Application Note

Food & Beverage Testing

Agilent

Trusted Answers

Determination of Over 300 Pesticides in Cayenne Pepper

Using Captiva EMR–GPD pass-through cleanup and LC/MS/MS and GC/MS/MS

Authors

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Abstract

This application note presents the development and optimization of a multiresidue method for the analysis of pesticide residues in cayenne pepper powder. The method involves sample extraction with the Agilent Bond Elut QuEChERS AOAC extraction kit followed by pass-through cleanup with Agilent Captiva Enhanced Matrix Removal–General Pigment Dry (EMR–GPD) for both LC/MS/MS and GC/MS/MS analysis. The newly developed method demonstrates a convenient and simplified pass-through cleanup, providing the efficient matrix removal, acceptable target quantitation results, and high pass rate for analysis of a large panel of pesticides in a challenging cayenne pepper powder matrix. Excellent method quantitation results were achieved for over 300 LC- and GC-amenable pesticides, with 70 to 120% average recovery achieved for >92% of targets, and <20% average RSD for >97% targets in cayenne pepper. The matrix removal assessment by dried residue weight indicated that ~60% of cayenne pepper co-extractives were removed. The pass-through cleanup was also demonstrated to be a simple method, saving time and effort for analysts.

Introduction

Cayenne pepper is consumed worldwide as a common spice for its special odor and taste. The agricultural practices of cayenne pepper planting, production, and storage usually involves pesticide application for pest and disease control. The wide concern about the environmental and health impacts of pesticides means that they must comply with existing national and regulatory agencies worldwide like the European Union (EU) and Codex Alimentarius Commission (CAC).

According to SANTE guideline, dried spices are classified as difficult or unique commodities.[1] Cayenne pepper powder is one of the most difficult matrices to analyze due to its high complexity and dryness. Usually, it contains 8 to 12% water, fatty oils, essential capsaicin oil, free amino acids, other organic acids, and so on. The complicated matrix significantly challenges sample preparation for simultaneous pesticide extraction and matrix removal. The most commonly used method for sample preparation involves the use of QuEChERS or modified QuEChERS extraction, followed by either dispersive solid phase extraction (dSPE) or SPE cleanup.[2,3]

Agilent Captiva EMR General Pigmented Dry (EMR–GPD) and EMR Low Pigmented Dry (EMR–LPD) cartridges are specifically targeted to offer fast and efficient pass-through cleanup for complex dry matrices. The cartridge format design provides a convenient, fast, and simplified pass-through cleanup using an optimized blended sorbent formula. 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 provides highly selective and efficient lipid removal, while PSA provides efficient acids removal, Carbon S provides efficient pigment removal, and 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 recommended.

In this study, sample preparation using Captiva EMR–GPD cartridges for pass-through cleanup was optimized for the analysis of over 300 common pesticides in cayenne pepper using LC/MS/MS and GC/MS/MS.

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.), or as individual standard stock solutions or powder from Sigma-Aldrich (St Louis, MO, U.S.). HPLC-grade acetonitrile (ACN) was from Honeywell (Muskegon, MI, U.S.). Reagent grade acetic acid, ammonium acetate, and ammonium fluoride were also from Sigma-Aldrich.

Solutions and standards

The combined LC- and GC-standard spiking solutions, and the IS spiking solution were prepared at 10 µg/mL in 1:1 ACN/water, or ACN only, and stored at –20 °C in a freezer. The standard and IS spiking solutions were warmed up thoroughly at room temperature, sonicated before use, and returned 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 (G6490). The Agilent 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 the Agilent 8890/7000E triple quadrupole GC/MS system (GC/TQ) system. 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 Agilent purged ultimate union (PUU) and controlled by the 8890 pneumatic switching device (PSD) module. See the application note by Andrianova[4] for the 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 and environmental pollutant MRM database (P&EP 4)[5], which was used to seamlessly create the MS method. Agilent 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 Geno/Grinder (SPEX, NJ, U.S.), a Multi Reax test tube shaker (Heidolph, Schwabach, Germany), pipettes and a repeater (Eppendorf, NY, U.S.), 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 target dMRM parameters, see the application note by Zhao and Wei.[6] Table 2 lists the GC/MS/MS conditions. For the target dMRM parameters, see the P&EP 4 database (part number G9250AA).

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

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% formic acid (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% FA
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.[6]

Table 2. Agilent 8890/7000E GC/MS/MS method conditions.

GC Conditions	
Columns	Agilent J&W HP-5ms Ultra Inert, 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 10 °C/min to 310 °C 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	Agilent Inert Extractor Source with a 3 mm lens, 280 °C
Vacuum Pump	Performance turbo
Quadrupole Temperature	150 °C
Source Temperature	280 °C
Data Monitoring	Dynamic MRM mode (dMRM)
EM Voltage Gain Factor	10
Solvent Delay	3 min

Figure 1 shows typical LC/MS/MS and GC/MS/MS MRM chromatograms of targeted pesticides in the fortified cayenne pepper sample at the level of 100 ng/g in cayenne pepper, prepared by QuEChERS AOAC extraction followed by Captiva EMR–GPD cleanup.

