Analysis of Pesticide Residues in Apple using Agilent SampliQ QuEChERS European Standard EN Kits by LC/MS/MS Detection

Application Note

Food Safety

Authors

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Abstract

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 method employed involves initial extraction in an 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 6 mL sample volume) are evaluated simultaneously after sample extraction. 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. The method is validated in terms of recovery and reproducibility. The 5 ng/g of limits of quantitation (LOQ) for pesticides in apple established in this application is well below their regulatory maximum residue limits (MRLs). The spiking levels for the recovery experiments are 10, 50, and 200 ng/g. Excluding pymetrozine, recoveries of the pesticides ranged between 73 and 111% (87% on average), and RSDs below 20% (5.8% on average).

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Introduction

Multi-residue analysis of pesticides in fruits, vegetables, and other foods is a 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 compatible sample preparation technique before injection into the detection system. It is unquestionable that the most efficient approach to pesticide analysis involves the use of multiclass, multi-residue methods. Once the preliminary analytical quality requirements of accuracy, precision, sensitivity, selectivity, and dynamic scope have been met to suit the need for a particular analysis, other practice considerations should be evaluated. These additional considerations include high sample throughput, ruggedness, ease of use, low cost, labor, minimal 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] There is also a European variation, the prEn method 15662: 2007 [3], [4]. In summary, the method uses acetonitrile extraction, followed by the salting out of water from the sample using anhydrous magnesium sulfate ($MgSO_4$), NaCl and buffering citrate salts to induce liquid-liquid partitioning. For 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.

The EN methodology is similar in principal to the AOAC method, but has several differences. First, the extraction buffered system in the EN method uses sodium chloride, sodium citrate dehydrate, and disodium citrate hydrogenate sesquihidrate instead of sodium acetate in the extraction step. Second, in the dispersive SPE step, the EN method uses 25 mg PSA per mL of extract rather than 50 mg PSA per mL of extract used by the AOAC method.

In this study, 16 pesticides are used to demonstrate the performance of Agilent SampliQ QuEChERS EN Buffered Extraction kit (p/n 5982-5650) and EN dispersive SPE kit (p/n 5982-5021 and 5982-5056) for General Fruits and Vegetables, suitable for common fruit and vegetable applications. Most of the pesticides are from the original 'representative pesticides' list [1]. 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 nine 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 have been set for 10 ng/g or higher. Table 1 shows the chemical and regulatory information of these pesticides.

Experimental

Reagents and Chemicals

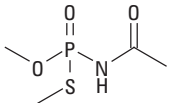
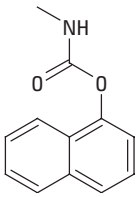
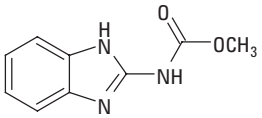
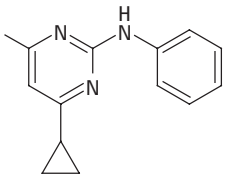
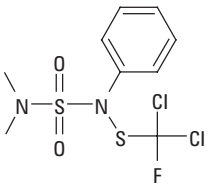
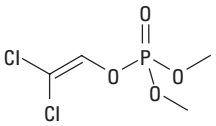
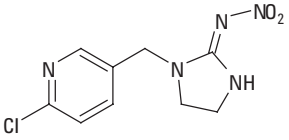
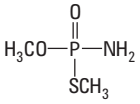
All reagents and solvents were HPLC or analytical grade. Acetonitrile (ACN), methanol (MeOH) were from Honeywell (Muskegon, MI, USA). Dimethyl sulfoxide (DMSO) was from Sigma-Aldrich (St Louis, MO, USA). Ammonium acetate (NH_4OAc) was from Fisher Chemicals (Fair Lawn, NJ, USA). Formic acid (FA) was from 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 1M NH_4OAc pH 5 stock solution was made by dissolving 19.27 g NH_4OAc powder in 250 mL Milli-Q water. The pH was adjusted to 5 with HAc and monitored with a pH meter. The solution was stored at 4 °C. 20:80 MeOH/H_2O 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.

