

Analysis of Pesticide Residues in Apples using Agilent SampliQ QuEChERS AOAC Kit by LC/MS/MS Detection

Application Note

Food Safety

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Abstract

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS), Association of Analytical Communities (AOAC) Official Method 2007.01; sample preparation approach for extraction and cleanup of 16 pesticide residues in apple. The 16 pesticides chosen represent various classes of interest. The method employed involves initial extraction in a buffered aqueous/acetonitrile system, an extraction/partitioning step after the addition of salt, and then a cleanup step utilizing dispersive solid phase extraction (dispersive SPE). The two different dispersive SPE clean-up approaches (1 mL and 8 mL) were evaluated simultaneously after sample extraction. The target pesticides in the apple extracts were then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode. The method was validated in terms of recovery and reproducibility. 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).



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Introduction

Multi-residue analysis of pesticides in fruits, vegetables, and other foods is the primary function of many regulatory, industrial, and contract laboratories throughout the world. Because of the wide variety of pesticides and complexity of food matrices, the sample must be initially cleaned up using a sample preparation technique prior to analysis. Without question, the most efficient approach to pesticide analysis involves the use of multiclass, multi-residue methods. Once the preliminary analytical quality requirements, including accuracy, precision, sensitivity, selectivity and dynamic range, have been met to suit the needs of a particular analysis, other considerations should be evaluated. These additional considerations include sample throughput, ruggedness, ease of use, cost of materials and labor, toxic solvent usage, and waste generation.

The QuEChERS method was introduced first by USDA scientists in 2003. [1] The method was then modified to address some problematic pesticides by using 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 salting out water from the sample using anhydrous magnesium sulfate (MgSO_4) to induce liquid-liquid partitioning. After removing an aliquot from the organic layer, for further cleanup a dispersive solid phase extraction (dispersive SPE) is conducted using a combination of primary secondary amine (PSA) to remove fatty acids from 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.

In this study, 16 pesticides were used for evaluating the performance of the Agilent AOAC Buffered Extraction kit (p/n 5982-5755) and SampliQ QuEChERS AOAC dispersive SPE kit for General Fruits and Vegetables (p/n 5982-5022 and 5982-5058), suitable for common fruit and vegetable applications. Apple was selected as the fruit matrix for the evaluation. Most of the pesticides are from the original "representative pesticides" list [2]. According to their experience, a method working well for these representative pesticides should work equally well for nearly all of the other pesticides

that are routinely monitored in multiclass, multi-residue methods. These pesticides are from 9 different pesticide classes, including acidic, basic, neutral, base-sensitive and acid-labile pesticides. Furthermore, the selected pesticides are suitable for LC/MS/MS analysis. 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 multiple class pesticides in apple.

Experimental

Reagents and Chemicals

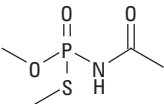
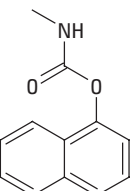
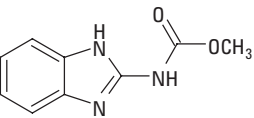
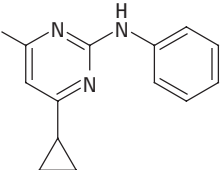
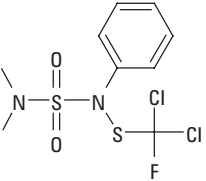
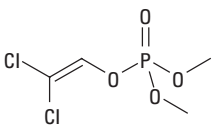
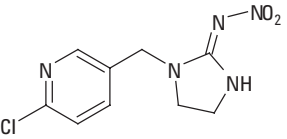
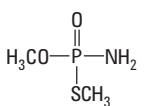
All reagents and solvents were HPLC or analytical grade. Acetonitrile (ACN), methanol (MeOH) were from Honeywell (Muskegon, MI, USA). Dimethyl sulfoxide (DMSO) and acetic acid (HAc) were from Sigma-Aldrich (St Louis, MO, USA). Ammonium acetate (NH_4OAc) was from Fisher Chemicals (Fair Lawn, NJ, USA).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

