Application Note

Food & Beverage Testing

Determination of Over 300 Pesticides in Cinnamon

Using Captiva EMR–GPD passthrough cleanup and LC/MS/MS and GC/MS/MS detection

Author

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Abstract

This application note presents the development and optimization of a multiresidue method for the analysis of pesticide residues in cinnamon powder. The method involves sample extraction with the Agilent Bond Elut QuEChERS AOAC extraction kit, followed by passthrough cleanup with Agilent Captiva Enhanced Matrix Removal–General Pigment Dry (EMR–GPD), then LC/MS/MS and GC/MS/MS analysis. The newly developed method provided efficient matrix removal, acceptable target quantitation results, and low failure rate for analysis of a large panel of pesticides in challenging cinnamon matrix. Excellent method quantitation results were achieved for over 300 pesticides, with 70 to 120% average recovery achieved for >95% of targets, and <20% average RSD for >97% targets in cinnamon. The matrix removal assessment by dried residue weight indicated that ~60% of cinnamon co-extractives were removed. The passthrough cleanup was also demonstrated to be a simplified method, saving time and effort for analysts.

Introduction

Cinnamon bark is consumed worldwide as an important species of medicinal and edible spice. However, cinnamon cultivation, storage, and production usually involves many pesticides being applied for pests, bacterial, and fungal control. These widely used pesticides raise concern of their environmental and health impact. The use of pesticides should therefore comply with existing national and/or international regulations, such as those provided by the European Union (EU) and Codex Alimentarius Commission (CAC).[1]

Dry spices are complex matrices that present significant challenges to reliable pesticides analysis.[2,3] Cinnamon powder is considered one of the most difficult matrices to analyze, with its high complexity and high pigment concentration. The powder usually contains 12 to 15% water, fatty oils, essential cinnamon oil, flavonoids, and glycosides. The complicated matrix significantly challenges sample preparation for simultaneous pesticide extraction and matrix removal. Commonly used sample preparation methods usually involve the use of QuEChERS or modified QuEChERS extraction, followed by dispersive SPE cleanup.[3,4]

Agilent 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 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 efficiently removes fatty acids, Carbon S sorbent effectively removes pigment, 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 favored.

In this study, sample preparation using Captiva EMR–GPD cartridges for passthrough cleanup was optimized for the analysis of over 300 common pesticides in cinnamon by LC/MS/MS and GC/MS/MS.

Experimental

Chemicals and reagents

Pesticide standards and internal standards (IS) were either obtained as 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

A combined LC-standard spiking solution and GC-standard spiking solution, and the IS spiking solution were prepared at 10 µg/mL in 1:1 ACN/water or ACN and stored at –20 °C in a freezer. The standard spiking solutions were warmed up thoroughly to room temperature, sonicated 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 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 8890 pneumatic switching device (PSD) module. Please see the application note by Andrianova[5] for the relevant GC/TQ configuration. Data were acquired in dynamic MRM (dMRM) mode. The acquisition method was retention timelocked 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, 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 AOAC extraction kit (part number 5982-5755), the Agilent Captiva EMR–GPD cartridge, 6 mL (part number 5610-2091), Agilent Bond Elut QuEChERS EMR–Lipid polish pouch, 3.5 g 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.[6] Table 2 lists the GC/MS/MS conditions. For targets' dMRM parameters, please see 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 cinnamon sample at the level of 100 ng/g, prepared by QuEChERS AOAC extraction followed by Captiva EMR–GPD cleanup.

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

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, see Table 4 from reference 1.

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

Columns	Agilent HP-5ms UI, 15 m × 0.25 mm, 0.25 µm (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
Run Time	20 min
Backflush Conditions	1.5 min post run 310 °C oven temperature Post run 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

