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Trusted Answers

Multiclass Multiresidue Analysis for Food Safety Application Workflows

An application compendium using Agilent Captiva EMR cartridges

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		Baby food	5994-7367EN
	Mycotoxins	Infant formula	5994-0365EN
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		Tobacco	5994-5777EN
		Black tea	5994-7436EN
		Botanical dietary material	5994-7361EN
	PFAS	Dry soybeans	5994-7371EN
		Coffee powder, protein powder	5994-8610EN
	Mycotoxins	Dry corn, dry soybean	5994-7373EN
Spices	Pesticides	Black pepper	5994-4768EN
		Cayenne pepper	5994-4965EN
			5994-5630EN
		Cinnamon	5994-5671EN
		Cumin powder	5994-6882EN
Nuts, Nut Butter	Pesticides	Tree nuts	5994-5129EN
		Walnut	5994-4965EN
	Mycotoxins	Peanut butter	5994-0366EN
Edible Oil	Pesticides	Olive oil	5994-0405EN
			5994-2370EN
	PFAS	Fish oil	5994-8610EN
	PAHs	Pumpkin seed, olive, avocado, almond, grape seed oil	5994-1483EN

Category	Analyte	Matrix	Application Highlight
Fish, Shellfish	Pesticides	Salmon	5994-1717EN
	Veterinary drugs	Salmon	5994-1124EN
	PFAS	Tuna, shrimp	5994-7368EN
		Tilapia	5994-8232EN
	PAHs	Salmon	5994-0553EN
Meat	Pesticides	Pork	5994-0357EN
		Beef	5994-5061EN
	Veterinary drugs	Beef	5991-8598EN
		Pork	5994-2007EN
		Chicken, pork, beef	5994-1932EN
		Pork, beef, lamb, chicken	5994-8233EN
	PFAS	Beef	5994-7368EN
	PAHs	Beef	5994-0553EN
Edible Offal	Pesticides	Porcine liver	5994-0357EN
	Veterinary drugs	Chicken kidney, liver	5994-3680EN
	PFAS	Bovine kidney	5994-7370EN
Pet Food	Mycotoxins	Dog food, cat food	5994-7471EN
Beverages	PFAS	Orange Juice	5994-7369EN
		Beer, wine	5994-8813EN
Environmental	PFAS	Biosolids	5994-8777EN
		Soil	5994-8778EN
Confectionery	CBD, THC	Chocolate	5994-2873EN
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Preface

Selective passthough cleanup

Multiclass multiresidue food analysis requires laboratories to extract and quantify a wide range of chemically diverse analytes from complex and highly variable matrices. Sample preparation remains a major challenge, as traditional cleanup approaches often require compromises between acceptable analyte recovery and effective matrix removal. Insufficient cleanup can result in significant matrix effects that influence quantitation accuracy and precision, instrument robustness, and overall data quality, whereas overly aggressive cleanup may result in target analyte loss. Consequently, there is a critical need for practical and reproducible sample preparation strategies that achieve an optimal balance, delivering clean extracts and consistent recoveries without adding unnecessary complexity to established workflows.

Enhanced Matrix Removal (EMR) passthrough cleanup methodology was developed to address these challenges through a fundamentally different approach. By selectively removing matrix components while allowing target analytes to pass through, EMR provides cleaner extracts, improved reproducibility, and reliable performance across a wide range of food matrices and analyte classes.

This compendium highlights application-driven EMR workflows and provides a comprehensive overview of Agilent publications utilizing EMR cleanup for food safety applications. The covered applications include the analysis of pesticides, veterinary drugs, mycotoxins, PFAS, PAHs, and other contaminants across diverse matrices, ranging from fresh produce and processed foods to dry herbal materials, spices, dairy foods, animal tissues and edible offal, and edible oils. In addition to food matrices, selected applications involving environmental, cosmetic, and personal care samples are also included.

Each application note is presented with a set of highlights, summarizing critical consumables, instrument detection, sample preparation methodology, and principal results. This format enables readers to quickly and easily grasp the most important information from each application note. For those seeking additional details on the methodology and results, a direct link to the full application note is provided.

For readers seeking a deeper understanding of EMR methodology, including EMR product selection based on target analytes and food matrices, method development and validation guidance, and comparison with traditional approaches and other commercially available cleanup products, the Captiva EMR reference guide serves as a comprehensive technical resource to support successful implementation.