Sample preparation

The organic cayenne pepper powder was purchased from a local grocery store. Cayenne pepper powder was weighed at 3 g into 50 mL centrifuge tubes. An aliquot of 6 mL of water was added. Samples were then vortexed for 15 minutes for complete wetting 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 pass-through cleanup. Sample elution was performed either with gravity or a low level of positive pressure (1 to 3 psi) at the consistent elution flow of

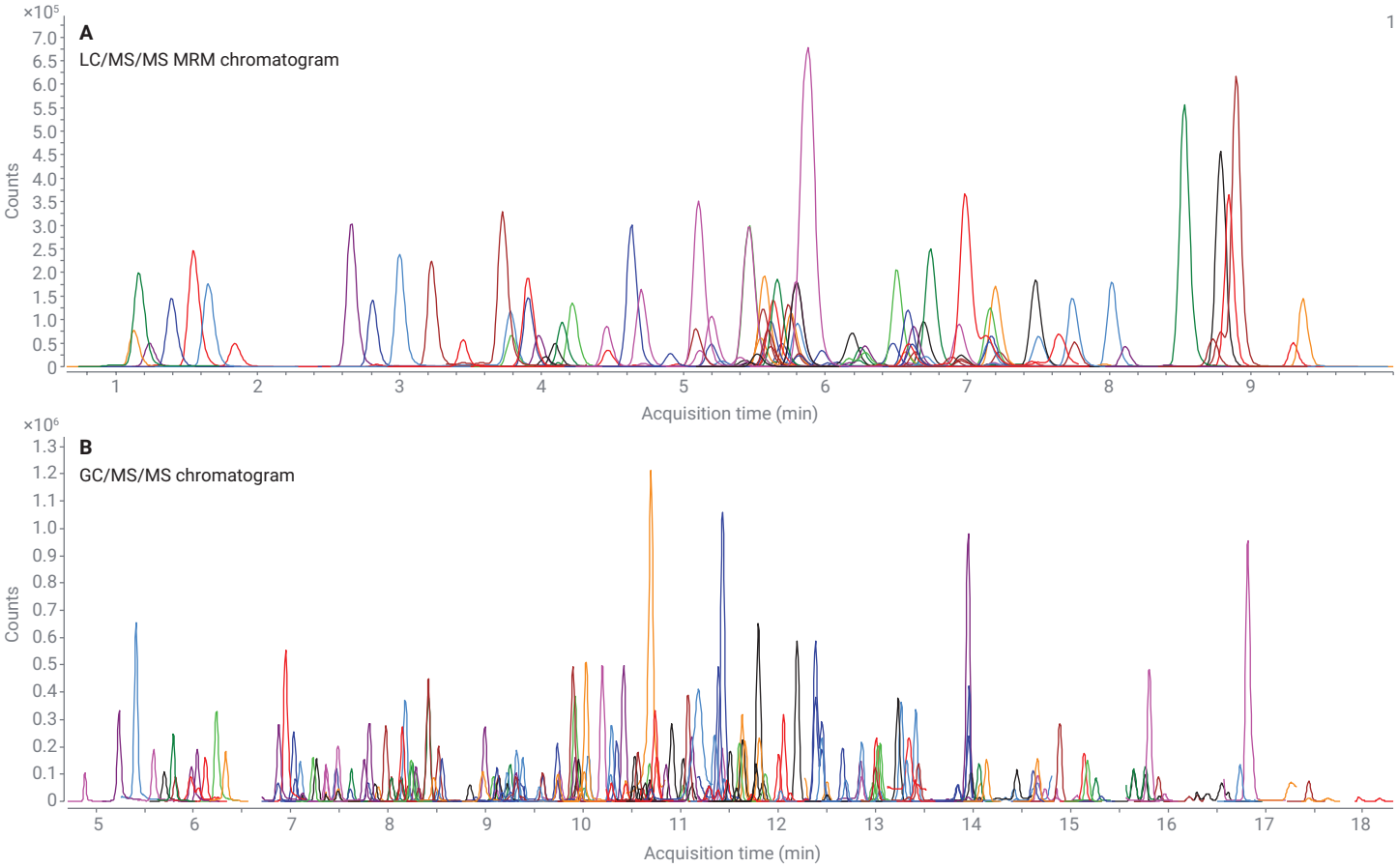

Figure 1. LC/MS/MS MRM chromatogram (A) and GC/MS/MS MRM chromatogram (B) for an extracted cayenne pepper powder sample fortified with 100 ng/g of targeted pesticides. The sample was prepared using the Agilent Bond Elut QuEChERS AOAC extraction kit, followed by Agilent Captiva EMR–GPD cleanup.