Solutions and Standards

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

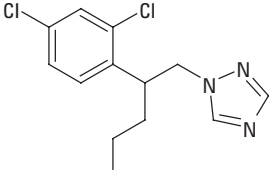
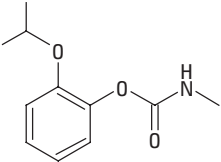
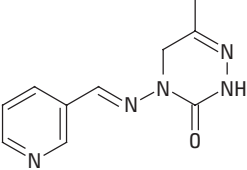
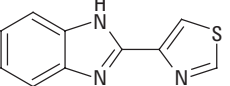
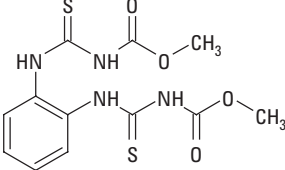
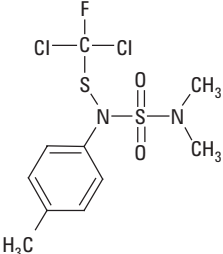
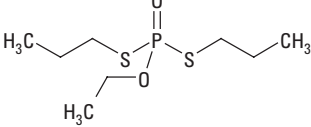
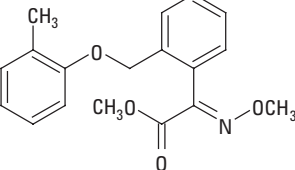
Standard and internal standard (IS) stock solutions (2.0 mg/mL for all, except 0.5 mg/mL for carbendazim) were made in MeOH, 0.1% FA in ACN, or DMSO, respectively, and stored at -20 °C. Three QC spiking solutions of 1.5, 7.5, and 30 $\mu\text{g}/\text{mL}$ were made fresh daily in 1:1 ACN/ H_2O (0.1% FA).

Table 1. Pesticides Chemical and Regulatory Information [4–6]

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Acephate	Organophosphate	-0.89	8.35		20
Carbaryl	Carbamate	2.36	10.4		50
Carbendazim	Benzimidazole	1.48	4.2		100
Cyprodinil	Anilinopyrimidine	4	4.44		50
Dichlofluanid	Sulphamide	3.7	NA		5000
Dichlorvos	Organophosphate	1.9	NA		10
Imidacloprid	Neonicotinoid	0.57	NA		500
Methamidophos	Organophosphate	-0.79	NA		10

(Continued)

Table 1. Hormones Used in this Study

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Penconazole	Triazole	3.72	1.51		50
Propoxur	Carbamate	0.14	NA		1000
Pymetrozine	Pyridine	-0.19	4.06		20
Thiabendazole	Benzimidazole	2.39	4.73 12.00		50
Thiophanate-methyl	Benzimidazole	1.45	7.28		100
Tolylfluanid	Sulphamide	3.9	NA		3000
Ethoprophos	Organophosphate	2.99	NA		5
Kresoxim-methyl	Strobilurin	3.4	NA		50

*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.

A 10 µg/mL standard solution in 1:1 ACN/H₂O (0.1% FA) was made for preparation of calibration curves in the matrix blank extract by appropriate dilution. A 15 µg/mL of TPP in 1:1 ACN/H₂O (0.1% FA) was used as an internal standard (IS).

Equipment and Material

- Agilent 1200 HPLC with Diode Array Detector (Agilent Technologies Inc., Santa Clara, CA, USA).
- Agilent 6410 Triple Quadrupole LC/MS/MS system with Electrospray Ionization (Agilent Technologies Inc., Santa Clara, CA, USA).
- Agilent SampliQ Buffered QuEChERS AOAC Extraction kit, p/n 5982-5755, and SampliQ QuEChERS AOAC Dispersive SPE kit 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. Plainfield, NJ, USA)
- Eppendorf microcentrifuge (Brinkmann Instruments, Westbury, NY, USA)
- Grinder (St. Joseph, MI, USA)

Instrument Condition

HPLC conditions

Column:	Agilent Eclipse Phenyl-Hexyl 150 mm x 3.0 mm, 3.5 µm (p/n 959963-312)		
Flow rate:	0.3 mL/min		
Column Temperature:	30 °C		
Injection volume:	10 µL		
Mobile Phase:	A: 5mM NH ₄ OAc, pH 5.0 in 20:80 MeOH/H ₂ O B: 5 mM NH ₄ OAc, pH 5.0 in ACN		
Needle wash:	1:1:1:1 ACN/MeOH/IPA/H ₂ O (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:	4000 V

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

Table 2. Instrument Acquisition Data Used for the Analysis of 16 Pesticides by LC/MS/MS

