Application Note

Food & Beverage Testing

Determination of Over 300 Pesticides in Tobacco

Using Agilent Captiva EMR–LPD Passthrough Cleanup and LC/MS/MS and GC/MS/MS Detection

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Abstract

This application note presents the development and optimization of a multiresidue method for the analysis of pesticide residues in tobacco. The method involves sample extraction with the Agilent Bond Elut QuEChERS EN extraction kit, followed by passthrough cleanup with Agilent Captiva Enhanced Matrix Removal–Low Pigment Dry (EMR–LPD), then analysis by LC/MS/MS and GC/MS/MS. The newly developed method demonstrated efficient matrix removal, acceptable target quantitation results, and low failure rate for analysis of a large panel of pesticides in tobacco. Excellent method quantitation results were achieved for over 300 LC- and GC-amenable pesticides, with 70 to 120% average recovery achieved for >95% of targets, and <20% average RSD for >98% targets in tobacco. The matrix removal assessment by dried residue weight indicated that ~60% of tobacco co-extractives were removed. The passthrough cleanup demonstrated as a simplified method that saves time and labor.

Introduction

Tobacco is an important commercial crop that is consumed worldwide. In order for growers to maintain crop quality and yield, pesticides are applied during cultivation, storage, and transportation for pest and disease control. Public exposure to pesticides through tobacco use has thus drawn significant global attention. Therefore, the monitoring of pesticides residues in tobacco requires reliable analytical methods for safety evaluation to comply with the guidance residue levels (GRLs) regulated by CORESTA.[1]

Tobacco is a complex dry matrix, containing a large variety of ingredients, including carbohydrates, proteins, fatty acids, waxes, pigments, alkaloids, and nicotine.[2,3] The complicated matrix makes sample preparation challenging for simultaneous multiresidue pesticides extraction and matrix removal. Commonly used sample preparation methods involve the use of QuEChERS or modified QuEChERS extraction, followed by dispersive SPE cleanup.[4, 6]

Captiva EMR with Carbon S cartridges applies passthrough cleanup methodology for fast and efficient sample matrix removal. Captiva EMR general pigmented dry (EMR–GPD) and EMR low pigmented dry (EMR–LPD) cartridges are specifically targeted to complex botanical dry matrices. Both cartridges contain the Agilent proprietary sorbents Carbon S and Captiva EMR–Lipid, blended with primary secondary amine (PSA) and C18 in an optimized formula. Captiva EMR–Lipid sorbent provides highly selective and efficient lipid removal, while PSA sorbent provides efficient removal of fatty acids and other acids, Carbon S sorbent effectively removes pigments, and EC-C18 provides further hydrophobic matrix cleanup. The blended formula was carefully developed and optimized to deliver the best balance between matrix removal and target recovery for complex dry matrices with different levels of pigment components. For general pigmented dry matrix, Captiva EMR–GPD is usually recommended, while for low pigmented dry matrix, Captiva EMR–LPD is advised.

In this study, a sample preparation method using QuEChERS extraction followed by Captiva EMR–LPD passthrough cleanup was developed for the LC/MS/MS and GC/MS/MS analysis of over 300 common pesticides in tobacco. A thorough comparison of the new cleanup method and traditional dispersive SPE (dSPE) cleanups was also conducted.

Experimental

Chemicals and reagents

Pesticide standards and internal standards (IS) were either obtained as the standard mix stock solutions from Agilent Technologies (part number 5190-0551) and Restek (Bellefonte, PA, U.S.A.) or as individual standard stock solutions or powder from Sigma-Aldrich (St Louis, MO, U.S.A.). HPLC grade acetonitrile (ACN) was from Honeywell (Muskegon, MI, U.S.A.). Reagent grade acetic acid, ammonium acetate, and ammonium fluoride were also from Sigma-Aldrich.

Solutions and standards

The standard spiking solutions, including LC- and GC-standard spiking solutions, and the IS spiking solutions were prepared at 10 µg/mL in 1:1 ACN/water or ACN and stored at –20 °C in a freezer. The standard and IS spiking solutions were warmed up thoroughly at room temperature, vortexed before use, and stored after use.

The ACN with 1% acetic acid extraction solvent was prepared by adding 10 mL of glacial acetic acid into 990 mL of ACN and stored at room temperature.

Equipment and material

The LC/MS/MS study was performed using an Agilent 1290 Infinity LC system coupled to an Agilent 6490 triple quadrupole LC/MS. The 1290 Infinity LC system consisted of an Agilent 1290 Infinity binary pump (G4220A), an Agilent 1290 Infinity autosampler (G4226A), and an Agilent 1290 Infinity thermostatted column compartment (G1316C). The coupled 6490 triple quadrupole LC/MS was equipped with an Agilent Jet Stream electrospray ion source. Agilent MassHunter Workstation software was used for data acquisition and analysis.

The GC/MS/MS study was performed using an Agilent 8890 GC and Agilent 7000E triple quadrupole GC/MS system (GC/TQ). The GC was configured with the Agilent 7693A automatic liquid sampler (ALS) and a 150-position tray. The system used a multimode inlet (MMI). Midcolumn backflush configuration was set up using two identical 15 m columns connected by an Agilent purged ultimate union (PUU) and controlled by the Agilent 8890 pneumatic switching device (PSD) module. Please refer the Agilent application note by Andrianova[7] for GC/TQ configuration.

Data were acquired in dynamic MRM (dMRM) mode. The acquisition method was retention time locked to match the retention times in the Agilent MassHunter Pesticide & Environmental Pollutant MRM Database (P&EP 4), which was used to seamlessly create the MS method. MassHunter Workstation software was used for data acquisition and analysis.