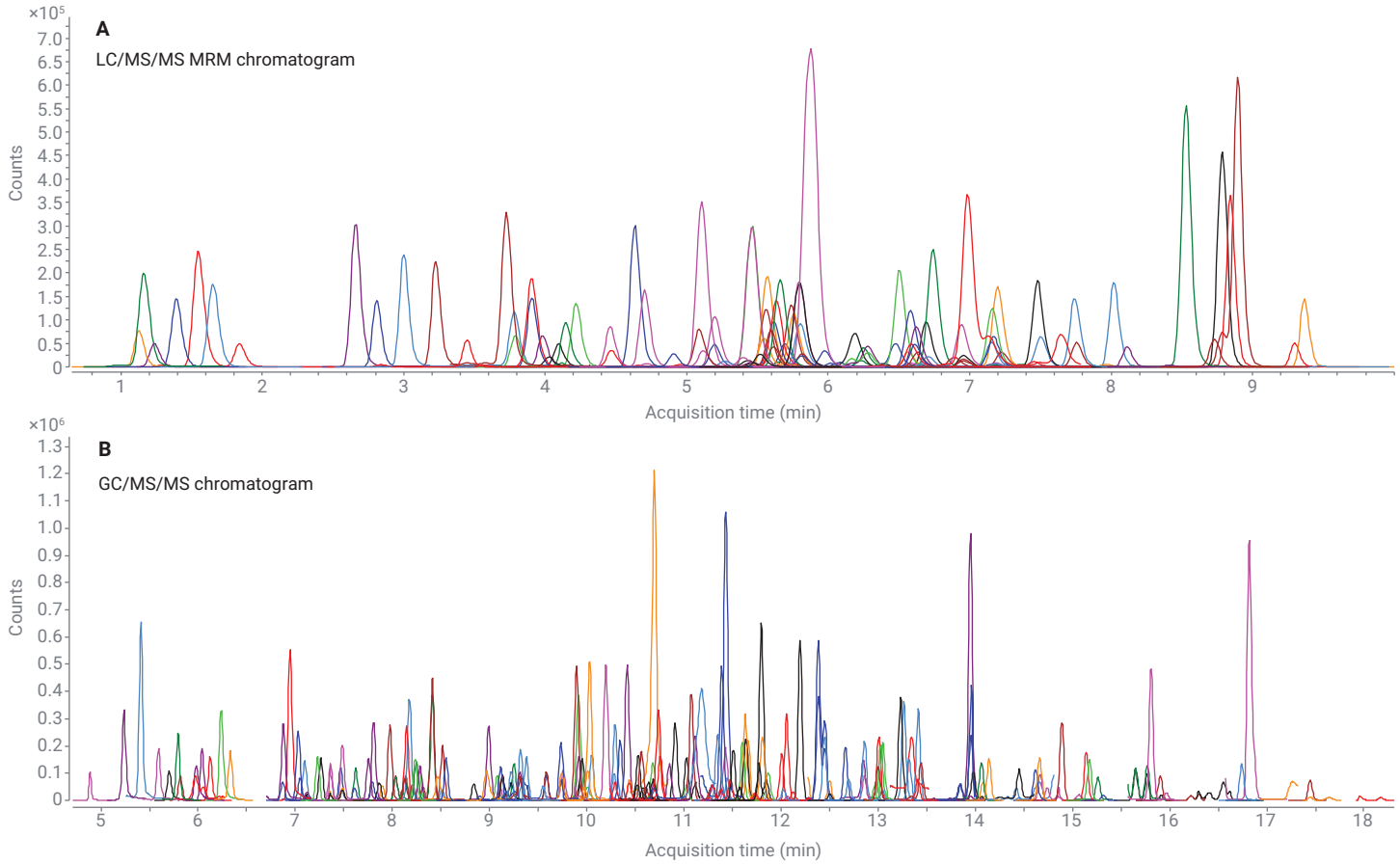

Figure 1. LC/MS/MS MRM chromatogram (A) and GC/MS/MS MRM chromatogram (B) for an extracted cinnamon sample fortified with 100 ng/g of targeted pesticides. The sample was prepared using the Agilent Bond Elut QuEChERS AOAC extraction kit, followed by Agilent Captiva EMR–GPD cleanup.

Sample preparation

The organic cinnamon powder was purchased from a local grocery store. Cinnamon powder was weighed at 1.5 g into 50 mL centrifuge tubes. An aliquot of 4 mL water with 0.1% formic acid was added. Samples were then vortexed for 15 minutes for complete hydration and equilibrating of the dry matrix. The sample mixture was extracted following the QuEChERS AOAC method. After the extraction, 2.7 mL of crude extract was mixed with 0.3 mL of water. The mixed sample was then transferred into the Captiva EMR–GPD 6 mL cartridges for passthrough cleanup. Sample elution was either with gravity or a low level of positive pressure (1 to 3 psi) at a consistent elution flow of 2 to 4 seconds per drop. The elution of 3 mL of sample mixture usually takes 10 to 15 minutes. The sample eluent was dried by anhydrous $MgSO_4$ to completely remove the water residue. Samples were then ready for direct GC/MS/MS analysis, or further dilution before LC/MS/MS analysis. The detailed sample preparation procedure is shown in Figure 2. The entire sample preparation procedure results in a 10× dilution factor from target concentration in cinnamon to the final cinnamon extract after sample extraction and matrix cleanup.

Method development

Cinnamon sample size and dilution factor were screened based on the study of cinnamon matrix complexity and co-extractives residue. The water addition for dry powder hydration was also investigated, with 5 mL of water compared to 10 mL of water based on matrix co-extractives evaluation.

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 cinnamon. To evaluate recovery, reproducibility, and

matrix effect, prespiked quality control (PR-QC) samples were prepared at 10 and 100 ng/g in cinnamon, in replicates of six, corresponding to 1 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 1 and 10 ng/mL. Neat QCs were directly spiked at 1 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. 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 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 0.5, 1, 2, 5, 10, 50, 100, 250, 400, and 500 ng/g in cinnamon matrix blank extract, corresponding to 5 to 5,000 ng/g in cinnamon. Analyte identification, confirmation, and quantitation were determined from retention times and MRM transitions.