Multiclass Multiresidue Pesticides Analysis in Food

Introduction

Food safety laboratories are required to monitor trace-level pesticide residues across a wide range of plant- and animal-derived foods, often involving hundreds of chemically diverse compounds. Achieving reliable results at regulatory limits demands effective matrix removal without compromising analyte recovery or instrument performance.

The applications in this compendium demonstrate how Agilent EMR passthrough cleanup supports robust pesticide residue analysis by enabling cleaner extracts and consistent performance for key workflows, including:

- Multiclass multiresidue pesticide analysis across diverse food matrices
- Improved cleanup for complex, high-fat, high-pigment, and dry botanical samples
- Reliable analysis by GC/MS/MS and LC/MS/MS
- Streamlined sample preparation compatible with established QuEChERS-based extractions
- A total of 27 application notes for multiclass multiresidue pesticides analysis

Analyte – Pesticides		
Sample Category	**Sample Matrix**	**Application Highlight**
Fresh Fruits, Vegetables	Tomato	5994-2370EN
	Berries	5994-4764EN
	Spring leaf mix	5994-4765EN
	Celery	5944-4766EN
	Bell peppers	5994-4767EN
	Olives	5994-6101EN
	Spinach	5994-4965EN
		5994-4967EN
		5994-6505EN
Dairy	Milk	5994-2038EN
Dry Plant Material	Wheat powder	5994-2370EN
	Tobacco	5994-5777EN
	Black tea	5994-7436EN
	Botanical dietary material	5994-7361EN
Spices	Black pepper	5994-4768EN
	Cayenne pepper	5994-5630EN
		5994-4965EN
	Cinnamon	5994-5671EN
	Cumin powder	5994-6882EN
Nuts, Nut Butter	Tree nuts	5994-5129EN
	Walnut	5994-4965EN
Edible Oil	Olive oil	5994-0405EN
		5994-2370EN
Fish, Shellfish	Salmon	5994-1717EN
Meat	Pork	5994-0357EN
	Beef	5994-5061EN
Edible Offal	Porcine liver	5994-0357EN

Application note highlight

Determination of Multiclass, Multiresidue Pesticides in Olive Oils by Captiva EMR–Lipid Cleanup and GC/MS/MS

Application note: 5994-0405EN

Regulation or official guideline? SANTE Guideline

Method summary

Method Parameter	Setting
Analytes	26 Pesticides (GC-amenable)
Sample Matrix	Olive oil
Instrument, Detection, and Critical Consumables	– Agilent 7890B GC with an Agilent 7010A GC/TQ – Agilent HP-5ms UI, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI) – 4 mm id Ultra Inert liner single taper with wool (p/n 5190-2293)
Sample Preparation Method	1) Two-step solvent extraction using 20:80 EtOAc:ACN, 2.5 g sample for extraction 2) Passthrough cleanup on EMR–Lipid cartridges for sample crude extract with 20% of water 3) Additional elution using 80:20 ACN:water 4) Post-drying step using anhydrous $MgSO_4$
Sample Preparation Products	– Agilent Captiva EMR–Lipid cartridge, 6 mL, 600 mg (p/n 5190-1004) – Agilent Bond Elut EMR–Lipid polish pouch, 3.5 g anhydrous $MgSO_4$ (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	1 ng/g for all pesticides, except tolyfluanid (5 ng/g), captan (5 ng/g), and captafol (2.5 ng/g)
Recovery	– 70% recovery, with an average recovery of 94% – Exception: 65% for aldrin
Relative Standard Deviation	4.3% average RSD
Method Calibration	– 1 to 500 ng/g for all pesticides – Exceptions: 5 to 500 ng/g for tolyfluanid and captan, and 2.5 to 500 ng/g range for captafol – $R^2 > 0.99$
Matrix Removal or Matrix Effect	> 85% of matrix co-extractives removal by residue weight

Table 2-3. Quantitative validation results for the analysis of multiclass multiresidue pesticides in olive oil using the optimized method of LLE followed with EMR—Lipid cartridge cleanup. Accuracy < 60 % or > 120 %, or RSD > 20 % are shown in bold text.