Analyte	MRM channels (<i>m/z</i>)	Fragmentor (V)	CE (V)	RT (min)
Acephate	1) 184.0 > 94.9	60	3	2.55
	2) 184.0 > 111.0		15	
Methamidophos	1) 142.0 > 94.0	60	8	2.54
	2) 142.0 > 124.9		8	
Pymetrozine	1) 218.1 > 105.0	115	20	2.97
	2) 218.1 > 78.0		50	
Carbendazim	1) 192.1 > 160.0	95	18	5.07
	2) 192.1 > 105.0		40	
Dichlorvos	1) 221.0 > 109.0	110	13	6.57
	2) 221.0 > 95.0		40	
Thiophanate methyl	1) 343.1 > 151.0	105	17	7.08
	2) 343.1 > 117.9		65	
Propoxur	1) 210.1 > 111.0	50	12	6.89
	2) 210.1 > 92.9		15	
Carbaryl	1) 202.0 > 145.0	50	3	7.30
	2) 202.0 > 115.0		40	
Cyprodinil	1) 226.1 > 93.0	120	35	9.23
	2) 226.1 > 108.0		35	
Dichlorfluanid	1) 333.0 > 123.0	85	28	9.40
	2) 333.0 > 223.9		5	
Ethoprophos	1) 243.1 > 130.9	80	15	8.50
	2) 243.1 > 172.9		15	
Penconazole	1) 284.1 > 158.9	80	32	8.95
	2) 284.1 > 172.9		32	
Tolyfluanid	1) 347.0 > 136.9	60	25	9.73
	2) 347.0 > 238.0		3	
Thiabendazole	1) 202.1 > 175.0	110	27	5.65
	2) 202.1 > 131.0		38	
Imidacloprid	1) 256.1 > 209.1	60	12	5.53
	2) 256.1 > 175.0		18	
TPP	1) 327.1 > 77.0	70	45	9.49
	2) 327.1 > 151.9		45	
Kresoxim methyl	1) 314.0 > 222.1	70	10	9.44
	2) 314.0 > 235.0		10	

1) Quantifier transition channel

2) Qualifier transition channel

Sample preparation

Sample comminution

In order to get the most reliable statistical results, it is important to spend the necessary effort and time on conducting proper sampling and homogenization procedures. 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 pit was discarded. The chopped apple cubes were put into a clean plastic bag and frozen at $-20\text{ }^{\circ}\text{C}$ overnight. The bag was massaged occasionally to make sure the cubes were frozen loosely, to avoid clumping. The following day, a portion of

frozen apple cubes were removed and thoroughly blended. Certain precautions were exercised while blending the sample. First, the chopped apple cubes remained in the freezer until the point of blending. Only the portion of apple cubes necessary for homogenizing were removed; the rest were kept in the freezer until the next comminution. Dry ice was added, when possible, while comminuting to keep the temperature low. Second, the blender container was kept dry to prevent clumping. In between blending, the container was rinsed and dried. Third, samples were comminuted thoroughly to obtain the best sample homogeneity. No pieces of apple were visible in the final sample.

Extraction/Partitioning

A 15 g (± 0.05 g) previously homogenized sample was placed into a 50 mL centrifuge tube from the SampliQ QuEChERS Extraction kit. QC samples were fortified with 100 μ L of appropriate QC spiking solution yielding QC samples with concentrations of 10, 50, and 200 ng/g. One hundred microliters of IS spiking solution (15 μ g/mL of TPP) were 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. Fifteen milliliters of 1% HAC in ACN were added to each tube using the dispenser. To each tube, an Agilent AOAC Buffered Extraction packet from the kit (p/n 5982-5755) containing 6 g of anhydrous $MgSO_4$ and 1.5 g of anhydrous NaOAc, was added directly to the tubes. No powders were left in the threads or rims of the tubes. Tubes were sealed tightly and shaken vigorously for 1 min by hand to ensure that the solvent interacted well with the entire sample and crystalline agglomerates were broken up. Sample tubes were centrifuged at 4000 rpm for 5 min.

Dispersive SPE Cleanup

A 1 mL aliquot of the upper ACN layer was transferred into a SampliQ QuEChERS AOAC 2 mL dispersive SPE tube (p/n 5982-5022) or 8 mL aliquot were transferred into an SampliQ QuEChERS AOAC 15 mL dispersive SPE 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 13000 rpm for 2 min, and the 15 mL tubes were centrifuged in a standard centrifuge at 4000 rpm for 5 min. Two hundred microliters of extract were transferred into an autosampler vial. Then 800 μ L of water or another appropriate standard solution (prepared in water) were added. The samples were capped and vortexed thoroughly. The samples were then ready for LC/MS/MS analysis.