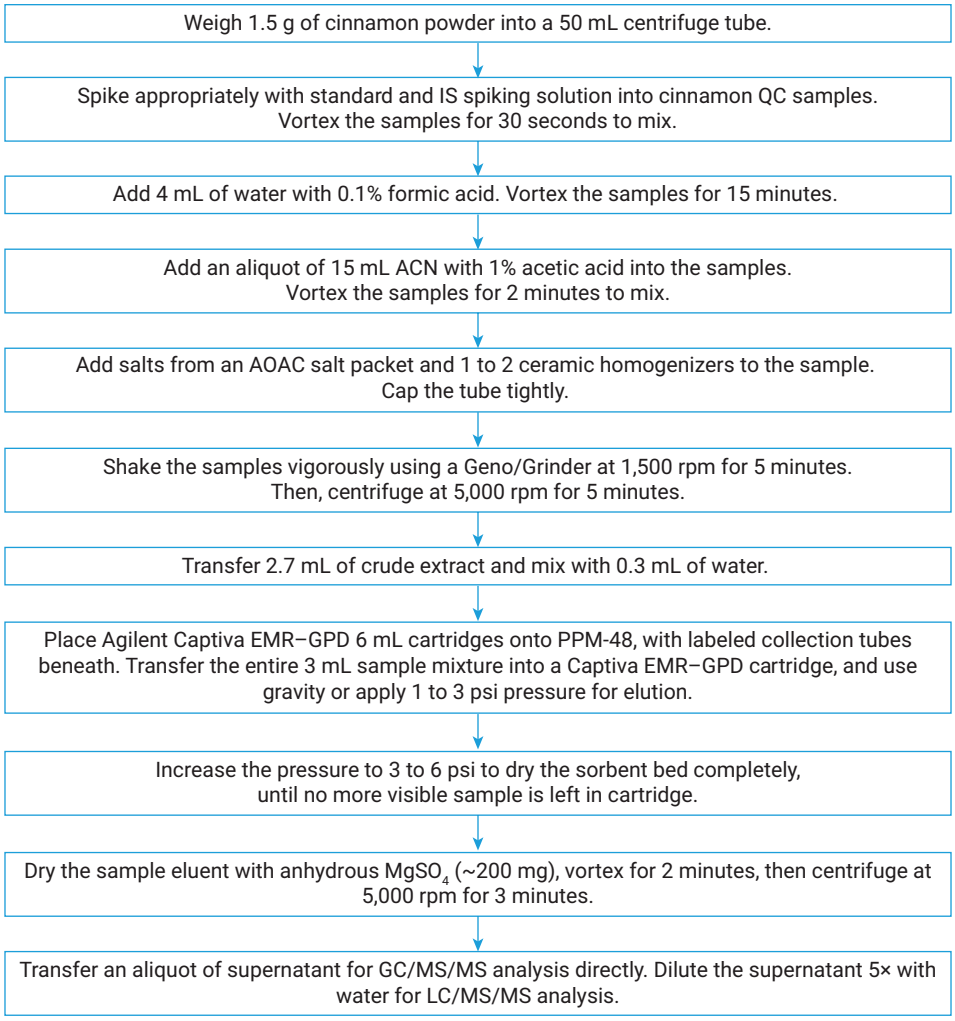

Figure 2. Sample preparation procedure for cinnamon samples by Agilent Bond Elut QuEChERS AOAC extraction followed by Agilent Captiva EMR–GPD passthrough cleanup.

Results and discussion

Method development and optimization

Cinnamon powder is dark brown in color and is considered a general pigmented dry matrix. For this reason, Captiva EMR–GPD is an appropriate choice for passthrough cleanup. Sample matrix was screened for preliminary matrix complexity and matrix removal efficiency using 1.5 g of cinnamon with 10× dilution. Figure 3A shows a typical cinnamon picture to demonstrate its color. Figure 3B shows the crude extract after QuEChERS extraction, which is a relatively dark brown color. Figure 3C shows the crude extract dried residue, weighing 8 to 10 mg per 1 mL of crude extract. Figure 3D shows the cinnamon extract GC/MS full scan chromatographic background, where the top chromatogram is the crude extract without cleanup, the middle two chromatograms are the extract with traditional dSPE cleanup, and the bottom chromatogram is the extract with Captiva EMR–GPD cleanup. The cinnamon matrix is highly pigmented, and these abundant interferences eluted in the relatively early retention window between 5 to 7 minutes, indicating that these interferences are relatively polar. The cleanup after QuEChERS extraction mostly removed the intermediate to late eluting interferences and reduced overall background baseline. Compared to the traditional dSPE cleanup, the Captiva EMR–GPD provided slightly higher cleanup efficiency with 4 to 10% improved matrix background cleanup. The matrix co-extractive residue removal for three cleanup methods was quite comparable, but EMR–GPD cleanup still provided the best residue cleanup efficiency.

Considering the <10 mg of co-extractive residues per 1 mL of crude extract, and the difficulty of early eluting matrix interference removal (RT window of 5 to 7 minutes), a 10× dilution factor proved necessary. As a result, 1.5 g of cinnamon bark powder was used with 10× dilution through the sample preparation.