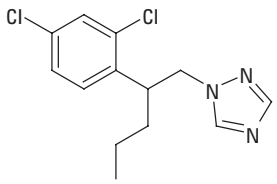
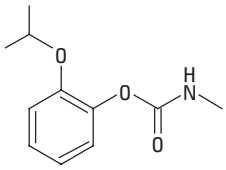
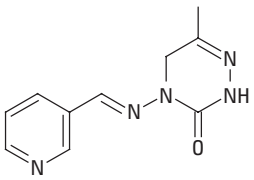
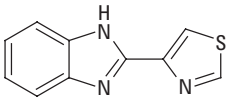
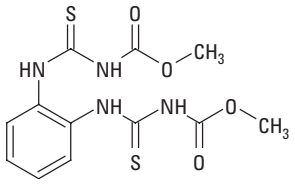
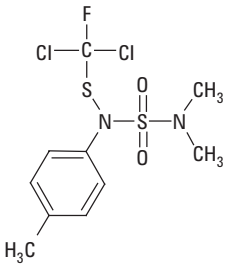
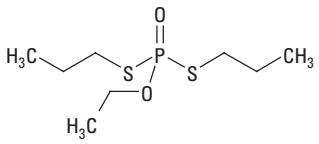
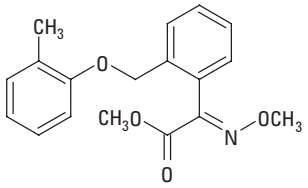
A 5 mM NH_4OAc in ACN solution was prepared by adding 5 mL of 1M NH_4OAc pH 5 stock solution to 1 L ACN, mixing well and sonicating 5 min. A 1% FA in ACN solution was prepared by adding 1 mL of FA to 100 mL of ACN.

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

Name	Class	Log P	pKa	Structure	MRLs in apple (ng/g)*
Acephate	Organophosphate	–0.89	8.35		20
Carbaryl	Carbamate	2.36	10.4		50
Carbendazim	Benzimidazole	1.48	4.2		100
Cyprodinil	Anilinopyrimidine	4	4.44		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. Pesticides Chemical and Regulatory Information [5–7]

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.

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, and 20 µg/mL were made fresh daily in 1:1 ACN/H_2O with 0.1% FA. A 10 µg/mL standard spiking solution in 1:1 ACN/H_2O with 0.1% FA was made for preparation of calibration curves in the matrix blank extract by appropriate dilution. A 10 µg/mL IS spiking standard of TPP was made in 1:1 ACN/H_2O (0.1% FA).

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 QuEChERS extraction kit, p/n 5982-5650, and dispersive SPE tubes, p/n 5982-5021 and 5982-5056 (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: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H2O B: 5 mM ammonium acetate, pH 5.0 in ACN
Needle wash:	1:1:1:1 ACN/MeOH/IPA/H_2O (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 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 (m/z)	Fragmentor (V)	CE (V)	RT (min)
Acephate	1) 184.0 > 94.9 2) 184.0 > 111.0	60	3 15	2.55
Methamidophos	1) 142.0 > 94.0 2) 142.0 > 124.9	60	8 8	2.54
Pymetrozine	1) 218.1 > 105.0 2) 218.1 > 78.0	115	20 50	2.97
Carbendazim	1) 192.1 > 160.0 2) 192.1 > 105.0	95	18 40	5.07
Dichlorvos	1) 221.0 > 109.0 2) 221.0 > 95.0	110	13 40	6.57
Thiophanate methyl	1) 343.1 > 151.0 2) 343.1 > 117.9	105	17 65	7.08
Propoxur	1) 210.1 > 111.0 2) 210.1 > 92.9	50	12 15	6.89
Carbaryl	1) 202.0 > 145.0 2) 202.0 > 115.0	50	3 40	7.30
Cyprodinil	1) 226.1 > 93.0 2) 226.1 > 108.0	120	35 35	9.23
Dichlorfluanid	1) 333.0 > 123.0 2) 333.0 > 223.9	85	28 5	9.40
Ethoprophos	1) 243.1 > 130.9 2) 243.1 > 172.9	80	15 15	8.50
Penconazole	1) 284.1 > 158.9 2) 284.1 > 172.9	80	32 32	8.95
Tolyfluanid	1) 347.0 > 136.9 2) 347.0 > 238.0	60	25 3	9.73
Thiabendazole	1) 202.1 > 175.0 2) 202.1 > 131.0	110	27 38	5.65
Imidacloprid	1) 256.1 > 209.1 2) 256.1 > 175.0	60	12 18	5.53
TPP	1) 327.1 > 77.0 2) 327.1 > 151.9	70	45 45	9.49
Kresoxim methyl	1) 314.0 > 222.1 2) 314.0 > 235.0	70	10 10	9.44