The flow chart in Figure 1 illustrates the sample preparation procedure.

Results and Discussion

In addition to being fast, easy, cheap, effective, rugged and safe, an additional key feature of the QuEChERS method is the potential for the simultaneous analysis of multi-pesticide residues. With the new design of SampliQ QuEChERS kits, the whole procedure is even faster, easier, and offers more time and labor savings, while ensuring consistency. An analyst can process 40–50 samples in just a few hours. Adding a food

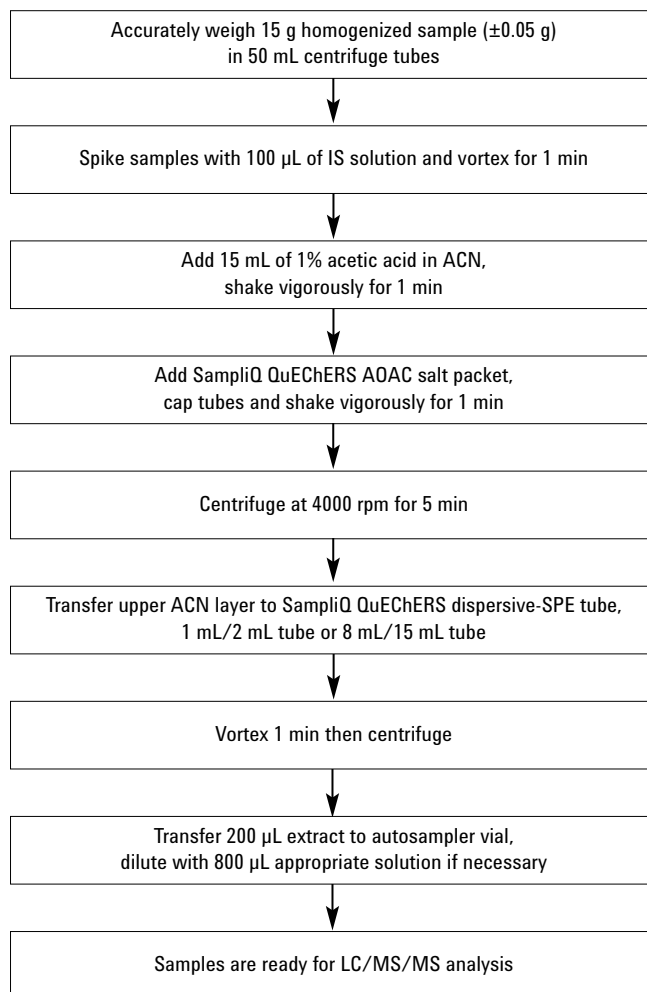


Figure 1. QuEChERS AOAC sample preparation procedures flow chart.

sample with a high percentage of water directly to the salts may create an exothermic reaction that can affect analyte recovery. Agilent's SampliQ salts and buffers are uniquely prepared in anhydrous packages. This allows addition **AFTER** adding solvent to the sample, as specified in the QuEChERS methodology. The final QuEChERS sample may contain food matrix impurities because it is a very simple sample extraction and cleanup procedure. The final apple extract appeared light green. But with the powerful selectivity of LC/MS/MS multiple reaction monitoring (MRM) mode, the extracted apple blank appeared to be clean and free of impurities, indicating the blank apple extract did not contribute any interferences with the target compounds. Figure 2 shows the chromatograms of a blank apple extract and a 10 ng/g fortified apple extract.