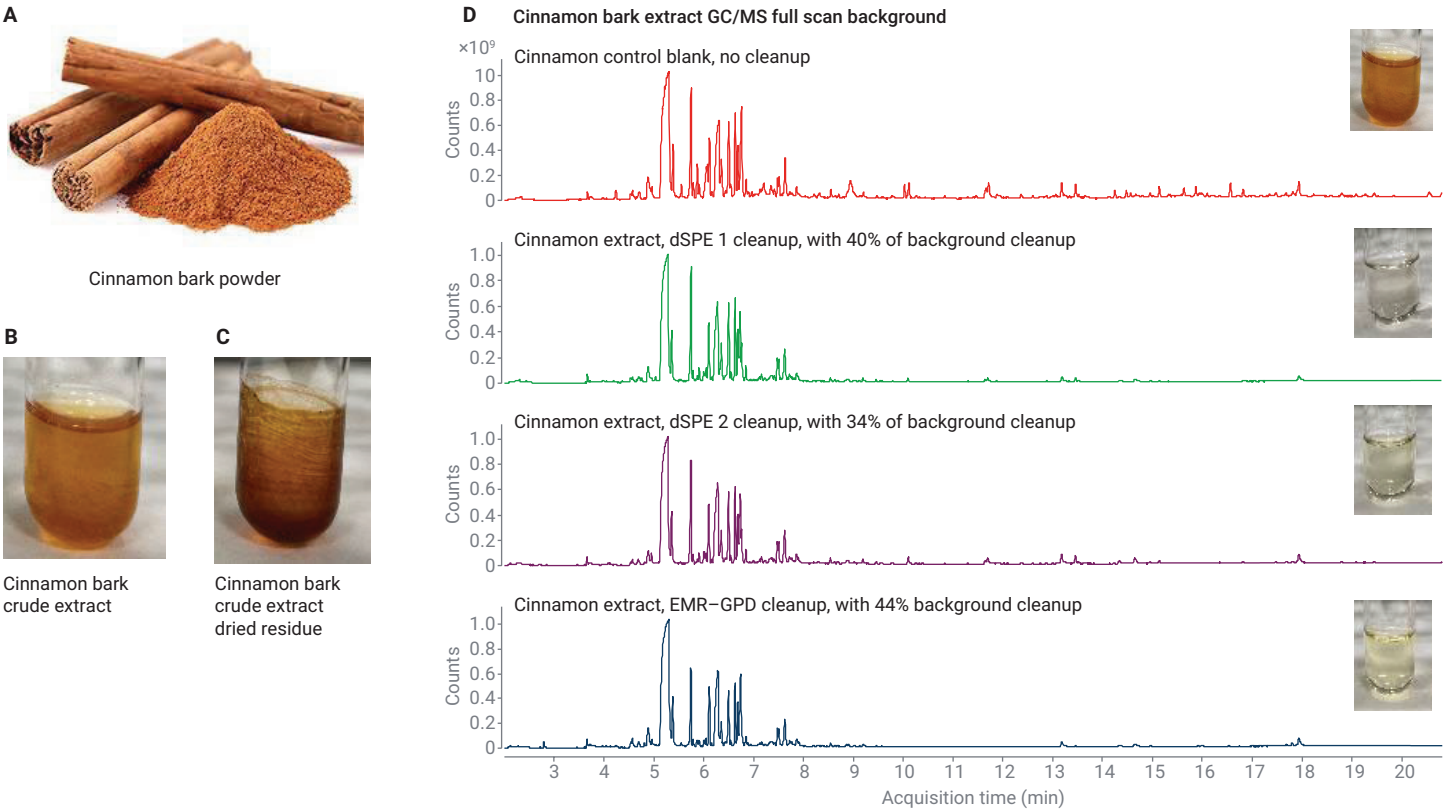

Figure 3. Preliminary study on cinnamon matrix. (A) Typical cinnamon bark powder; (B) crude extract after QuEChERS extraction; (C) dried residue of crude extract; (D) cinnamon bark extract GC/MS full scan chromatographic background.

In addition, the matrix study also showed that less water used for hydration reduced the matrix co-extractives. Compared to the use of 10 mL of water for sample hydration, the matrix co-extractives were reduced by approximately 30% when using 5 mL of water. However, the use of 5 mL water for cinnamon dry sample prewetting compromised the acidic pesticides recovery more significantly. Even so, the use of acidic buffer can help to recover some acidic pesticides. Considering the pros and cons of water hydration, the 4 mL of water with 0.1% formic acid buffer was added to 1.5 g of cinnamon bark powder for sample hydration.