2 to 4 seconds per drop. The sample eluent was dried by anhydrous $MgSO_4$ to completely remove the water residue. For GC/MS/MS detection, an aliquot of dried sample was transferred to a 2 mL vial directly. For LC/MS/MS detection, an aliquot of 200 µL eluent was taken to mix with 800 µL of water in a 2 mL vial. The diluted sample was then ready for LC/MS/MS analysis. The detailed sample preparation procedure is shown in Figure 2. The entire sample preparation procedure resulted in 5x dilution factor from target concentration in cayenne pepper to the final cayenne pepper extract after sample extraction and matrix cleanup.

Method development

Cayenne pepper sample size and dilution factor were screened based on the study of cayenne pepper matrix complexity and co-extractive residue.

For Captiva EMR–GPD cleanup, the premixing with water was optimized by comparing the recovery results with 0, 5, and 10% water addition. Cayenne pepper crude blank extract was spiked at 10 ppb and used for the parallel comparison.

Matrix removal was assessed based on the dried residue weight of the sample, and a GC/MS analysis in full scan data acquisition mode to compare the samples with and without the Captiva EMR–GPD 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 cayenne pepper. To evaluate recovery, reproducibility, and matrix effect, prespiked quality control (PR-QC) samples were prepared at 5 and 50 ng/g in cayenne pepper, 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 for sample preparation method reproducibility RSD calculation. The peak area ratios of corresponding targets in PO-QCs versus neat QCs were used for the 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/mL in cayenne pepper matrix blank extract, corresponding to 2.5 to 2,500 ng/g in cayenne pepper. Analyte identification, confirmation, and quantitation were determined from retention times and MRM transitions.

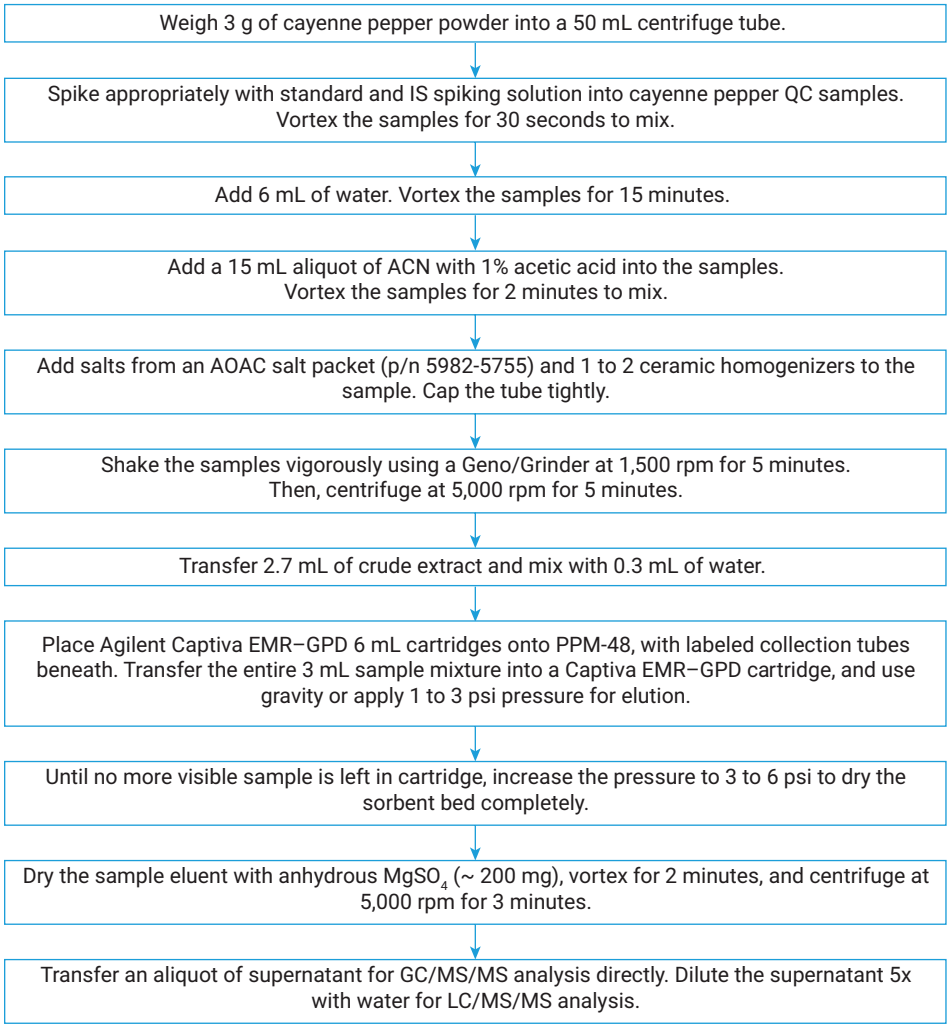

Figure 2. Sample preparation procedure for cayenne pepper samples by Agilent Bond Elut QuEChERS AOAC extraction followed by Agilent Captiva EMR–GPD pass-through cleanup.