Other equipment used for sample preparation included: a Centra CL3R centrifuge (Thermo IEC, MA, U.S.A.), a Geno/Grinder (SPEX, NJ, U.S.A.), a Multi Reax test tube shaker (Heidolph, Schwabach, Germany), pipettes and a repeater (Eppendorf, NY, U.S.A), an Agilent positive pressure manifold 48 processor (PPM-48) (part number 5191-4101), the Agilent Bond Elut QuEChERS EN extraction kit (part number 5982-5650), the Agilent Captiva EMR–LPD cartridge, 6 mL (part number 5610-2092), the Agilent Bond Elut QuEChERS EMR–Lipid polish pouch, 3.5 anhydrous $MgSO_4$ (part number 5982-0102), and ceramic homogenizers, 50 mL tubes, 100/pk (part number 5982-9313).

Instrument conditions

Table 1 lists the LC/MS/MS conditions. For targets' dynamic multiple reaction monitoring (dMRM) parameters, please see the application note by Zhao.[8] Table 2 lists the GC/MS/MS conditions. For targets' dMRM parameters, please refer to the Agilent MassHunter Pesticides & Environmental Pollutant MRM Database (P&EP 4) (part number G9250AA).

Figure 1 shows a typical MRM chromatogram of targeted pesticides in the fortified tobacco sample at the level of 100 ng/g, prepared by QuEChERS EN extraction followed by Captiva EMR–LPD cleanup.

Table 1. LC/MS method conditions using an Agilent 1290 Infinity LC and Agilent 6490 triple quadrupole LC/MS.

Parameter	Value
LC Conditions	
Columns	Agilent ZORBAX Eclipse Plus C18 column, 2.1 × 100 mm, 1.8 µm (p/n 959758-902) Agilent ZORBAX Eclipse Plus C18 column, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901)
Flow Rate	0.3 mL/min
Column Temperature	40 °C
Injection Volume	2 µL
Mobile Phase	**A:** 10 mM ammonium formate, 0.5 mM ammonium fluoride in water, 0.125% FA **B:** 10 mM ammonium formate, 0.5 mM ammonium fluoride in 95:5 ACN:water, 0.125% FA
Needle Wash	1:1:1:1 ACN:MeOH:IPA:water, 0.2% formic acid
Gradient	**Time (min) %B Flow (mL/min)** 0.0 15 0.3 6.0 95 0.3 8.01 100 0.3
Stop Time	10 min
Post Time	2.3 min
MS Conditions	
Ionization Mode	Electrospray ionization (ESI)
Gas Temperature	120 °C
Gas Flow	20 L/min
Nebulizer	40 psi
Sheath Gas Heater	225 °C
Sheath Gas Flow	11 L/min
Capillary Voltage	4,500 V (positive and negative)
Nozzle Voltage	0 V (both positive and negative)
iFunnel Parameters	**High-pressure RF:** 150 V (positive), 90 V (negative) **Low-pressure RF:** 60 V (positive), 60 V (negative)
Polarity	Positive and negative, refer to Table 4 from reference 1.

Table 2. GC/MS/MS method conditions using an Agilent 8890 GC and Agilent 7000E triple quadrupole GC/MS.

Parameter	Value
Columns	Agilent HP-5ms UI, 15 m × 0.25 mm, 0.25 µm film thickness (two) (p/n 19091S-431UI-KEY)
Carrier Gas	Helium
Column 1 Flow	1.016 mL/min
Column 2 Flow	1.216 mL/min
Injection Volume	1 µL cold splitless
Inlet Liner	Agilent Ultra Inert 2 mm dimpled liner (p/n 5190-2297)
MMI Temperature Program	60 °C for 0.1 min, 600 °C/min to 280 °C and hold
Oven Temperature Program	60 °C for 1 min; 40 °C/min to 170 °C, and then 10 °C/min to 310 °C and hold for 2.25 min

Parameter	Value
Run Time	20 minutes
Backflush Conditions	1.5 min postrun 310 °C oven temperature Postrun total flow 25 mL/min
Transfer Line Temperature	280 °C
Source	Inert extractor source with a 3 mm lens, 280 °C
Vacuum Pump	Performance turbo
Quadrupole Temperature	150 °C
Data Monitoring	Dynamic MRM mode (dMRM)
EM Voltage Gain Factor	10
Solvent Delay	3 min