It was reported that the water premixing of sample crude extract impacts analyte recovery when using Captiva EMR–GPD passthrough cleanup.[7] The water premixing ratio before EMR–GPD cleanup was also studied using the following ratios of water to crude cinnamon extract: 0:100, 5:95, and 10:90. The target recovery results for sensitive pesticides are compared in Figure 4. The comparison results show that: A) The addition of water and premixing with the crude extract improved the recoveries of many sensitive targets. B) However, water premixing also compromised recovery of several pesticides, such as dioxacarb and boscalid. As a result, 10% water premixing ratio was shown to be optimal for Captiva EMR–GPD cleanup.

Method quantitation performance assessment

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

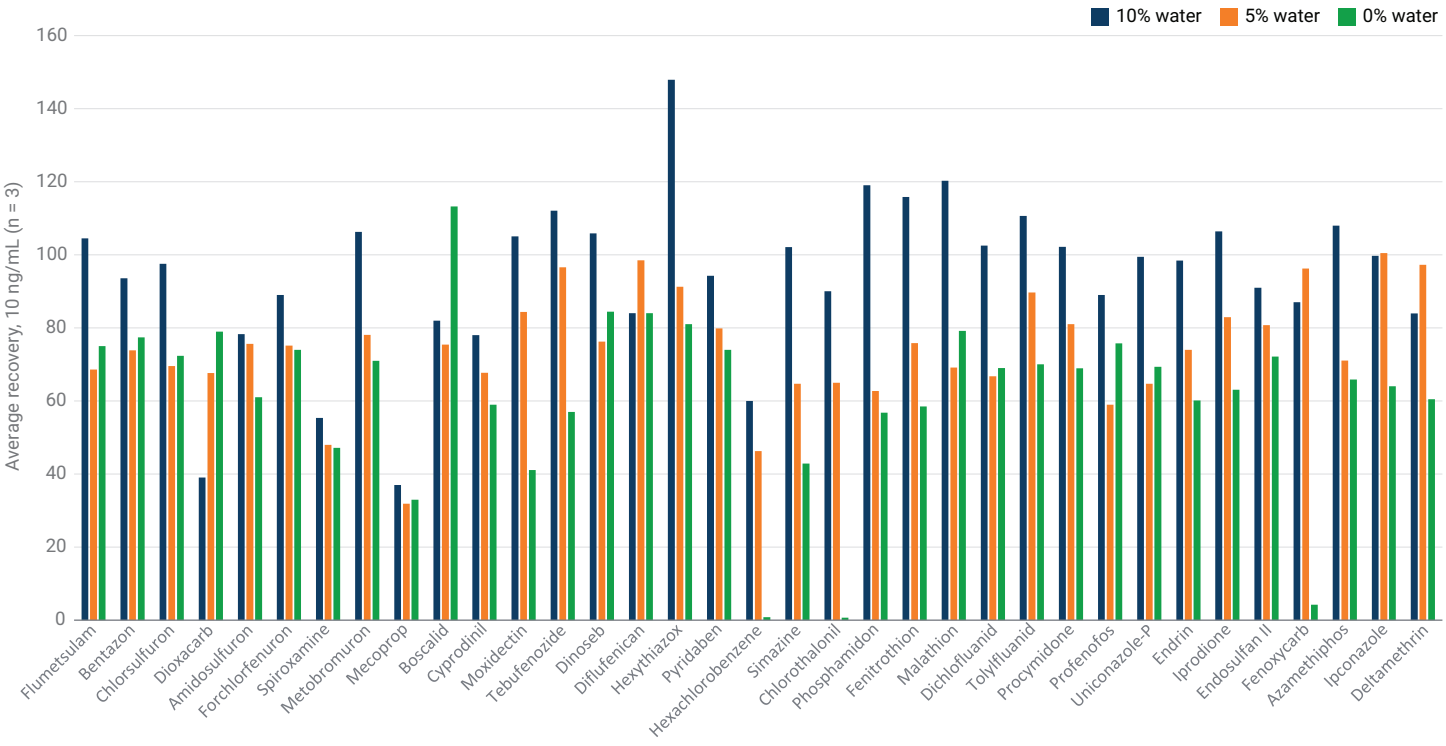

Figure 4. Optimization of water addition before Agilent Captiva EMR–GPD cleanup. Crude cinnamon extract spiked with 10 ng/mL level was used for the comparison.

Target recovery, reproducibility, and matrix effect

These parameters are directly related to method quantitation accuracy and data quality. Therefore, it is very important to use these parameters to demonstrate quantitation method performance. The SANTE/11312/2021 guideline was referred to for method performance assessment.[1] Figure 5 shows the individual target results at 10 and 100 ng/g in cinnamon for pesticide recoveries, reproducibility (RSD), and matrix effect (LC/TQ only) with detection by LC/MS/MS and GC/MS/MS. Results were calculated based on the average of 10 and 100 ng/g spiking levels, with six replicates of each level. The statistical data analysis shows that over 95% of targets received 70 to 120% recovery, and over 98% of targets received 40 to 120% recovery. For reproducibility, over 97% of targets received <20% RSD. In terms of matrix effect on LC/MS/MS, over 85% of targets are within the 60 to 130% window. Of the over 300 pesticides investigated, 12 were not detectable at the 10 ng/g level, due to either matrix interferences or matrix effect. Target stability may also cause the loss of sensitivity at the 10 ng/g level.