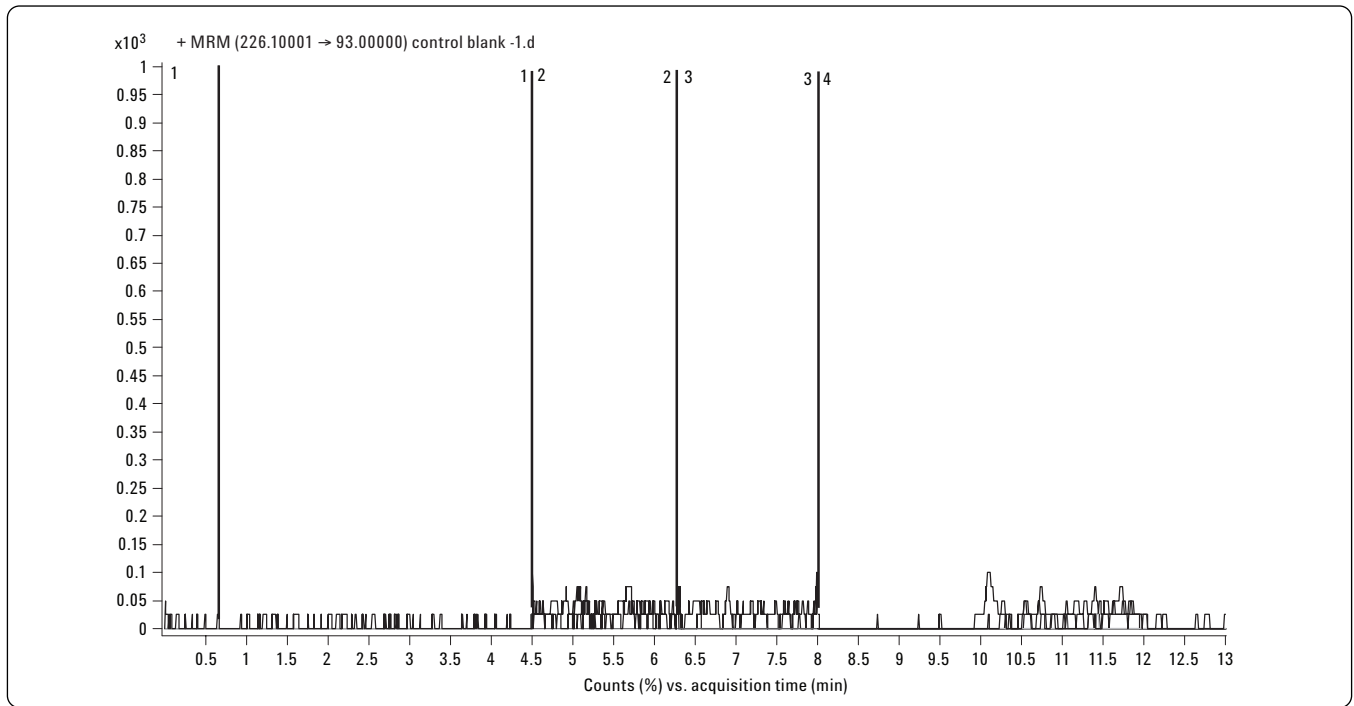


Figure 2a Chromatograms of apple extract blank. No interference was found in the blank.