Results and discussion

Method development and optimization

Cayenne pepper is dark red and is considered a general pigmented dry matrix; therefore, Captiva EMR–GPD is an appropriate choice for cleanup. Sample matrix was screened for preliminary matrix complexity and matrix removal efficiency using 1.5 g of cayenne pepper with 10x dilution. Figure 3A demonstrates the typical color of cayenne pepper; Figure 3B shows the crude extract after QuEChERS extraction, which is a dark orange color; and Figure 3C shows the crude extract dried residue, weighing 7 to 9 mg per 1 mL of crude extract.

Figure 3D shows the GC/MS full scan chromatographic background collected from cayenne pepper extract, where the top chromatogram is the crude extract without cleanup; the middle two chromatograms are the extract with traditional dispersive SPE (dSPE) cleanup; and the bottom chromatogram is the extract with Captiva EMR–GPD cleanup. Cayenne pepper matrix turns out to be quite fatty, and the highly abundant matrix interferences were eluted in the mid to late retention time (RT) window from 10 to 17 minutes, indicating more intermediate to nonpolar interferences (Figure 3). Specifically, the broad peak eluting between 10 to 13 minutes is likely related to the fatty acids from the sample matrix. Compared to the traditional dSPE cleanup, Captiva EMR–GPD provided significantly better matrix cleanup. The Captiva EMR–GPD cleanup also provided significantly cleaner background between the RT window of 10 to 17 minutes with almost 60% background cleanup, which was twice as clean as the background from the dSPE cleanup samples. The matrix co-extractive residue removal was also increased from ~40% for dSPE cleanup up to ~60% for EMR–GPD cleanup.

Considering the <10 mg of co-extractive residues per 1 mL of crude extract, and the high efficiency of matrix removal provided by Captiva EMR–GPD cleanup, 3 g of cayenne pepper was used throughout the sample preparation, which resulted in a 5x dilution factor.