Matrix matched calibration and LOQ

Matrix matched calibration standards were made by postspiking the standards into a final sample extract at the range of 0.5 to 500 ng/mL. Considering different 10x dilution factors introduced during sample extraction, this range corresponded to 5 to 5,000 ng/g in cinnamon. 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 of individual target was determined based on the specific target's sensitivity and selectivity at low concentration levels, and whether high concentration level stayed tightly with the calibration curve. Figure 6 shows the summary for the results of targeted pesticides' matrix matched calibration curves in cinnamon. Results show that, for the over 300 pesticides studied, full dynamic calibration range (5 to 5,000 ng/g in cinnamon) with linear regression and R^2 >0.99 was achieved for 88% of targets; full dynamic range with quadratic regression and R^2 >0.99 was achieved for approximately 5% of targets. Approximately 6% of 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 the low end, or matrix positive contribution. The rest of approximately 1% targets did not generate a dynamic range with R^2 >0.99.

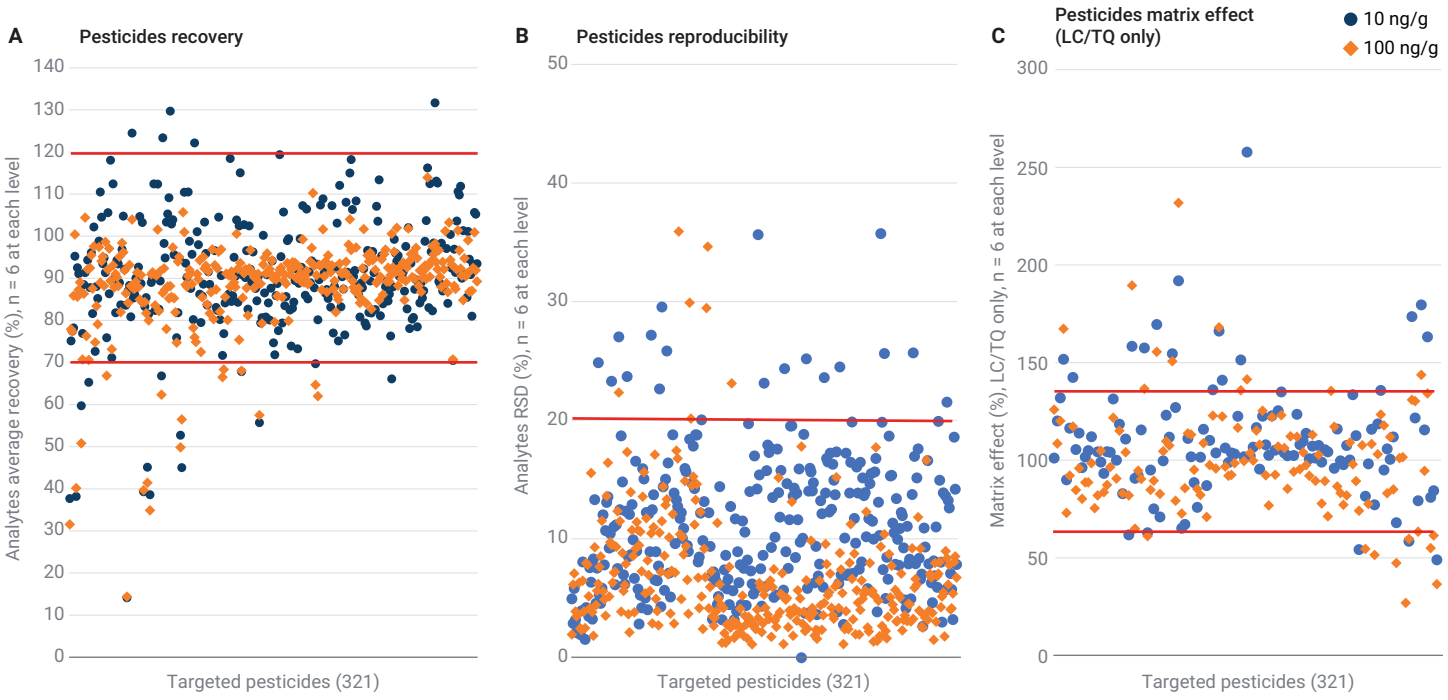

Figure 5. Method quantitation using individual target results at 10 and 100 ng/g in cinnamon for (A) pesticides recovery, (B) pesticides reproducibility, and (C) pesticides matrix effect (LC/TQ only).

Comparison of Captiva EMR–GPD with traditional dSPE cleanup

Compared with traditional dSPE cleanup after QuEChERS extraction, the Captiva EMR–GPD passthrough cleanup improves matrix cleanup efficiency and sensitive pesticides recovery. Figure 7 shows the sensitive pesticides with >30% of recovery difference between Captiva EMR–GPD cleanup and either dSPE cleanup technique. The improved sensitive pesticides recovery can be attributed to: 1) the use of Carbon S sorbent in EMR–GPD blended sorbents, instead of traditional GCB sorbent use in the classic dSPE kit; 2) a better buffering effect during passthrough cleanup, with a small percentage of water in the sample mixture.