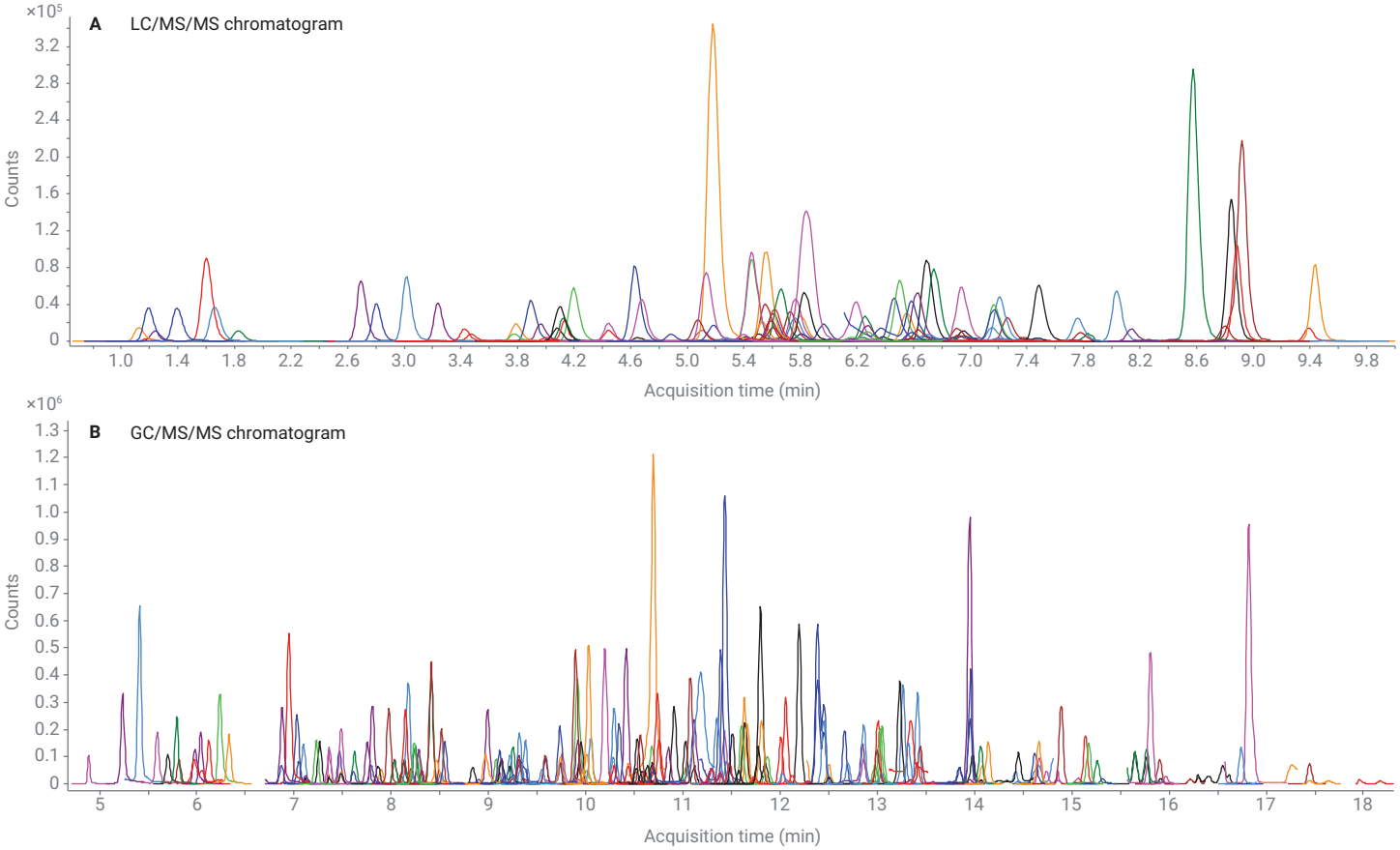


Figure 1. LC/MS/MS MRM chromatogram (A) and GC/MS/MS MRM chromatogram (B) for an extracted tobacco sample fortified with 100 ng/g of targeted pesticides. The sample was prepared using the Agilent Bond Elut QuEChERS EN extraction kit, followed by Agilent Captiva EMR–LPD cleanup.

Sample preparation

The tobacco was purchased from a local grocery store and tobacco leaf pieces were taken out and ground to powder. One gram of tobacco powder was weighed into 50 mL centrifuge tubes. A 5 mL aliquot of water was added. Samples were then vortexed for 20 minutes for complete hydrating and equilibrating of the dry matrix. The sample mixture was extracted following the QuEChERS EN method. The crude extract was then cleaned using the Captiva EMR–LPD 6 mL cartridges and dried by anhydrous $MgSO_4$. The drying step is only needed when using a GC type detection, and when using a combined LC and GC type detection. When LC type detection is used only, the drying step can be obsoleted. The final sample eluent can be injected or with further water or buffer dilution. Samples were then ready for direct injection on GC/MS/MS, or with further dilution before LC/MS/MS detection. The detailed sample preparation procedure is shown in Figure 2. The entire sample preparation procedure results in a 10x dilution factor from target concentration in tobacco to the final tobacco extract after sample extraction and matrix cleanup.

Method performance evaluation

The developed sample preparation method was evaluated in terms of matrix removal; target recovery, reproducibility, and matrix effect; and matrix-matched calibration curve linearity and limits of quantitation (LOQs) in tobacco. To evaluate recovery, reproducibility, and matrix effect, prespiked quality control (PR-QC) samples were prepared at 20 and 100 ng/g in tobacco, in replicates of six, corresponding to 2 and 10 ng/mL in crude sample extract after extraction. The spiked samples and matrix blank samples were then prepared using the developed method. Postspiked QCs (PO-QC) were prepared in matrix blank extract before water dilution, corresponding to 2 and 10 ng/mL. Neat QCs were directly spiked at 2 and 10 ng/mL in reagent blank (ACN with 1% acetic acid), using LC-standard spiking solution only, and then diluted appropriately with water. Six replicates of each type of QC were prepared.