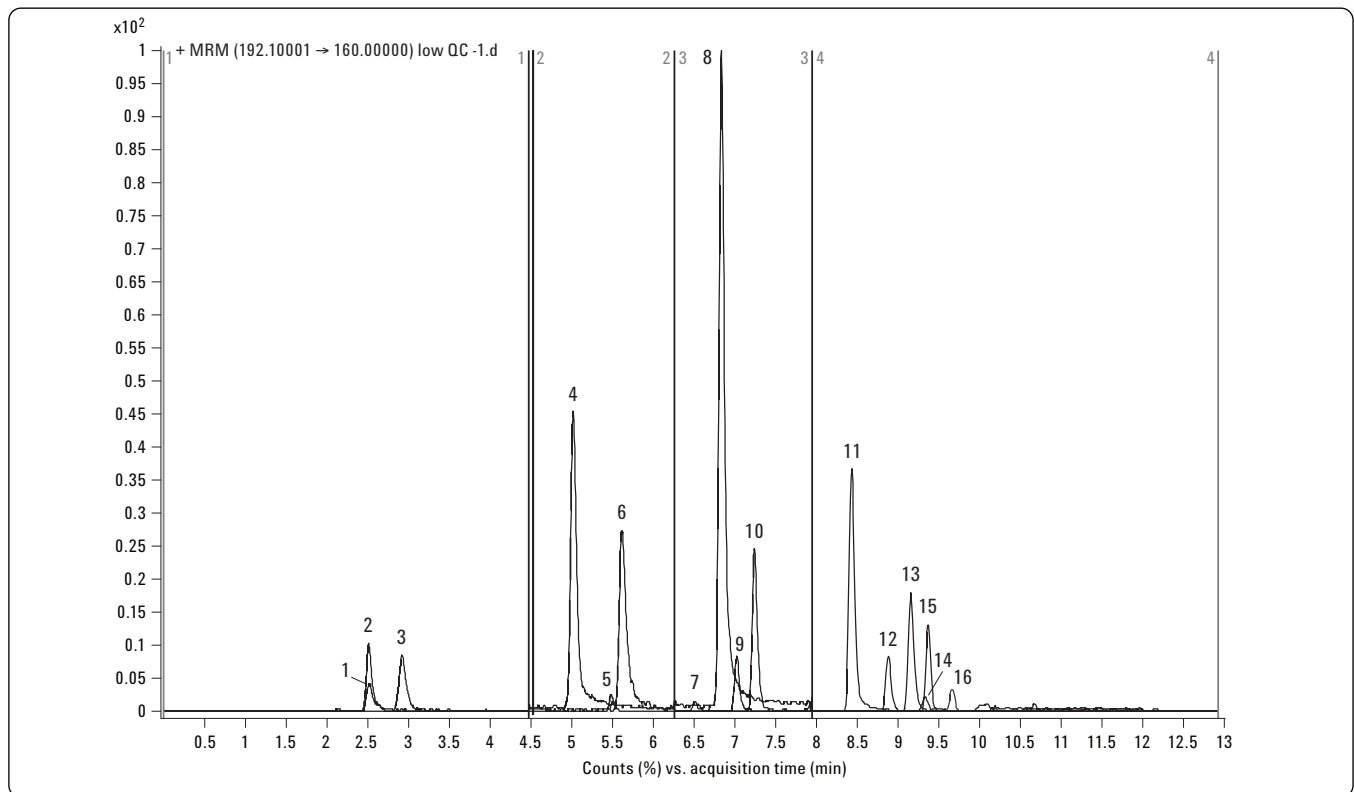


Figure 2b. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlofluanid,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

Linearity and Limit of Quantification (LOQ)

The linear calibration range for all the pesticides was 5 – 250 ng/g. Since two different dispersive SPE volumes (1 mL and 8 mL) were used for evaluation and comparison, two sets of calibration curves were generated respectively. Matrix blanks were prepared for each size. Calibration curves, spiked in matrix blanks, were made at levels of 5, 10, 50, 100, 200, and 250 ng/g. The TPP (IS) was used at 100 ng/g level.

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 5 ng/g quantification limits LOQ (5 ppb) established for all of the pesticides is lower than the MRLs of these pesticides in fruit and vegetables. Table 3 shows the regression equation and correlation coefficient (R^2) for both 1 mL and 8 mL dispersive SPE volumes.

Table 3. Linearity of Pesticides in Apple Extract

Analytes	1 mL dispersive SPE Regression equation	R^2	8 mL dispersive SPE Regression equation	R^2
Methamidophos	$Y = 0.2349X - 0.0013$	0.9949	$Y = 0.2300X - 0.0007$	0.9981
Acephate	$Y = 0.1118X - 0.0012$	0.9881	$Y = 0.1094X - 0.0014$	0.9980
Pymetrozine	$Y = 0.2671X - 0.0016$	0.9950	$Y = 0.2290X - 0.0014$	0.9975
Carbendazim	$Y = 0.9441X + 0.0063$	0.9895	$Y = 0.8583X + 0.0006$	0.9968
Imidacloprid	$Y = 0.0513X - 0.0009$	0.9905	$Y = 0.0500X - 0.0007$	0.9933
Thiabendazole	$Y = 0.7049X + 0.0044$	0.9868	$Y = 0.6198X + 0.0043$	0.9961
Dichlorvos	$Y = 0.0265X + 0.0001$	0.9884	$Y = 0.0247X + 0.0006$	0.9439
Propoxur	$Y = 2.0348X - 0.0091$	0.9951	$Y = 2.0264X - 0.0090$	0.9965
Thiophanate methyl	$Y = 0.2024X - 0.0054$	0.9307	$Y = 0.5090X - 0.0041$	0.9682
Carbaryl	$Y = 0.4984X - 0.0002$	0.9965	$Y = 0.4889X - 0.0029$	0.9976
Ethoprophos	$Y = 0.8203X - 0.0064$	0.9952	$Y = 0.8536X - 0.0076$	0.9971
Penconazole	$Y = 0.1775X - 0.0006$	0.9903	$Y = 0.1783X - 0.0019$	0.9848
Cyprodinil	$Y = 0.3529X - 0.0023$	0.9960	$Y = 0.3528X - 0.0022$	0.9958
Dichlorfluand	$Y = 0.0453X - 0.0004$	0.9869	$Y = 0.0460X - 0.0006$	0.9954
Kresoxim methyl	$Y = 0.2498X - 0.0024$	0.9932	$Y = 0.2490X - 0.0013$	0.9927
Tolyfluand	$Y = 0.0718X - 0.0016$	0.9823	$Y = 0.0755X - 0.0006$	0.9788

