for General Fruits and Vegetables: Extraction and Dispersive SPE kits provided a simple, fast and effective method for the purification and enrichment of representative volatile to semi-volatile pesticides in apple. The recovery and reproducibility, based on matrix spiked standards, were acceptable for multiclass, multi-residue pesticide determination in apple. The impurities and matrix effects from apple did not interfere with the quantitation of target compounds. The LOQs of the pesticides were lower than regulated MRLs in apple. As the selected pesticides

represented a broad variety of different classes and properties, the Agilent SampliQ QuEChERS AOAC Extraction and Dispersive SPE kits for General Fruits and Vegetables is an excellent choice for other pesticides in similar food matrices.

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