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 the pit was discarded. Then, the chopped apple cubes were put into a clean plastic bag and frozen at –20 °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 was kept in the freezer until the next comminution. Dry ice was added 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 get the best sample homogeneity. There were not any pieces of apple visible in the final sample.

Extraction/Partitioning

A 10 g (±0.05g) previously homogenized sample was placed into a 50 mL centrifuge tube. QC samples were fortified with 100 µL of appropriate QC spiking solution. A 100 µL of IS spiking solution (10 µg/mL of TPP) were added to all of samples except the control blank to yield a 100 ng/g concentration in sample. Tubes were capped and vortexed for 1 min. Ten milliliters of ACN was added to each tube using the dispenser. Tubes were then capped and shaken by hand for 1 min. To each tube, an Agilent SampliQ QuEChERS EN extraction salt packet (p/n 5982-5650), containing 4 g anhydrous $MgSO_4$, 1g NaCl, 1g sodium citrate, and 0.5 g disodium citrate sesquihydrate, was added directly. 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 sufficiently. Sample pH was checked with pH paper, and 5N NaOH was added to adjust the pH to 5-5.5. 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 an Agilent SampliQ QuEChERS EN dispersive SPE 2 mL tube (p/n 5982-5021); or 6 mL of aliquot was transferred into an Agilent SampliQ QuEChERS EN dispersive SPE 15 mL tube (p/n 5982-5056). The 2 mL tube contained 25 mg of PSA and 150 mg of anhydrous $MgSO_4$; while the 15 mL tube contained 150 mg of PSA and 900 mg of anhydrous $MgSO_4$. The tubes were capped tightly and vortexed for 1 min. The 2 mL tubes were centrifuged with a micro-centrifuge at 13,000 rpm for 2 min, and the 15 mL tubes with a standard centrifuge at 4000 rpm for 5 min. Ten microlitres of extract were transferred into an autosampler vial. A 10 µL of a 1% FA in ACN solution was added immediately, in addition to 800 µL of water or appropriate standard solution (prepared in water). The samples were capped and vortexed thoroughly, to prepare for LC/MS/MS analysis.