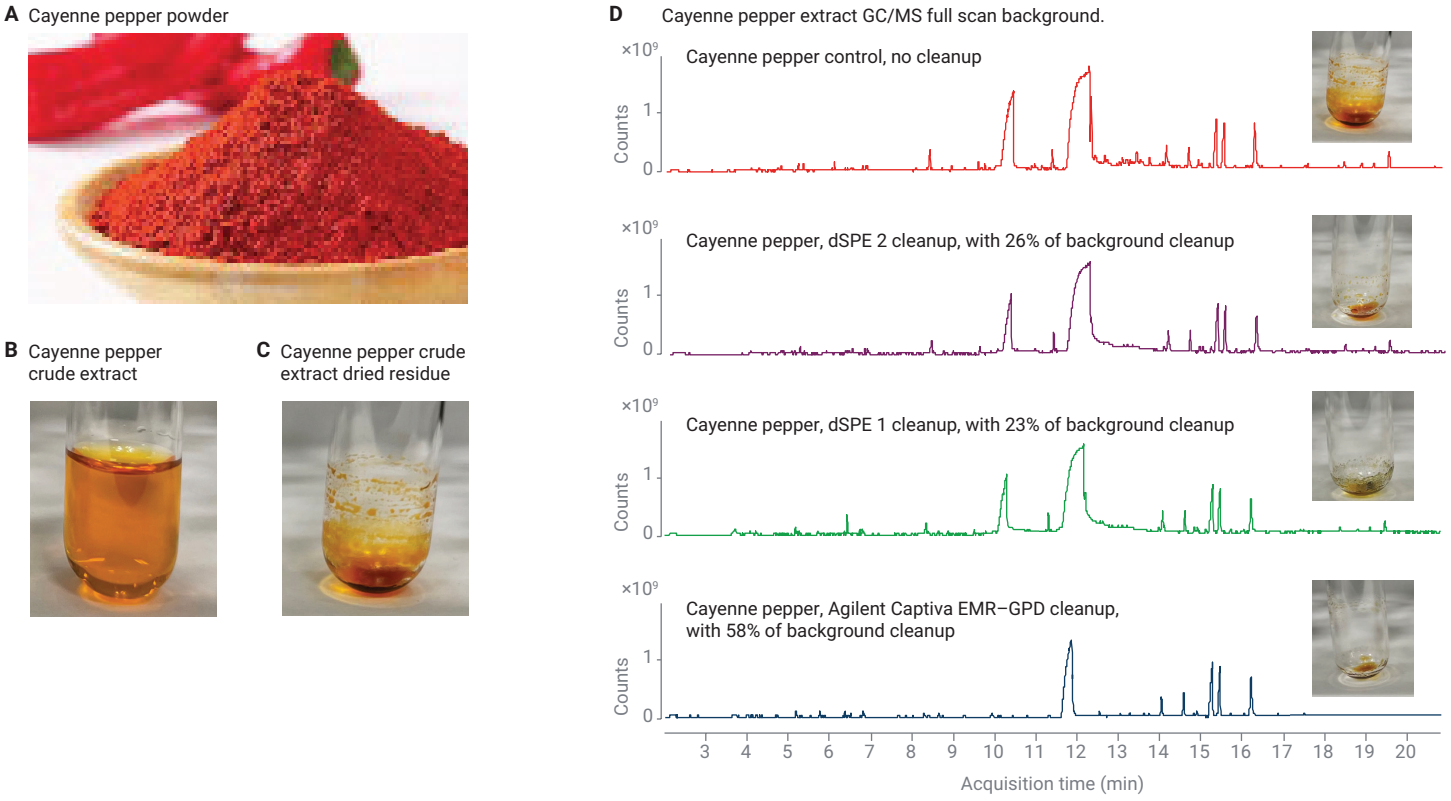

Figure 3. Preliminary study on cayenne pepper matrix. (A) Typical cayenne pepper powder; (B) crude extract after QuEChERS extraction; (C) dried residue of crude extract; (D) cayenne pepper extract GC/MS background total ion chromatogram (TIC) in full scan mode.

It was reported that the premixing of sample crude extract with water can impact analyte recoveries when using Captiva EMR–LPD for the cleanup of dry nut matrices.[7] For a sample to be loaded on EMR–GPD cartridges, the following three different ratios of water to crude cayenne pepper extract were investigated: 0:100, 5:95, and 10:90. The target recovery comparison results on either LC/MS/MS or GC/MS/MS for sensitive pesticides are shown in Figure 4. The comparison results show that:

1. The addition of water and premixing with the crude extract improved the recoveries of many sensitive targets, especially for acids and acidic targets. This is in alignment with previous findings, and can be attributed to the better buffering effect with water in the sample, as well as preventive interactions between water and PSA sorbent, which reduce the unwanted retention of acidic targets.
2. Water premixing slightly compromised the recoveries of several critical GC-amenable pesticides, such as hexachlorobenzene. Plus, the further reduced water ratio did not improve their recoveries. As a result, a 10% water premixing ratio was shown to be optimal for Captiva EMR–GPD cleanup.