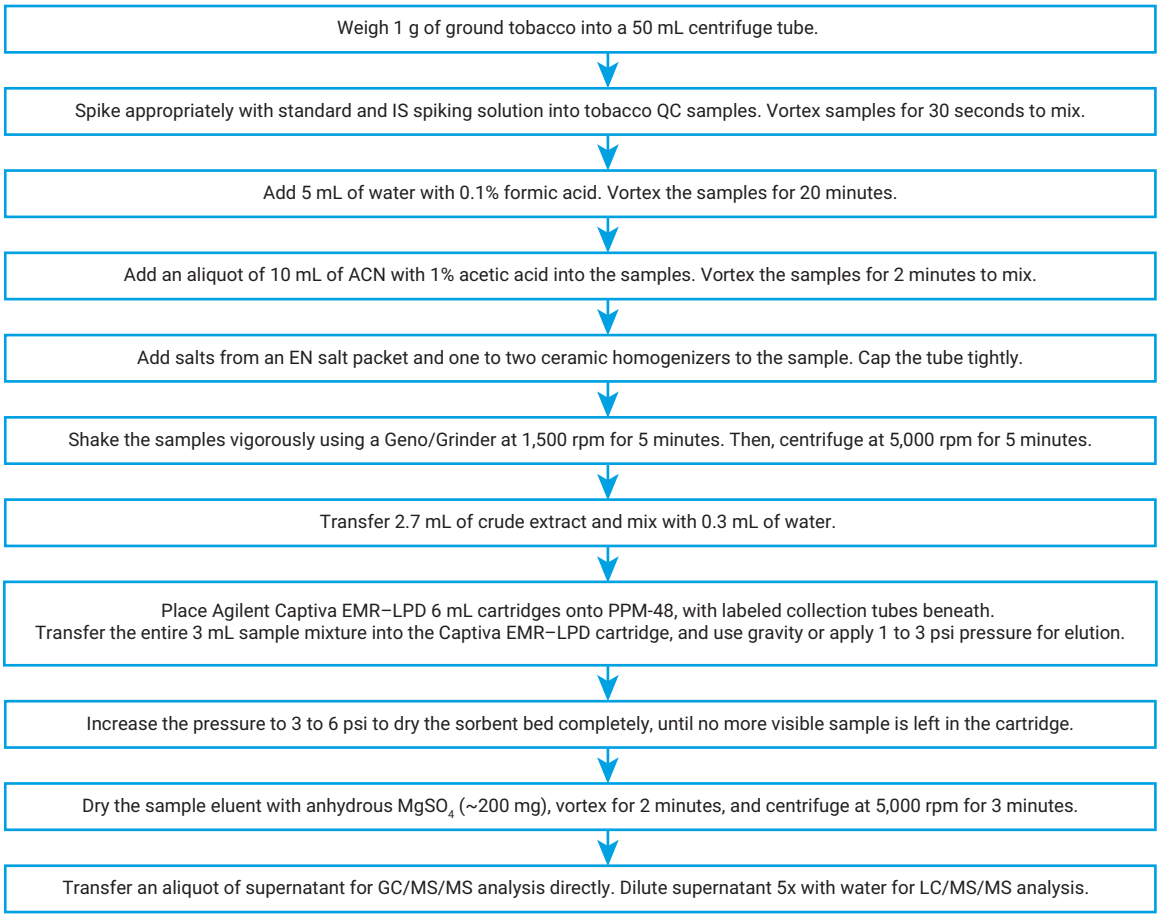


Figure 2. Sample preparation procedure for tobacco samples by Agilent Bond Elut QuEChERS EN extraction followed by Agilent Captiva EMR–LPD passthrough cleanup.

The peak area ratios of corresponding targets in PR-QCs versus PO-QCs were used to calculate target recovery. The peak areas in PR-QCs were used to determine the sample preparation method reproducibility through RSD calculation. The peak area ratios of the corresponding target in PO-QCs versus neat QCs were used for target matrix effect calculation. Matrix-matched calibration curve linearity and LOQs were evaluated by postspiking at the levels of 1, 2, 5, 10, 50, 100, 250, 400, and 500 ng/g in tobacco matrix blank extract, corresponding to 10 to 5,000 ng/g in tobacco. Analyte identification, confirmation, and quantitation were determined from retention times and MRM transitions.

Results and discussion

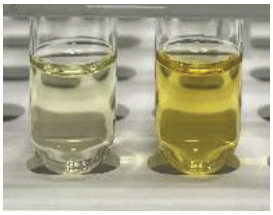
Method development and optimization

Tobacco leaf powder is a yellow to light brown color. The crude extract after QuEChERS extraction is a light yellow color, and so Captiva EMR–LPD is an appropriate choice. The ground tobacco powder was very dry and thus required 5 mL of aqueous buffer addition per 1 g of dry powder, followed with longer mixing by vortex (20 to 30 minutes). This generated a completely hydrated homogenate.

A

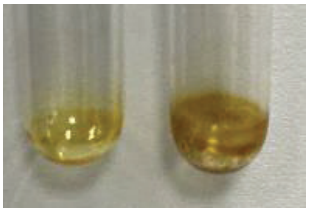
Tobacco

B



Tobacco extract with Agilent Captiva EMR–LPD cleanup (left) versus without cleanup (right)

C



Tobacco extract dried residue with Captiva EMR–LPD cleanup (left) versus without cleanup (right)

D Cinnamon bark extract GC/MS full scan background

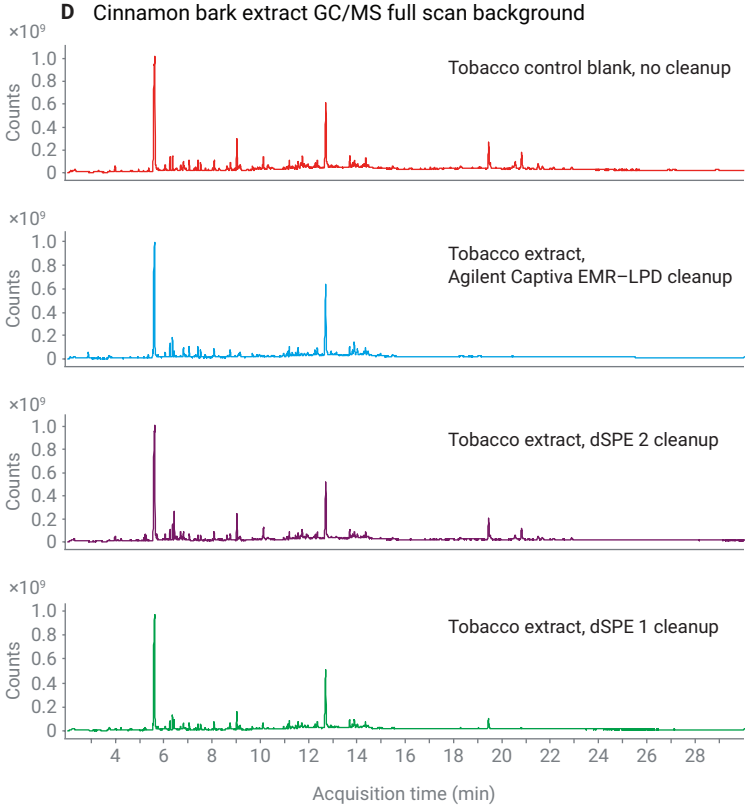


Figure 3. Preliminary study on tobacco matrix. (A) Typical tobacco dry leaf, (B) tobacco extract after QuEChERS extraction with and without Captiva EMR–LPD cleanup, (C) tobacco extract dried residue with and without Captiva EMR–LPD cleanup, (D) tobacco extract GC/MS full scan chromatographic background.