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

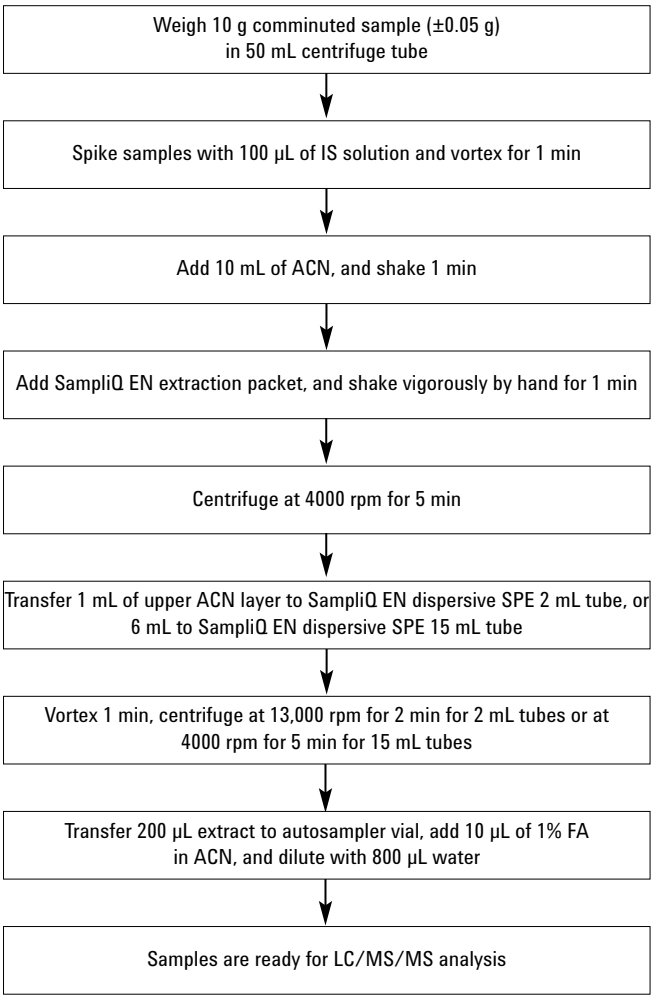


Figure 1. QuEChERS EN sample preparation procedures flow chart.

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 Agilent's 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 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 methology. The final QuEChERS sample still contains 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 mode, the extracted apple blank appeared to be clean and free of coeluting impurities, indicating that the cleaned-up 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.