		Calibration Curve				Accuracy and Precision					
						Low QC (n = 6) (1 or 5 ng/g)		Mid QC (n = 6) (10 ng/g)		High QC (n = 6) (100 ng/g)	
Analyte	LOQ (ng/g)	Range (ng/g)	R^2	Regression Fit	Weight	Mean Accuracy %	RSD %	Mean Accuracy %	RSD %	Mean Accuracy %	RSD %
Dichlorvos	1	1–500	0.9983	Linear	$1/x^2$	96	1.4	103	1.1	103	0.5
Trichlorfon	1	1–500	0.9946	Linear	$1/x^2$	98	9.2	96	7.4	94	3.9
2- Phenylphenol	1	1–500	0.9961	Linear	$1/x^2$	85	6.9	99	2.4	104	1.3
Ethalfluralin	1	1–500	0.9978	Linear	$1/x^2$	99	4.1	100	1.3	101	1.1
Sulfotep	1	1–500	0.9985	Linear	$1/x^2$	99	7.3	104	0.9	105	0.7
Lindane	1	1–500	0.9981	Linear	$1/x^2$	88	2.9	87	1.0	89	0.7
Diazinon	1	1–500	0.9976	Linear	$1/x^2$	83	3.6	97	0.9	100	0.6
Chlorothalonil	1	1–500	0.9979	Linear	$1/x^2$	81	8.3	99	1.7	102	0.7
Chlorpyriphos-Me	1	1–500	0.9983	Linear	$1/x^2$	90	3.4	94	1.2	95	0.8
Dichlofluanid	1	1–500	0.9986	Linear	$1/x^2$	102	6.1	100	1.1	101	0.4
Aldrin	1	1–500	0.9969	Linear	$1/x^2$	78	4.5	68	1.9	70	1.2
Tolylfluanid*	5	5–500	0.9933	Linear	$1/x^2$	103	2.9	99	4.7	99	3.3
Procymidone	1	1–500	0.9981	Linear	$1/x^2$	98	10.6	95	0.9	98	0.7
Folpet	1	1–500	0.9918	Linear	$1/x^2$	99	4.6	94	1.8	95	1.7
Endosulfan	1	1–500	0.9898	Linear	$1/x^2$	116	3.8	111	0.9	116	1.8
Bupirimate	1	1–500	0.9985	Linear	$1/x^2$	95	4.3	91	1.2	93	1.2
Endrin	1	1–500	0.9964	Linear	$1/x^2$	110	7.5	110	2.2	110	1.1
DDT	1	1–500	0.9982	Linear	$1/x^2$	91	2.2	91	0.8	94	0.5
Captan*	5	5–500	0.9932	Linear	$1/x^2$	100	6.5	96	10.9	83	4.3
Captafol*	2.5	2.5–500	0.9927	Linear	$1/x^2$	98	4.9	92	2.4	94	1.4
Iprodione	1	1–500	0.9935	Linear	$1/x^2$	101	9.5	94	3.4	90	1.5
Phosmet	1	1–500	0.9914	Linear	$1/x^2$	97	5.3	99	2.8	99	2.1
Permethrin	1	1–500	0.9971	Linear	$1/x^2$	112	2.7	113	2.0	119	2.8
Coumaphos	1	1–500	0.9880	Linear	$1/x^2$	95	3.5	97	2.1	98	0.5
Pyraclostrobin	1	1–500	0.9908	Linear	$1/x^2$	**136**	8.0	**138**	1.1	**129**	1.7
Deltamethrin	1	1–500	0.9918	Linear	$1/x^2$	101	7.8	87	5.2	90	3.0

* Raised LOQ due to matrix interference contribution or limited sensitivity.

Application highlights

- A simple, rugged, and reliable method using LLE followed by Captiva EMR–Lipid cartridge cleanup was developed and validated for the multiclass multiresidue analysis of pesticides in olive oil.
- The extraction efficiency of lipophilic pesticides from the hydrophobic oil matrix was improved using a two-step LLE with a mixture of ethyl acetate and acetonitrile.
- Captiva EMR–Lipid cartridges provided efficient and selective cleanup of olive oil matrix.
- > 96% of tested pesticides provided > 70% average recoveries, and 100% of analytes gave excellent reproducibility with < 15% average RSD.
- The optimized method provides high matrix cleanup, excellent analyte recovery and precision results for multiclass multiresidue analysis of pesticides in edible oil.
- A similar sample preparation approach was applied for determination of mycotoxins and pesticides in olives in application note 5994-6101EN, using Captiva EMR–Lipid 3 mL cartridges.