Sample matrix was screened for preliminary matrix complexity and matrix removal efficiency using 1 g of tobacco with 10x dilution. Figure 3A shows a typical tobacco picture to demonstrate its color; 3B shows the tobacco extract with and without Captiva EMR–LPD cleanup, indicating efficient pigment removal provided through cleanup; and 3C shows the tobacco extract dried residue with and without EMR–LPD cleanup. Figure 3D shows the tobacco extract GC/MS full scan chromatographic background, where the top panel is the crude extract without cleanup, the second is the extract with Captiva EMR–LPD cleanup, and the bottom two are the extract with traditional dSPE cleanup. Compared to the traditional dSPE cleanup, Captiva EMR–LPD provided cleanup efficiency equivalent to dSPE 1 cleanup, but better cleanup efficiency than dSPE 2 cleanup.

Sample size was investigated by comparing the extraction of 1 and 2 g of tobacco sample. Tobacco leaf powder was very dry, and so more aqueous buffer was needed for hydration. For 1 g of sample, 5 mL of aqueous buffer was added, while for 2 g of sample, 10 mL of aqueous buffer was added. The sample puree was then extracted and cleaned using the same extraction and cleanup procedure. The evaluation was based on the consideration of matrix removal and quantitation results. The use of 2 g tobacco for extraction resulted in almost double the matrix co-extractives residue and a more complicated chromatogram background than the extraction of 1 g of tobacco, and thus challenged more for sample cleanup after extraction. However, Captiva EMR–LPD cleanup can still provide acceptable matrix removal efficiency, though 10 to 15% lower. For 1 g sample extraction, the cleanup removed 70% of co-extractives residue, while for 2 g of sample, the cleanup removed 56% of co-extractives residue. The comparison of the target quantitation results is shown in Figure 4.

The quantitation results are shown as the statistical targets pass rate for acceptable criteria, which include 70 to 120% recovery (REC), <20% RSD, and 60 to 130% matrix effect (ME). Overall, the 1 g sample size provided slightly better results than 2 g sample size, with 5 to 6% higher pass rate for targets recovery, and 6 to 10% higher pass rate for targets ME. Considering the acceptable method sensitivity and cleaner sample matrix benefits for instrument, the 1 g sample size was used in this study. However, when method sensitivity cannot meet the detection limit, the 2 g sample size can be used directly.

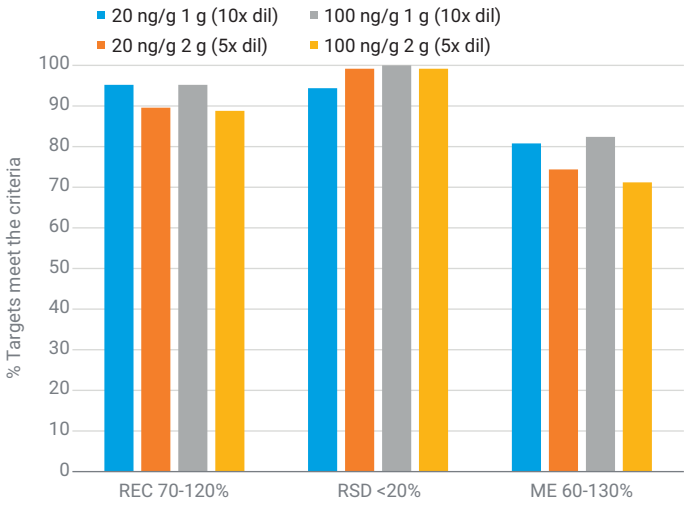


Figure 4. Targets pass rate with acceptable quantitation results: comparison in sample size (1 g versus 2 g). Tobacco samples were spiked at 20 and 100 ng/g for performance evaluation on recovery, reproducibility (RSD), and matrix effect (LC/TQ only).

Method quantitation performance assessment

The method quantitation performance was evaluated by target recovery, reproducibility, and matrix effect on LC/MS/MS, as well as matrix-matched calibration linearity and limits of quantitation (LOQs).

A) Target recovery, reproducibility, and matrix effect

These parameters are directly related to method quantitation accuracy and data quality. Therefore, it is particularly important to use these parameters to demonstrate quantitation method performance. Figure 5 shows the method performance statistical results. Results were calculated based on the average of 20 and 100 ng/g spiking levels, with six replicates of each level. The results show that over 97% of targets received 70 to 120% recovery. For reproducibility, over 98% of targets received <20% RSD. For matrix effect on LC/MS/MS, over 80% of targets are within the 60 to 130% window.