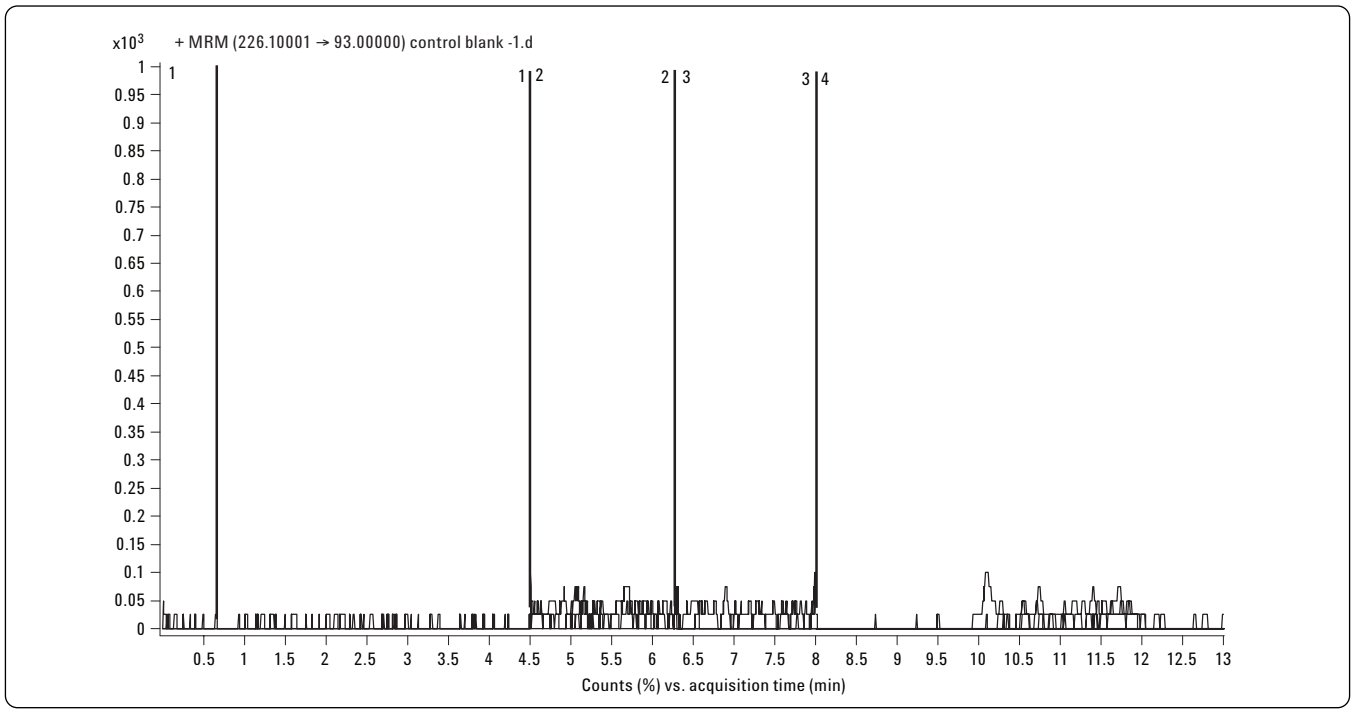


Figure 2a Chromatograms of apple extract blank. No interference was found in the blank.

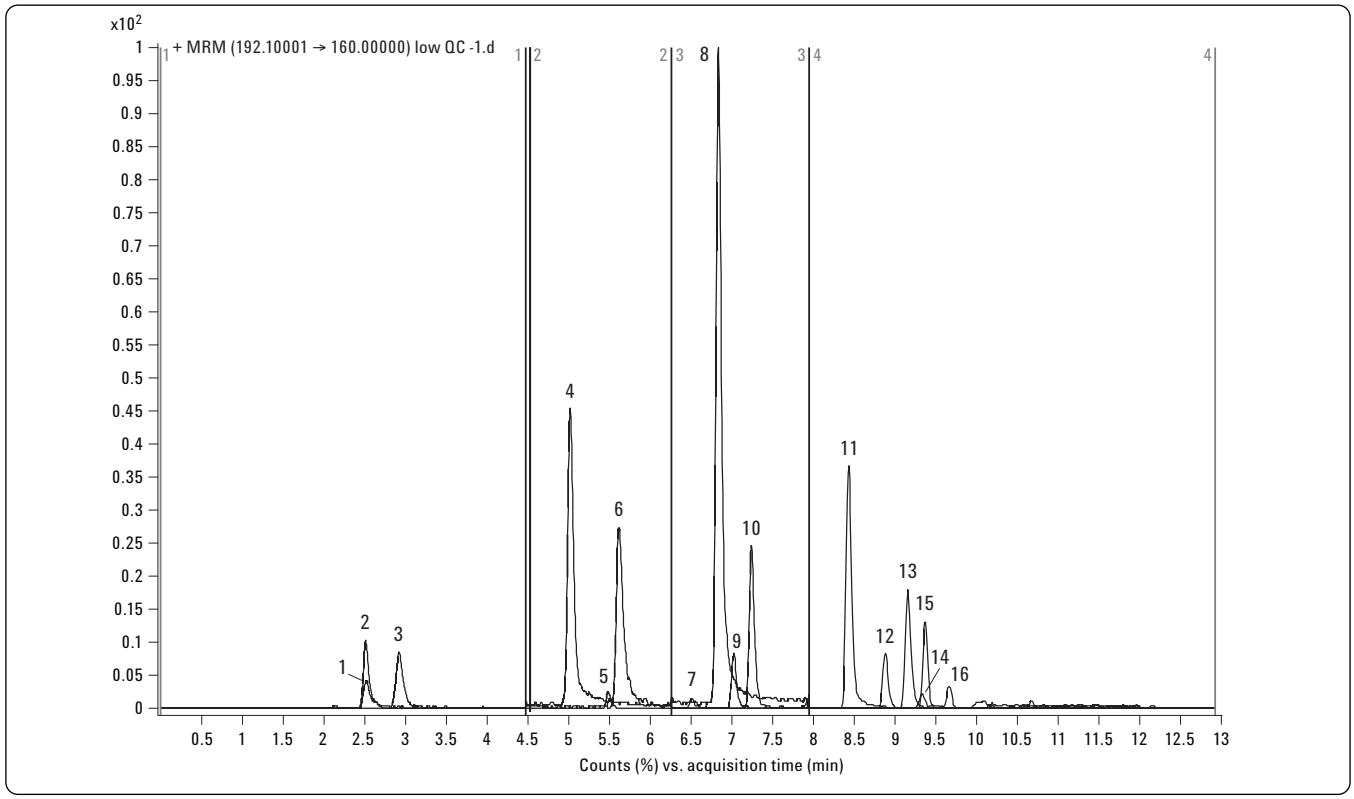


Figure 2b. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlofluanid,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