Method quantitation performance assessment

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

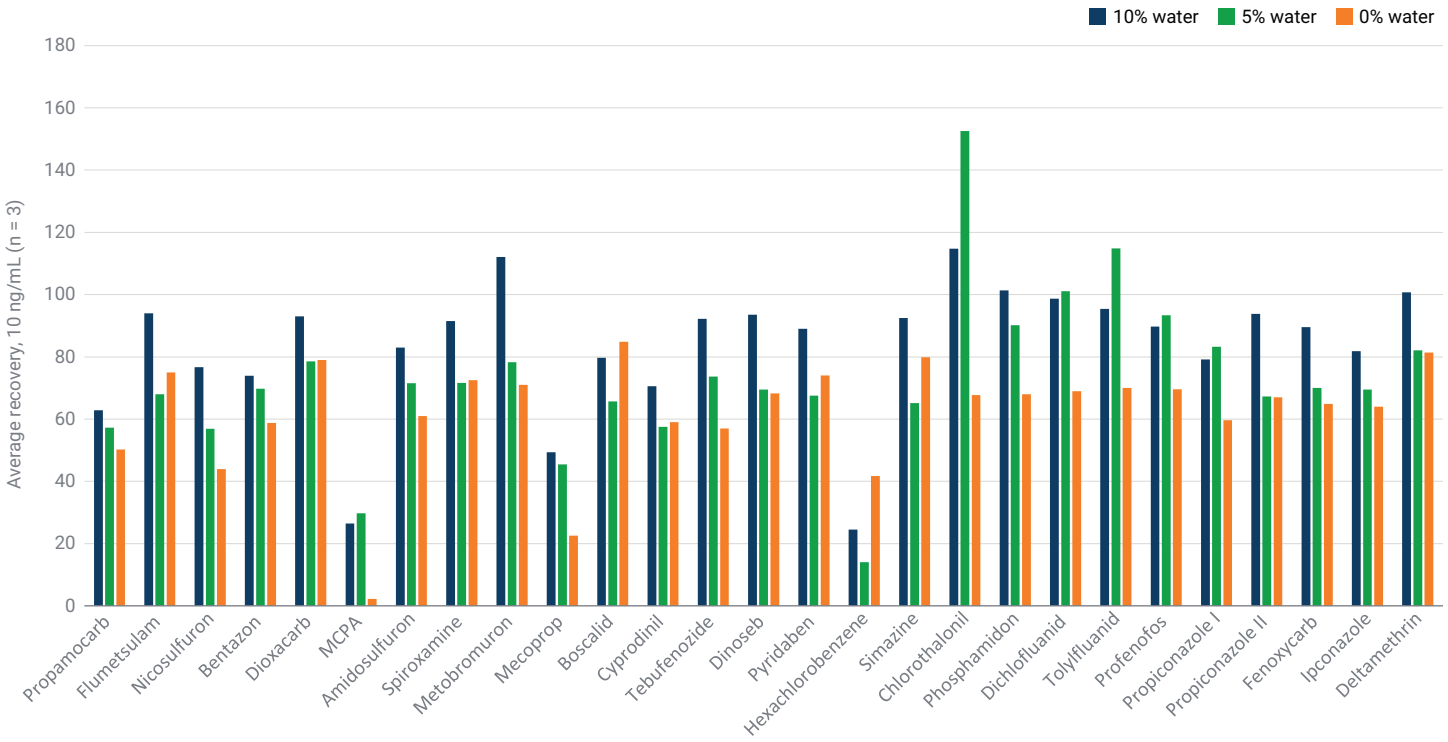

Figure 4. Optimization of water addition prior to Agilent Captiva EMR–GPD cleanup. Crude cayenne pepper extract spiked with 10 ng/mL level was used for the comparison. Representative targets were either with LC/MS/MS or GC/MS/MS detection.

Target recovery, reproducibility, and matrix effect

The above parameters are directly related to method quantitation accuracy and data quality. Therefore, it is important to use these parameters to demonstrate quantitation method performance. The SANTE/11312/2021 guideline was referred to for method performance assessment.[1] Results were calculated based on the average of 5 and 50 ng/g spiking levels, with six replicates of each level. The results show that over 92% 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. For matrix effect on LC/MS/MS, over 81% of targets were within the 60 to 130% window. Figure 5 shows the individual target results at 5 and 50 ng/g in cayenne pepper for recoveries, reproducibility (RSD), and matrix effect (LC/TQ only) with the detection of LC/MS/MS and GC/MS/MS.