Application note highlight

Analysis of Multiclass Multiresidue Pesticides in Milk Using Agilent Captiva EMR–Lipid with LC/MS/MS and GC/MS/MS

Application note: 5994-2038EN

Regulation or official guideline? SANTE Guideline

Method summary

Method Parameter	Setting
Analytes	171 Pesticides (both GC- and LC-amenable)
Sample Matrix	Milk
Instrument, Detection, and Critical Consumables	– Agilent 8890 GC with an Agilent 7010 GC/TQ – Agilent HP-5ms UI, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI) – 4 mm id Ultra Inert liner single taper with wool (p/n 5190-2293) – Agilent 1290 Infinity II LC with an Agilent 6470A LC/TQ – Agilent ZORBAX Eclipse Plus C18, 3.0 × 150 mm, 1.8 µm (p/n 959759-302)
Sample Preparation Method	1) QuEChERS extraction using ACN with 1% AA, 5 g sample for extraction 2) Passthrough cleanup on EMR–Lipid cartridges for sample crude extract with 20% of water 3) Additional elution using 80:20 ACN:water 4) Post-drying step using anhydrous $MgSO_4$ for GC/TQ analysis, and direct injection for LC/TQ analysis
Sample Preparation Products	– Agilent Captiva EMR–Lipid, 6 mL cartridges, 600 mg (p/n 5190-1004) – Agilent QuEChERS extraction kit EN 15662 method with 50 mL tubes with ceramic homogenizers (p/n 5982-5650CH) – Agilent Bond Elut EMR–Lipid polish pouch, anhydrous $MgSO_4$ only (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	1 to 10 ng/g for all pesticides, except captan (50 ng/g)
Recovery	60 to 120% recovery for > 98% of analytes
Relative Standard Deviation	< 10% RSD for > 95% of analytes
Method Calibration	– 1 to 10 to 500 ng/g for all pesticides, except captan (50 to 500 ng/g) – R^2 > 0.990
Matrix Removal or Matrix Effect	> 99% matrix removal, by GC/MS full scan profile background comparison

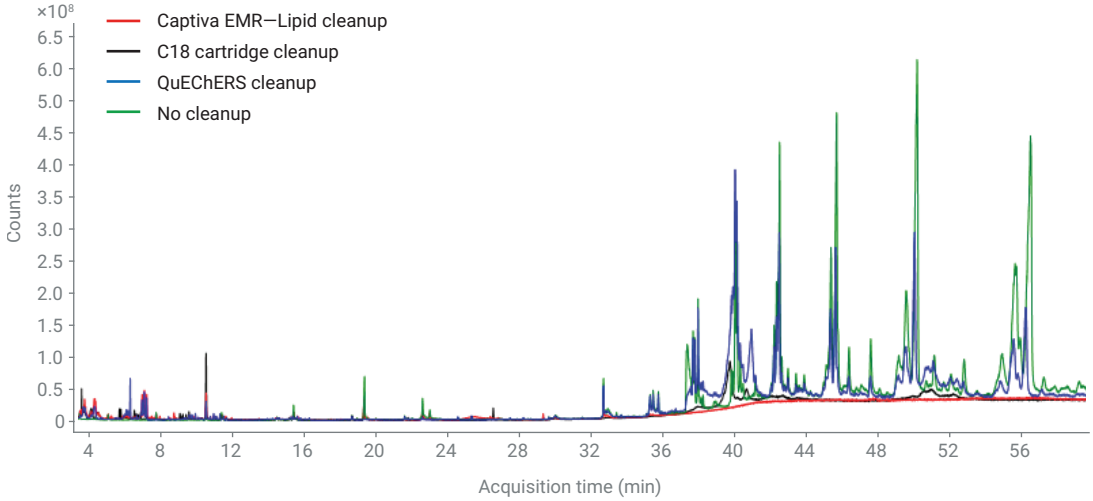


Figure 2-14. GC/MS full scan for the milk samples with different cleanup method.