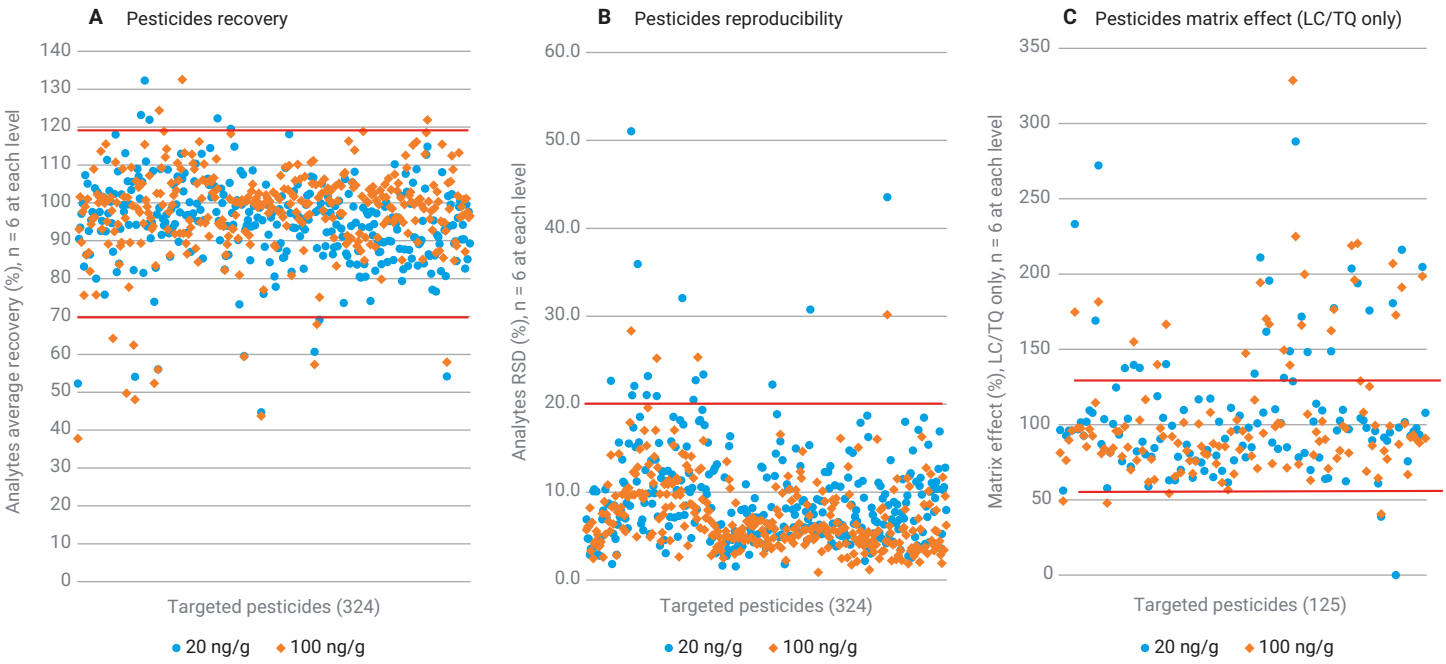


Figure 5. Method quantitation individual target results at 20 and 100 ng/g level in tobacco for (A) pesticides recovery, (B) pesticides reproducibility, and (C) pesticides matrix effect (LC/TQ only).

B) Matrix matched calibration and LOQ

Matrix matched calibration standards were made by postspiking the standards into a final sample extract at the range of 1 to 500 ng/mL. Considering the 10x dilution factors introduced during sample extraction, this corresponded to 10 to 5,000 ng/g in tobacco. Linear regression and $1/x^2$ weight were used for calibration curve generation, with quadratic regression or 1/x weight being used for some exceptions. The calibration dynamic range was determined based on LOQ sensitivity and selectivity requirements, and high concentration level alignment with the calibration curve. Figure 6 shows the summary for the results of targeted pesticides' matrix matched calibration curves in tobacco. Results show that, for the total of over 300 pesticides, full dynamic calibration range (10 to 5,000 ng/g in tobacco) with linear regression and R^2 >0.99 was achieved for 84% of targets; full dynamic range with quadratic regression and R^2 >0.99 was achieved for about 4% of targets. Thirty-four out of 325 targets showed a modified range with either linear or quadratic regression and R^2 >0.99, due to either the lack of sensitivity or selectivity at low calibration levels, or matrix positive occurrence resulting in a raised LOQ.

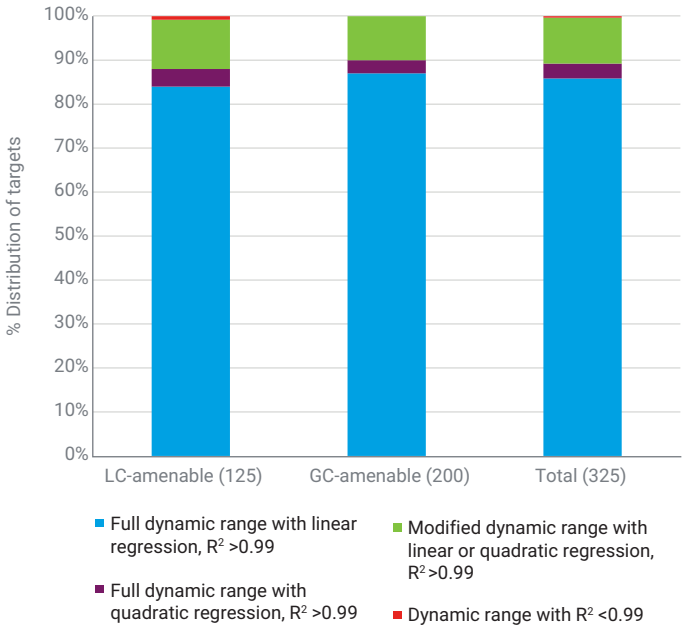


Figure 6. Results for targeted pesticides' matrix-matched calibration curves in tobacco by LC/MS/MS and GC/MS/MS detection. The full dynamic range was 10 to 5,000 ng/g in tobacco.

C) Comparison of Captiva EMR–LPD with traditional dSPE cleanup

The Captiva EMR–LPD passthrough cleanup was thoroughly compared to traditional dispersive SPE (dSPE) cleanup in target quantitation results. To directly compare the impact of the cleanup methods, the crude tobacco matrix blank was collected in bulk, then spiked with standard at the 20 ng/mL level and used for different cleanup methods. The dSPE 1 kit contains PSA (50 mg), GCB (7.5 mg), C18 (50 mg), and $MgSO_4$ (150 mg), while the dSPE 2 kit contains PSA (25 mg), GCB (2.5 mg), and $MgSO_4$ (150 mg). The results were compared based on targets recovery, RSD, and matrix effect on LC/MS/MS. Figure 7 shows the results comparison using the representative and sensitive pesticides for (A) recovery, (B) RSD, and (C) ME.