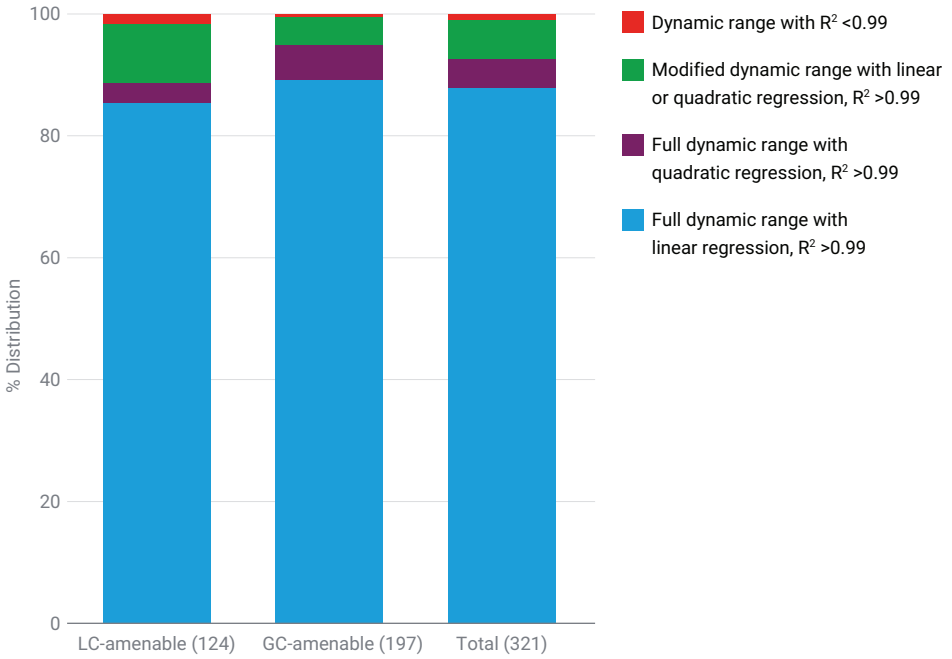

Figure 6. Results for targeted pesticides' matrix matched calibration curves in cinnamon by LC/MS/MS and GC/MS/MS detection. The full dynamic range was 5 to 5,000 ng/g in cinnamon bark powder.