Recovery and Reproducibility

The recovery and reproducibility were evaluated by spiking pesticide standards in homogeneous apple samples 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 (RSD) data of 1 mL and 8 mL dispersive SPE sample volumes are shown in Tables 4 and 5, respective-

ly. It can be seen from the results that all of the pesticides give acceptable recoveries (average of 97.5% for 1 mL and 93.3% for 8 mL) and precision (average of 4.5% RSD for 1 mL and 4.1% RSD for 8 mL). The notoriously base-sensitive pesticides such as dichlorfluanid and tolyfluanid showed excellent recovery and precision. Acid labile pesticide, pymetrozine, also showed acceptable recovery and precision.

Table 4. Recovery and Repeatability of Pesticides in Fortified Apple With 2 mL Dispersive SPE Tube (p/n 5982-5022)

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	83.6	5.6	81.3	2.6	83.4	1.4
Acephate	106.8	5.8	95.6	2.3	97.3	2.0
Pymetrozine	78.3	11.4	76.6	11.6	108.1	5.3
Carbendazim	101.0	6.5	98.5	4.3	91.0	2.6
Imidacloprid	107.0	6.5	97.6	3.4	107.4	3.0
Thiabendazole	106.2	6.6	103.7	2.6	95.5	2.0
Dichlorvos	78.2	11.4	94.2	7.2	95.8	1.8
Propoxur	106.3	0.8	105.7	1.2	101.2	1.6
Thiophanate methyl	79.0	15.4	76.7	15.4	102.2	8.1
Carbaryl	93.4	1.9	98.4	2.2	97.5	1.1
Ethoprophos	95.8	4.5	96.1	1.8	94.7	1.3
Penconazole	117.0	4.8	111.9	2.3	111.0	1.6
Cyprodinil	106.9	4.0	102.0	2.8	102.4	1.8
Dichlorfluanid	92.5	6.5	96.3	2.2	99.4	2.6
Kresoxim methyl	98.2	9.3	101.9	2.7	104.1	1.8
Tolyfluanid	96.6	9.5	105.1	1.8	102.2	1.7

Table 5. Recovery and Repeatability of Pesticides in Fortified Apple With 15 mL Dispersive SPE Tube (p/n 5982-5058)

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	80.6	9.3	79.4	2.9	83.1	2.5
Acephate	94.6	7.0	93.7	3.4	95.1	2.5
Pymetrozine	88.8	12.1	87.7	10.1	118.4	5.5
Carbendazim	85.9	3.9	90.4	2.7	85.5	2.2
Imidacloprid	101.8	3.5	99.3	3.7	106.0	0.9
Thiabendazole	92.5	6.4	92.2	2.6	89.5	1.5
Dichlorvos	73.7	14.8	91.8	7.3	95.5	2.0
Propoxur	96.2	1.6	98.2	0.6	97.2	1.2
Thiophanate methyl	81.4	4.9	78.2	13.4	102.3	5.8
Carbaryl	86.5	2.6	90.3	1.4	91.1	1.2
Ethoprophos	89.6	2.9	92.1	1.0	94.1	1.1
Penconazole	102.1	2.5	106.0	3.0	111.0	1.6
Cyprodinil	93.9	3.7	97.4	0.9	99.7	2.0
Dichlorfluanid	81.7	8.7	96.9	5.6	98.1	2.6
Kresoxim methyl	91.8	5.8	93.9	2.0	98.3	1.2
Tolyfluanid	94.1	7.9	95.2	4.0	97.5	2.6

Figure 3 shows the recovery and precision results for 1 mL and 8 mL dispersive SPE. The two different dispersive SPE clean-ups were performed by using 1 mL or 8 mL of ACN extract from the same sample tube after 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 two dispersive SPE clean-up approaches appeared to be independent of volume used. Both approaches provided efficient sample clean-up, and generated relatively equivalent results.