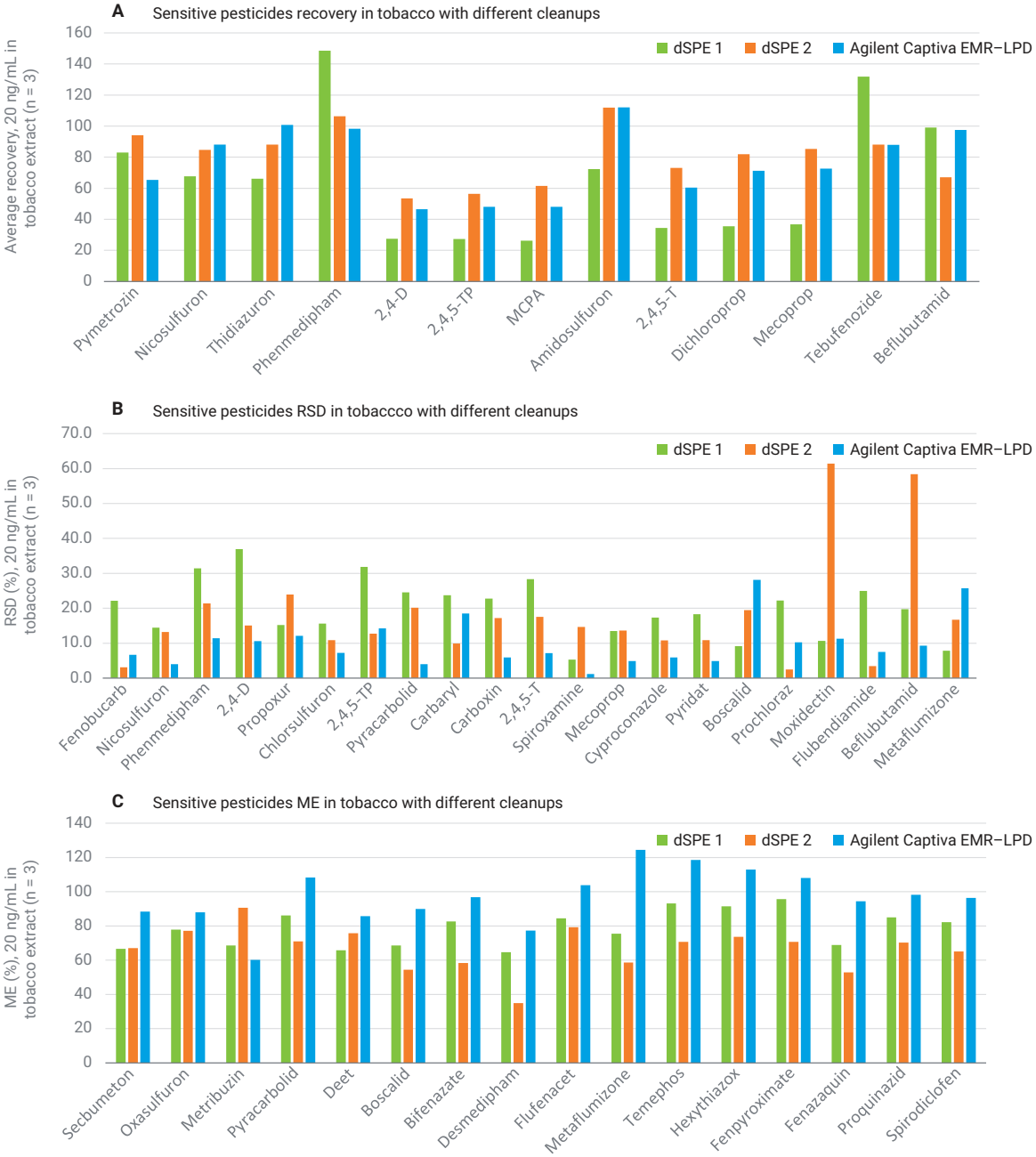


Figure 7. Comparison of cleanup in tobacco matrix on sensitive pesticides between Agilent Captiva EMR–LPD passthrough cleanup and two common dSPE cleanups in terms of (A) recovery, (B) RSD, and (C) matrix effect.

Compared to dSPE 1 cleanup, Captiva EMR–LPD cleanup delivers roughly equivalent matrix cleanup efficiency and matrix effect, but better recovery and reproducibility results. Compared to dSPE 2 cleanup, Captiva EMR–LPD cleanup delivers improved matrix cleanup efficiency and less matrix effect, without compromising on targets' recovery and reproducibility, especially sensitive targets such as acidic pesticides. Considering the cleanup procedure, the passthrough cleanup is a more simplified method relative to traditional dSPE cleanup, saving analysts' bench time and effort when manipulating the small dSPE tubes (for example, uncapping and capping, centrifuging, and transferring sample). Cartridge elution can usually be done by gravity, with 10 to 15 minutes required for 3 mL of sample elution.

Conclusion

A simple, rapid, and reliable method using Agilent Bond Elut QuEChERS EN extraction followed by Agilent Captiva EMR–LPD cartridge passthrough cleanup was developed and verified for over 300 pesticides in tobacco by LC/MS/MS and GC/MS/MS. The novel Captiva EMR–LPD cleanup method provides convenient and simplified sample passthrough cleanup; selective and efficient matrix removal for cigarette tobacco; and a high pass rate for targets with acceptable pesticide recovery, reproducibility, and matrix effect.

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Appendix: Targeted pesticides lists

1) LC-amenable targets

- Pymetrozin
- Mathamidophos
- Acephate
- Omethoate
- Aminocarb
- Propamocarb
- Dinotefuran
- Carbendazim
- Monocrotophos
- Nitenpyram
- Thiabendazole
- Fuberidazole
- Thiamethoxam
- Cymoxanil
- Mexacarbate
- Ethirimol
- Metamitron
- Fenuron
- Chloridazon
- Imidacloprid
- Cymiazol
- Dimethoate
- Fenobucarb
- Acetamiprid
- Metsulfuron
- Flumetsulam
- Tebuthiuron
- 4-Nitrophenol
- Thiacloprid
- Nicosulfuron
- Thidiazuron
- Secbumeton
- Oxasulfuron
- Bentazon
- Carfentrazone-ethyl
- Imazalil
- Lenacil
- Metribuzin
- Cyazofamid
- Phenmedipham
- 2,4-D
- Propoxur
- Chlorsulfuron
- Dioxacarb
- Carbofuran
- Methabenz thiazurone
- 2,4,5-TP
- MCPA
- Amidosulfuron
- Cycluron
- Chlorotoluron
- Flutriafol
- Pyracarbolid
- Fluometuron
- Forchlorfenuron
- Carbaryl
- Fosthiazate
- Azaconazole
- Methoprotryne
- Deet
- Fenpropidin
- Carboxin
- Diuron
- 2,4,5-T
- Spiroxamine
- Metobromuron
- Mecoprop
- Dimethomorph I
- Dimethachlor
- Chlorantraniliprole
- Clomazone
- Dimethomorph II
- Cyproconazole
- Furalaxyl
- Chloroxuron
- Spinosad A
- Linuron
- Iprovalicarb
- Halofenozide
- Pyridat
- Fenamiphos
- Promecarb
- Myclobutanil
- Azoxystrobin
- Manipropamid
- Fenamidone
- Boscalid
- Spinosad D
- Fluopicolide
- Isoxaben
- Bifenazate
- Desmedipham
- Penconazole
- Prochloraz
- Fluoxastrobin
- Isoprothiolane
- Rotenone
- Flufenacet
- Dimoxystrobin
- Cyprodinil
- Moxidectin
- Azinphos-ethyl
- Tebufenozide
- Flubendiamide
- Beflubutamid
- Dinoseb
- Kresoxim-methyl
- Picoxystrobin
- Pyraclostrobin
- Isofenphos-methyl
- Diflufenican
- Trifloxystrobin
- Metrafenone
- Metaflumizone
- Fluazinam
- Temephos
- Pyripoxyfen
- Hexythiazox
- Tralkoxydim
- Buprofezin
- Fenpyroximate
- Fenazaquin
- Proquinazid
- Pyridaben
- Spirodiclofen