Linearity and Limit of Quantification (LOQ)

The linear calibration range for all of the pesticides was 5 – 250 ng/g. Since two different dispersive SPE sizes (1 mL and 6 mL sample volume) 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 was used as an internal standard (IS) 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 significantly 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 6 mL dispersive SPE.

Table 3. Linearity of Pesticides in Apple Extract

Analytes	1 mL dispersive SPE Regression equation	R^2	6 mL dispersive SPE Regression equation	R^2
Methamidophos	Y = 0.3203X – 0.0005	0.9972	Y = 0.3255X – 0.0018	0.9957
Acephate	Y = 0.1373X – 0.0021	0.9975	Y = 0.1375X – 0.0010	0.9953
Pymetrozine	Y = 0.4688X – 0.0009	0.9961	Y = 0.3821X + 0.0007	0.9782
Carbendazim	Y = 1.4253X + 0.0126	0.9931	Y = 1.3379X + 0.0045	0.9903
Imidacloprid	Y = 0.0647X – 0.0004	0.9944	Y = 0.0636X – 0.0006	0.9974
Thiabendazole	Y = 0.9014X + 0.0127	0.9922	Y = 0.8600X + 0.0050	0.9942
Dichlorvos	Y = 0.0364X + 0.0002	0.9884	Y = 0.0362X + 0.0002	0.9892
Propoxur	Y = 2.4398X – 0.0001	0.9989	Y = 2.4272X + 0.0029	0.9994
Thiophanate methyl	Y = 0.3171X – 0.0015	0.9965	Y = 0.2869X – 0.0020	0.9904
Carbaryl	Y = 0.6378X + 0.0017	0.9989	Y = 0.6363X + 0.0003	0.9988
Ethoprophos	Y = 1.0897X – 0.0030	0.9984	Y = 1.0628X – 0.0001	0.9992
Penconazole	Y = 0.2334X – 0.0012	0.9978	Y = 0.2186X – 0.0003	0.9979
Cyprodinil	Y = 0.4805X + 0.0008	0.9992	Y = 0.4697X – 0.0017	0.9985
Dichlorfluanid	Y = 0.0552X – 0.0003	0.9970	Y = 0.0562X – 0.0012	0.9946
Kresoxim methyl	Y = 0.2958X – 0.0005	0.9978	Y = 0.2762X – 0.0003	0.9966
Tolyfluanid	Y = 0.0860X – 0.0011	0.9918	Y = 0.0845X – 0.0008	0.9968

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 (RSD) data of 1 mL and 6 mL dispersive SPE are shown in Tables 4 and 5, respectively. It can be seen from the results, that all of the pesticides but pymetrozine give acceptable recoveries (average of 85.7% for 1 mL and 88.2% for 6 mL) and precision (average of 6.0% RSD for 1 mL and 5.7% RSD for 6 mL). The notorious base-sensitive pesticides such as dichlorfluanid and tolyfluanid showed excellent recovery and precision. Pymetrozine, an acid labile pesticide, shows poor recovery using the European method when compared to the AOAC method [2]. With the AOAC method, an average recovery of 88% with 9.4% average RSD for pymetrozine was obtained [8].