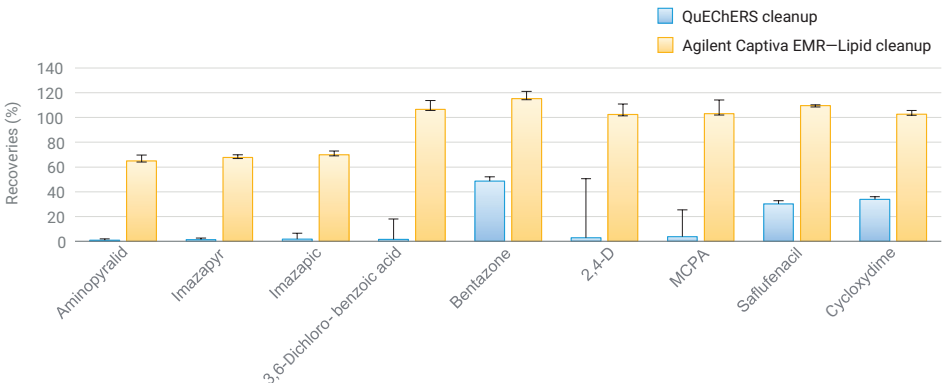


Figure 2-15. Comparison of the recoveries of acidic pesticides with Agilent QuEChERS dSPE cleanup and Agilent Captiva EMR—Lipid cleanup at 40 ng/mL spiking level by LC/MS/MS.

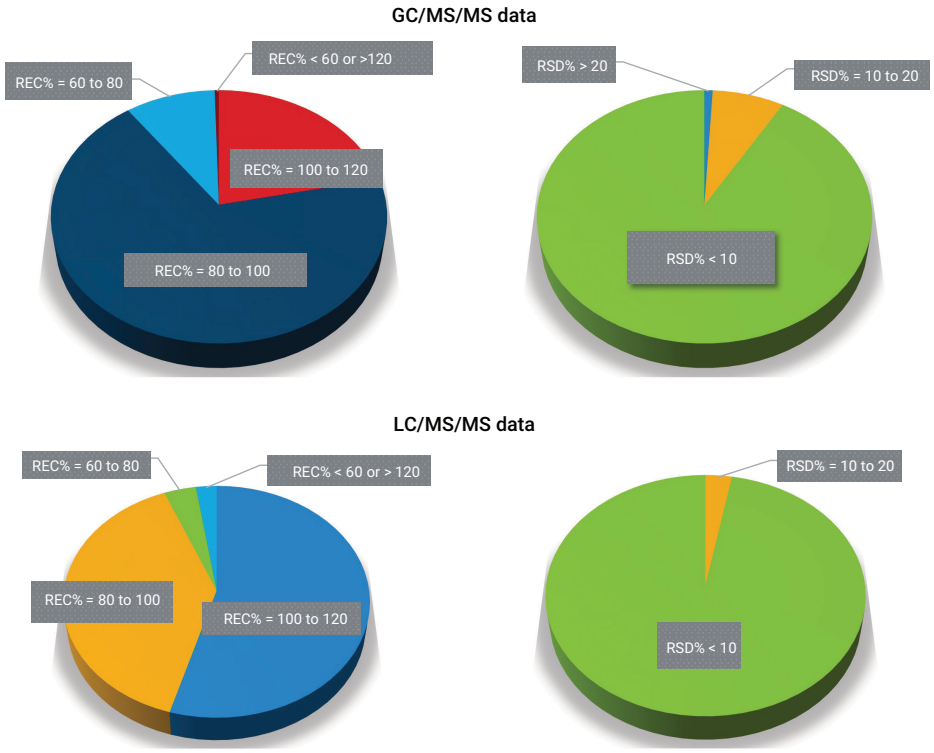


Figure 2-16. Statistical summary of the analysis of pesticides in milk method validation.

Application highlights

- A rapid, reliable, and robust workflow using QuEChERS extraction followed by Agilent Captiva EMR–Lipid cartridge cleanup was developed and verified for the analysis of 171 pesticide multiresidues in milk using LC/MS/MS and GC/MS/MS.
- Captiva EMR–Lipid delivers highly efficient removal of matrix interferences in milk and allows detection of most pesticides down to 1 ng/mL.
- For 171 pesticides studied, 98% of recoveries were within 60 to 120%, and over 95% achieve reproducibility of ≤ 10% RSD.
- A similar sample preparation approach was employed for the analysis of over 500 LC-amenable pesticides in fatty food matrix in application note 5994-2370EN, demonstrating the applicability of the method to a large panel of compounds.