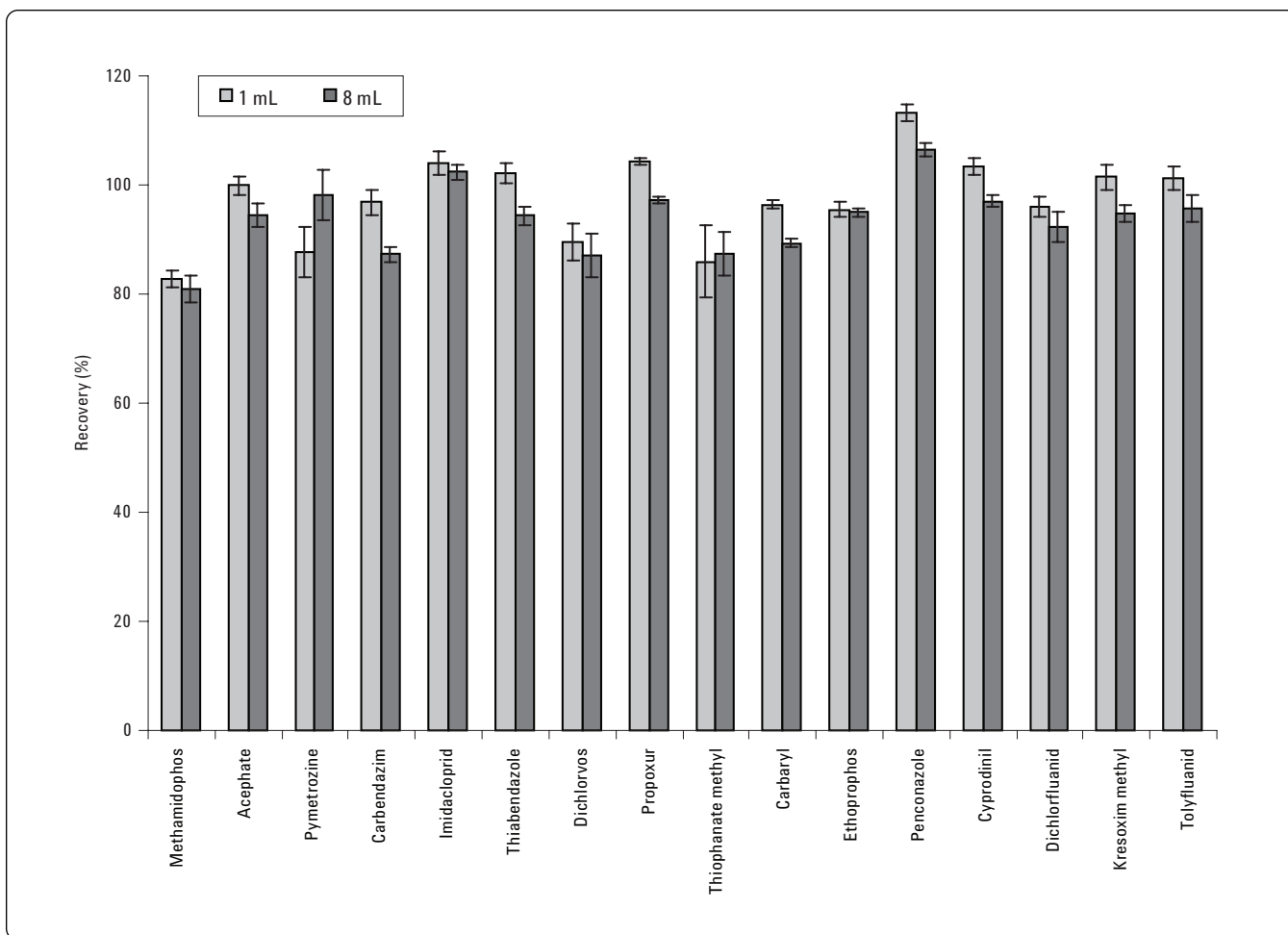


Figure 3. Results comparison of 1 mL and 8 mL dispersive SPE sample volume.

Conclusions

Agilent SampliQ AOAC Buffered Extraction kit and SampliQ AOAC dispersive SPE kit for General Fruits and Vegetables provided a simple, fast, and effective method for the purification of representative 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 were minimal and did not interfere with the quantitation of any target compound. The LOQs of the pesticides were significantly lower than their 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 kit for General Fruits and Vegetables can be used for other pesticides in similar fruit matrices.

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