Table 4. Recovery and Repeatability of Pesticides in Fortified Apple With 2 mL EN Dispersive SPE Tube (p/n 5982-5021)

	10 ng/g fortified QC		50 ng/g fortified QC		200 ng/g fortified QC	
Analytes	**Recovery**	**RSD (n=6)**	**Recovery**	**RSD (n=6)**	**Recovery**	**RSD (n=6)**
Methamidophos	73.0	5.6	75.6	3.1	84.3	5.3
Acephate	92.8	4.2	87.2	5.6	95.6	5.8
Pymetrozine	27.1	18.2	24.9	10.5	28.1	12.3
Carbendazim	85.1	5.9	89.5	3.4	84.1	4.7
Imidacloprid	91.0	3.3	102.7	5.4	107.2	4.9
Thiabendazole	84.8	6.8	90.4	3.5	86.7	4.0
Dichlorvos	83.1	13.9	92.2	5.2	93.1	4.6
Propoxur	97.8	2.6	100.2	2.9	100.7	3.8
Thiophanate methyl	79.9	8.5	79.9	2.9	85.5	5.7
Carbaryl	89.3	2.8	92.5	3.6	95.8	4.1
Ethoprophos	93.7	1.6	93.5	2.9	95.7	3.4
Penconazole	109.2	6.7	108.1	5.7	110.6	4.4
Cyprodinil	98.9	6.9	101.2	2.7	102.9	4.3
Dichlorfluanid	85.1	7.8	92.2	4.4	99.4	5.5
Kresoxim methyl	90.4	4.8	99.6	3.9	103.7	4.5
Tolyfluanid	98.3	13.7	102.0	4.0	106.0	4.4

Table 5. Recovery and Repeatability of Pesticides in Fortified Apple With 15 mL EN Dispersive SPE Tube (p/n 5982-5056)

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	77.6	4.9	77.8	6.4	81.2	2.1
Acephate	86.6	7.6	87.8	5.5	91.5	1.5
Pymetrozine	28.1	24.3	27.5	12.2	29.1	10.4
Carbendazim	89.9	6.8	88.9	3.3	81.8	3.6
Imidacloprid	105.3	10.8	105.2	4.8	106.6	5.0
Thiabendazole	89.8	4.8	87.6	3.1	84.2	1.4
Dichlorvos	97.8	14.5	98.2	5.6	98.1	2.5
Propoxur	99.5	3.8	104.0	2.6	100.9	3.3
Thiophanate methyl	87.4	5.8	88.3	4.7	89.0	7.6
Carbaryl	92.9	6.1	93.7	3.0	93.6	2.4
Ethoprophos	94.8	5.5	99.2	3.0	98.8	3.8
Penconazole	106.8	4.9	111.2	3.0	109.0	4.1
Cyprodinil	102.7	4.5	105.7	3.5	102.4	2.6
Dichlorfluanid	99.7	18.9	97.4	4.5	98.9	5.5
Kresoxim methyl	102.6	12.0	106.1	2.0	106.1	5.6
Tolyfluanid	92.0	9.3	105.5	3.3	105.1	4.3

Figure 3 shows the recovery and precision results comparison of 1 mL dispersive SPE and 6 mL dispersive SPE. The two different dispersive SPE clean-ups were performed by transferring 1 mL or 6 mL of ACN extract from the sample tube after the extraction step. 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 appear to be independent of volume used. There was < 10% difference in recovery and < 5% difference for RSD. Both approaches provided efficient sample clean-up, and generated relatively equivalent results.