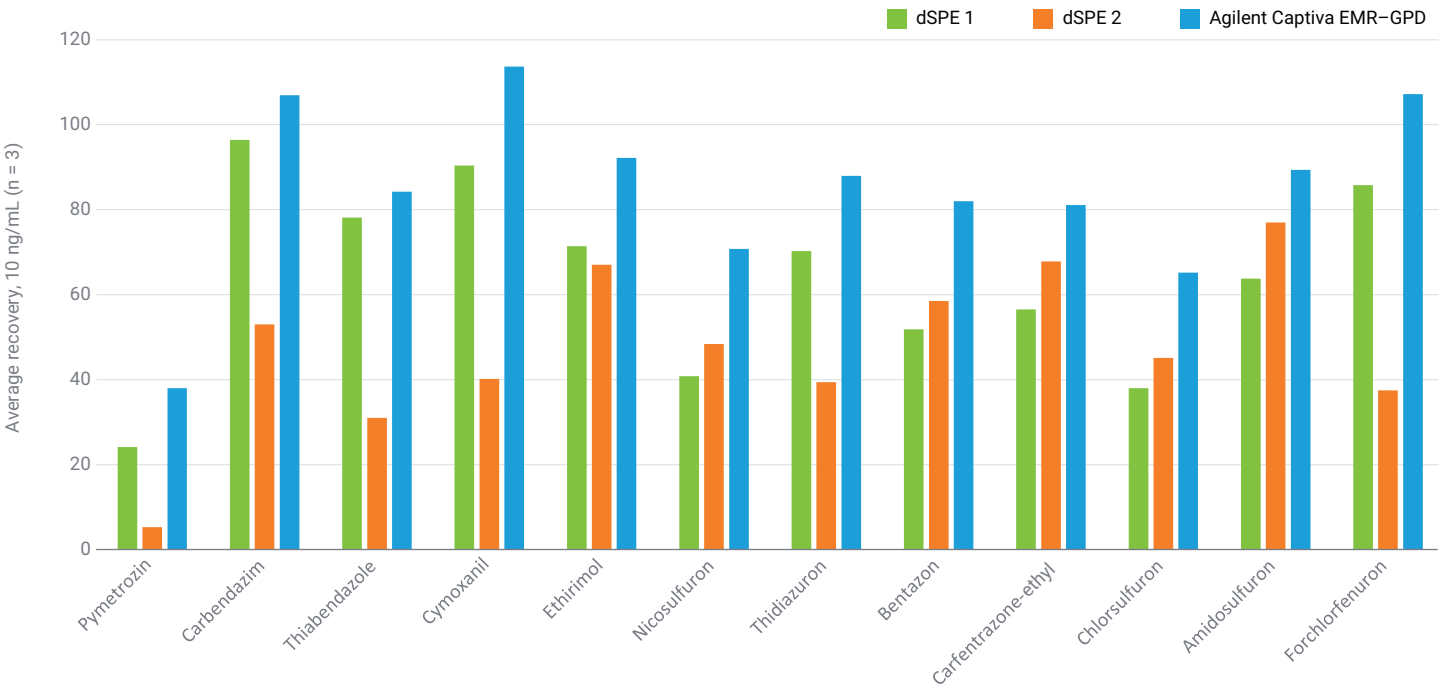

Figure 7. Sensitive pesticide recovery during cinnamon bark matrix cleanup: A comparison between Agilent Captiva EMR–GPD passthrough cleanup and two common dSPE cleanup methods.

Conclusion

A simple, rapid, and reliable method using Agilent Bond Elut QuEChERS AOAC extraction followed by Agilent Captiva EMR–GPD cartridge passthrough cleanup was developed and verified for over 300 pesticides in cinnamon bark powder by LC/MS/MS and GC/MS/MS. The novel Captiva EMR–GPD cleanup method provides convenient and simplified sample passthrough cleanup; selective and efficient matrix removal for cinnamon powder; and acceptable pesticide recovery, reproducibility, and matrix effect.

References

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5. Andrianova, A. A.; Zhao, L. Five Keys of Unlock Maximum Performance in the Analysis of Over 200 Pesticides in Challenging Food Matrices by GC/MS/MS. *Agilent Technologies application note*, 5994-4965EN, **2022**.
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7. Zhao, L.; Andrianova, A. A. Determination of Over 300 Pesticides in Cayenne Pepper Using Captiva EMR–GPD Passthrough Cleanup and LC/MS/MS and GC/MS/MS. *Agilent Technologies application note*, 5994-5630EN, **2023**.

Appendix

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
- Propoxur
- Chlorsulfuron
- Dioxacarb
- Carbofuran
- Methabenzthiazurone
- MCPA
- Amidosulfuron
- Cycluron
- Chlorotoluron
- Flutriafol
- Pyracarbolid
- Fluometurons
- Forchlorfenuron
- Carbaryl
- Fosthiazate
- Azaconazole
- Methoprotryne
- DEET
- Fenpropidin
- Carboxin
- Diuron
- 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
- Diflubenzuron
- Penconazole
- Prochloraz
- Fluoxastrobin
- Isoprothiolane
- Rotenone
- Flufenacet
- Dimoxystrobin
- Cyprodinil
- Moxidectin
- Azinphos-ethyl
- Tebufenozide
- Flubendiamide
- Beflubutamid
- Dinoseb
- Kresoxim-methyl
- Picoxystrobin
- Pyraclostrobin
- Isofenphos-methyl
- Diflufenican
- Trifloxystrobin
- Metrafenone
- Cycloate
- Metaflumizone
- Fluazinam
- Temephos
- Pyripoxyfen
- Hexythiazox
- Tralkoxydim
- Buprofezin
- Fenpyroximate
- Fenazaquin
- Proquinazid
- Pyridaben
- Spirodiclofen

GC-amenable targets

- Allidochlor
- Dichlorobenzo nitrile, 2,6-
- Biphenyl
- Mevinphos, E-
- Pebulate
- Etridiazole
- N-(2,4-dimethylphenyl) formamide
- *cis*-1,2,3,6-Tetrahydro phthalimide
- Methacrifos
- Chloroneb
- 2-Phenylphenol
- Pentachloro
- benzene
- Propachlor
- Tecnazene
- Diphenylamine
- 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

- Pentachlorobenzonitrile
- 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
- Pentachlorothioanisole
- Dichlofluanid
- Metolachlor
- Anthraquinone
- Fenthion
- Aldrin
- Chlorpyrifos
- Parathion
- Triadimefon
- Dichlorobenzophenone, 4,4'-
- DCPA
- Fenson
- Bromophos
- Diphenamid
- Pirimiphos-ethyl
- Isopropalin
- Cyprodinil
- Isodrin
- MGK-264
- Pendimethalin
- Metazachlor
- Penconazole
- Chlozolinate
- Allethrin
- Heptachlor exo-epoxide
- 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 (beta isomer)
- 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
- Tetramethrin I
- Pyridaphenthion
- Endrin ketone
- Bifenthrin
- Phosmet
- Bromopropylate
- EPN
- Methoxychlor, *p,p'-*
- Fenpropathrin
- Tebufenpyrad
- Phenothrin I
- Tetradifon
- Phosalone
- Azinphos-methyl
- Pyriproxyfen
- Leptophos
- Cyhalothrin
- Mirex
- Acrinathrin
- Fenarimol
- Pyrazophos
- Azinphos-ethyl
- Pyraclofos
- 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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