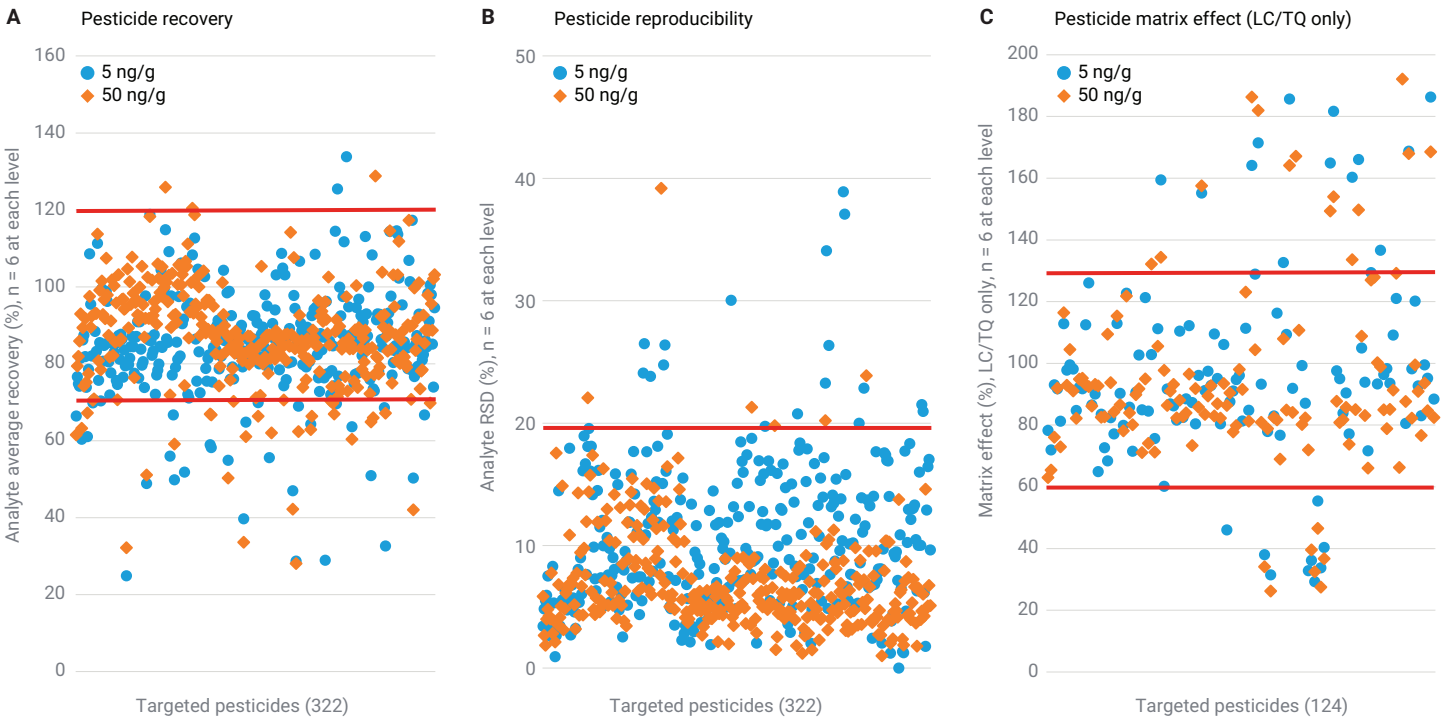

Figure 5. Method quantitation individual target results at 5 and 50 ng/g level in cayenne pepper for (A) pesticide recoveries, (B) pesticide reproducibility, and (C) pesticide matrix effect (LC/TQ only).

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 dilution factors introduced during sample extraction, this corresponded to 2.5 to 2,500 ng/g in cayenne pepper. 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 requirements and high concentration-level alignment with the calibration curve. Figure 6 shows the summary for the results of target pesticide matrix-matched calibration curves in cayenne pepper. Results show that for the total number of pesticides (>300), full dynamic calibration range (2.5 to 2,500 ng/g in cayenne pepper) with linear regression and R^2 >0.99 was achieved for 86% of targets, and full dynamic range with quadratic regression and R^2 >0.99 was achieved for approximately 6% of targets. About 8% 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 low calibration levels, or matrix positive contribution.

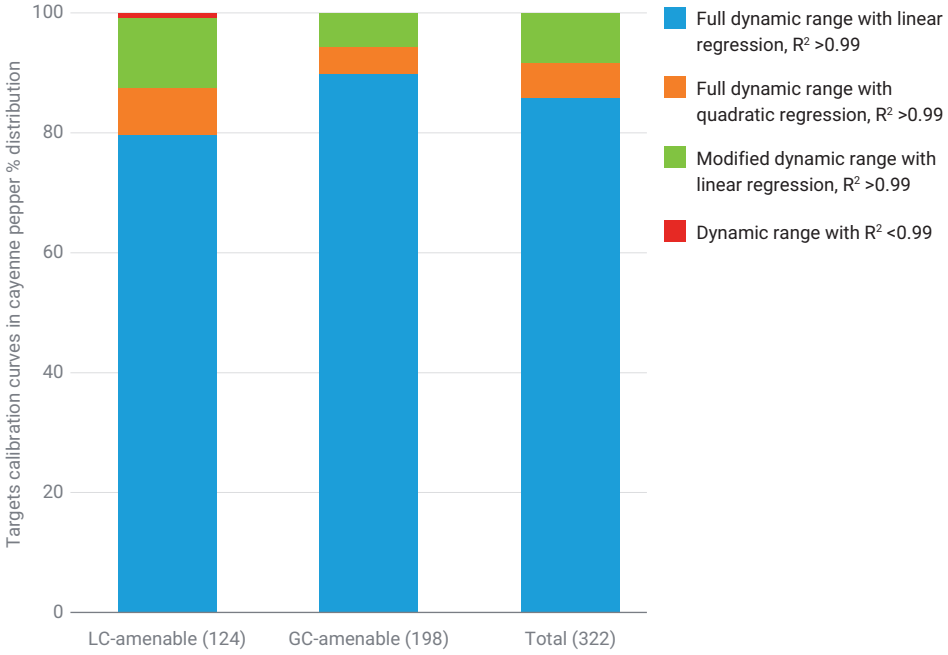

Figure 6. Results for target pesticide matrix-matched calibration curves in cayenne pepper by LC/MS/MS and GC/MS/MS detection. The full dynamic range was 2.5 to 2,500 ng/g in cayenne pepper powder.

Comparison of Captiva EMR–GPD with traditional dSPE cleanup

Compared to traditional dSPE cleanup after QuEChERS extraction, the Captiva EMR–GPD pass-through cleanup improved the matrix cleanup efficiency, as well as sensitive pesticide recoveries. When using Captiva EMR–GPD cleanup, the reduced ion suppression caused by cleaner sample matrix improved the sensitive target responses significantly on LC/MS/MS. Figure 7B shows individual chromatograms of spirodiclofen and fenproximate in cayenne pepper prepared by Captiva EMR–GPD cleanup versus two traditional dSPE cleanups. The chromatograms clearly demonstrate that 5x higher responses were achieved when using Captiva EMR–GPD cleanup. The use of Captiva EMR–GPD cleanup also improved sensitive pesticide recoveries. Figure 7A demonstrates a >30% difference in sensitive pesticide recoveries after Captiva EMR–GPD cleanup and either of the dSPE cleanups. Overall, the average recovery of LC-amenable pesticides was improved from 73% by dSPE cleanup to 87% by EMR–GPD cleanup, while the average recovery of GC-amenable pesticides was improved from 72% by dSPE cleanup to 82% by EMR–GPD cleanup.