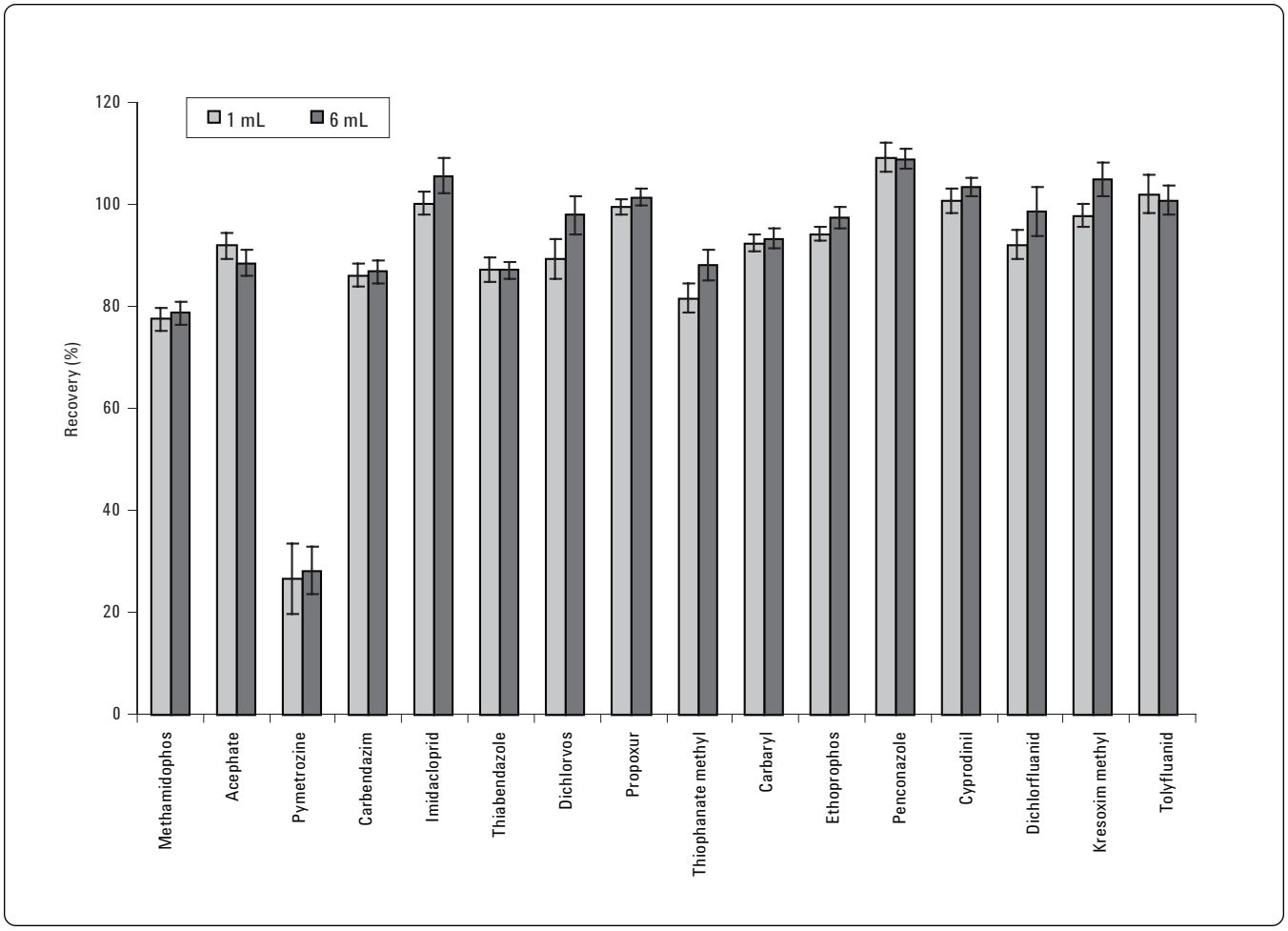


Figure 3. Results comparison of 1-mL dispersive SPE and 6-mL dispersive SPE.

Conclusions

The Agilent SampliQ QuEChERS EN fruit and vegetable kit provides a simple, fast and effective method for the purification and enrichment of selective representative pesticides in apple. The recovery and reproducibility results, based on matrix spiked standards, were acceptable for selected pesticide residue determination in apple. The impurities and matrix effect from apple were minimal and did not interfere with the quantitation of target compounds. The LOQs of the pesticides were lower than their MRLs in fruits and vegetables. As the selected pesticides represented a broad variety of different classes and properties, the Agilent SampliQ QuEChERS EN kit for General Fruits and Vegetables can be used for other pesticides in similar food matrices.

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