2) GC-amenable targets

- Allidochlor
- Dichlorobenzo nitrile, 2,6-
- Biphenyl
- Mevinphos, E-
- 3,4-Dichloroaniline
- Pebulate
- Etridiazole
- *cis*-1,2,3,6-Tetrahydro phthalimide
- N-(2,4-dimethylphenyl) formamide
- Methacrifos
- Chloroneb
- 2-Phenylphenol
- Pentachloro benzene
- Tecnazene
- Diphenylamine
- Propachlor
- Cycloate
- 2,3,5,6-Tetrachloroaniline
- Chlorpropham
- Ethalfluralin
- Trifluralin
- Benfluralin
- Sulfotep
- Diallate I
- Phorate
- BHC-alpha
- Hexachlorobenzene
- Dichloran
- Pentachloroanisole
- Atrazine
- Clomazone
- BHC-beta
- Profluralin
- BHC-gamma
- Terbuthylazine
- Terbufos
- Propyzamide
- Pentachloro nitrobenzene
- Fonofos
- Pentachlorobenzo nitrile
- Diazinon
- Pyrimethanil
- Fluchloralin
- Tefluthrin
- Disulfoton
- Terbacil
- BHC-delta
- Isazofos
- Triallate
- Chlorothalonil
- Endosulfan ether
- Pentachloroaniline
- Propanil
- Dimethachlor
- Acetochlor
- Vinclozolin
- Transfluthrin
- Parathion-methyl
- Chlorpyrifos-methyl
- Tolclofos-methyl
- Alachlor
- Propisochlor
- Heptachlor
- Metalaxyl
- Ronnel
- Prodiamine
- Fenitrothion
- Pirimiphos-methyl
- Linuron
- Malathion
- Pentachlorothio anisole
- Dichlofluanid
- Metolachlor
- Anthraquinone
- Fenthion
- Aldrin
- Chlorpyrifos
- Parathion
- Triadimefon
- Dichlorobenzo phenone, 4,4'-
- DCPA
- Fenson
- Bromophos
- Diphenamid
- Pirimiphos-ethyl
- Isopropalin
- Isodrin
- MGK-264
- Pendimethalin
- Metazachlor
- Penconazole
- Chlozolinate
- Allethrin
- Heptachlor exo-Tolylfluanid
- Fipronil
- Chlorfenvinphos
- Bromfenvinfos-methyl
- Triflumizole
- Quinalphos
- Triadimenol
- Folpet
- Procymidone
- Chlorbenside
- Bromophos-ethyl
- Chlordane-trans
- DDE-o,p'
- Paclobutrazol
- Tetrachlorvinphos
- Endosulfan I
- Chlordane-cis
- Flutriafol
- Nonachlor, *trans*-
- Chlorfenson
- Flutolanil
- Bromfenvinfos
- Iodofenphos
- Fenamiphos
- Prothiofos
- Fludioxonil
- Profenofos
- Pretilachlor
- DDE-p,p'
- Oxadiazon
- Dieldrin
- Oxyfluorfen
- Tricyclazole
- DDD-o,p'
- Myclobutanil
- Flusilazole
- Bupirimate
- Nitrofen
- Fluazifop-p-butyl
- Ethylan
- Chlorfenapyr
- Endrin
- Chlorobenzilate
- Endosulfan II
- DDD-p,p'
- DDT-o,p'
- Ethion
- Nonachlor, *cis*-
- Chlorthiophos
- Endrin aldehyde

- Sulprofos
- Triazophos
- Carbophenothion
- Methoxychlor olefin
- Carfentrazone-ethyl
- Edifenphos
- Norflurazon
- Endosulfan sulfate
- DDT-p,p'
- Lenacil
- Methoxychlor, o,p'-
- Hexazinone
- Tebuconazole
- Piperonyl butoxide
- Resmethrin
- Iprodione
- Nitraline
- Tetramethrin I
- Pyridaphenthion
- Endrin ketone
- Phosmet
- Bromopropylate
- EPN
- Methoxychlor, p,p'-
- Fenpropathrin
- Tebufenpyrad
- Phenothrin I
- Tetradifon
- Phosalone
- Pyriproxyfen
- Leptophos
- Cyhalothrin
- Mirex
- Acrinathrin
- Fenarimol
- Pyrazophos
- Azinphos-ethyl
- Permethrin, (1R)-*cis*-
- Permethrin, (1R)-*trans*-
- Pyridaben
- Fluquinconazole
- Coumaphos
- Prochloraz
- Cyfluthrin I
- Cypermethrin I
- Flucythrinate I
- Ethofenprox
- Fluridone
- Fenvalerate I
- Fluvalinate-tau I
- Deltamethrin

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