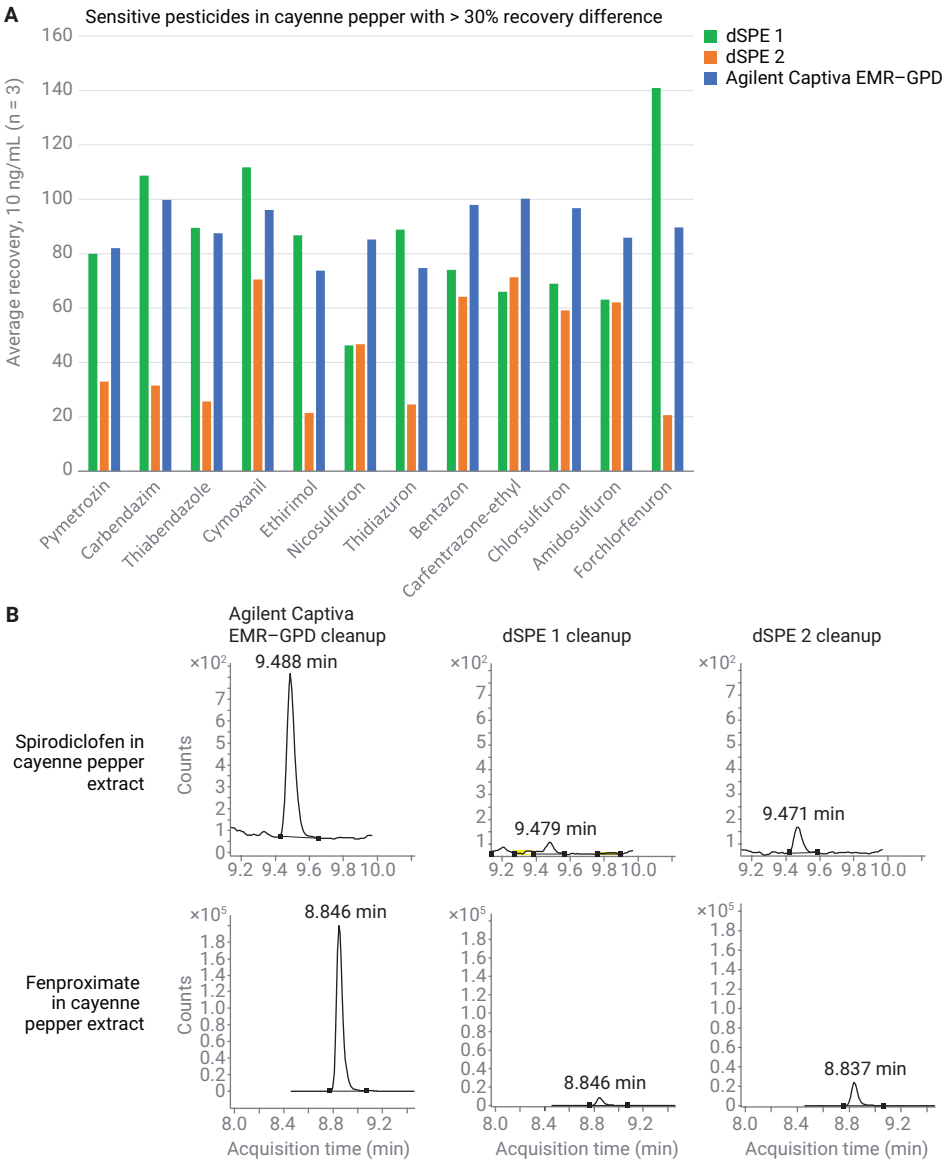

Figure 7. Cayenne pepper matrix cleanup performance comparison between Agilent Captiva EMR–GPD pass-through cleanup and two common dSPE cleanups. (A) Sensitive pesticide recovery comparison. (B) Matrix effect on the target LC/MS/MS responses for two representative pesticides, spirodiclofen and fenproximate, in cayenne pepper extract.

Conclusion

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

References

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7. Zhao, L. Determination of Multiclass, Multiresidue Pesticides in Tree Nuts by Captiva EMR–LPD Passthrough Cleanup and LC/MS/MS, *Agilent Technologies application note*, publication number 5994-5129EN, **2022**.

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
- Methabenz thiazurone
- 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
- Metaflumizone
- Fluazinam
- Temephos
- Pyripoxyfen
- Hexythiazox
- Tralkoxydim
- Buprofezin
- Fenpyroximate
- Fenazaquin
- Proquinazid
- Pyridaben
- Spirodiclofen

GC-amenable targets

- Allidochlor
- Dichlorobenzo nitrile, 2,6-
- Biphenyl
- Mevinphos, E-
- 3,4-Dichloroaniline
- 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
- 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
- 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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