Application note highlight

Analysis of Multiresidue Pesticides in Salmon Using Agilent Captiva EMR–Lipid with GC/MS/MS

Application note: 5994-1717EN

Regulation or official guideline? SANTE Guideline

Method summary

Method Parameter	Setting
Analytes	38 Pesticides (GC-amenable)
Sample Matrix	Salmon
Instrument, Detection, and Critical Consumables	– Agilent Intuvo 9000 GC with an Agilent 7010B GC/TQ – Agilent HP-5ms UI, 30 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-433UI-INT) – Agilent Ultra Inert liner, splitless, single taper, glass wool (p/n 5190-3167) – Agilent Intuvo SSL guard chip (p/n G4587-60565)
Sample Preparation Method	1) Two-step solvent extraction using 20:80 EtOAc:ACN, 2.5 g sample for extraction 2) Passthrough cleanup on EMR–Lipid cartridges for sample crude extract with 20% of water 3) Additional elution using 16:64:20 EtOAc:ACN:water 4) Post-drying step using solvent back extraction with isooctane
Sample Preparation Product	Agilent Captiva EMR–Lipid, 3 mL cartridges (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.5 µg/kg except cyhalothrin lambda and fenpropimorph (1 µg/kg)
Recovery	83 to 125% recovery
Relative Standard Deviation	< 25% RSD
Method Calibration	– 0.5 to 25 µg/kg for all pesticides – R^2 > 0.9900

Application highlights

- A method using Agilent Captiva EMR–Lipid cleanup and an Agilent Intuvo GC/MS/MS was developed and validated for the analysis of pesticides in salmon.
- Captiva EMR–Lipid cleanup provides efficient removal of major interferences such as lipids.
- 38 pesticides were determined in a 20-minute run, presenting good linearity ($R^2 \geq 0.990$) in a concentration range of 0.5 to 25 µg/kg.
- Recoveries ranged from 83% to 125% with RSD < 25%.

Application note highlight

Comparison of Different Sample Matrix Cleanup Techniques for Multiresidue Pesticide Determination in Bovine Meat Extracts

Application note: 5994-5061EN

Regulation or official guideline? SANTE Guideline

Method summary

Method Parameter	Setting
Analytes	56 Pesticides (GC-amenable)
Sample Matrix	Beef
Instrument, Detection, and Critical Consumables	– Agilent Intuvo 9000 GC with an Agilent 7010B GC/TQ – Agilent HP-5ms UI, 30 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-433UI-INT) – Agilent Ultra Inert liner, splitless, single taper, glass wool (p/n 5190-3167) – Agilent Intuvo SSL guard chip (p/n G4587-60565)
Sample Preparation Method	1) One-step solvent extraction/homogenization using ACN, 5 g sample for extraction 2) Passthrough cleanup on EMR–Lipid cartridges for sample crude extract with 20% of water 3) Post-drying step using anhydrous $MgSO_4$
Sample Preparation Product	Agilent Captiva EMR–Lipid, 3 mL cartridges (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	10 µg/kg, which is below the MRLs in bovine meat for most pesticides that are regulated by both EU and Brazil official methods
Recovery	62 to 119% recovery for all 56 pesticides
Relative Standard Deviation	< 16% RSD
Matrix Removal or Matrix Effect	Compared to other matrix cleanup methods, Captiva EMR–Lipid cleanup provides the best balance on matrix cleanup and target recovery

Application highlights

- Agilent Captiva EMR–Lipid cleanup was demonstrated to be a superior cleanup method to the Agilent Bond Elut NH2 and C18 methods, as verified in bovine meat matrix.
- The efficient sample cleanup method can also be beneficial to reduce GC/MS/MS system maintenance frequency, increase column and consumable lifetimes, and deliver reliable quantification results.
- Overall recoveries of 56 pesticide residues ranged from 62 to 119% with RSD ≤16%.
- Method LOQs meet most EU and Brazil MRLs.
- A similar sample preparation approach was employed for the analysis of formamidine pesticides and metabolites in pork and porcine liver, as demonstrated in application note 5994-0357EN. This work was in compliance with EU regulation.

Application note highlight

Determination of Multiclass, Multiresidue Pesticides in Berries

Application note: 5994-4764EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	108 Pesticides (GC-amenable)
Sample Matrix	Berries (blackberry, blueberry, raspberry)
Instrument, Detection, and Critical Consumables	– Agilent 8890 GC with an Agilent 7000D GC/TQ – Agilent HP-5ms UI, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI) – 4 mm id Ultra Inert liner single taper with wool (p/n 5190-2293)
Sample Preparation Method	1) QuEChERS EN extraction using ACN with 1% AA, 10 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–GPF cartridges for sample crude extract 3) Post-drying step using anhydrous $MgSO_4$
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit (p/n 5982-5650CH) – Agilent Captiva EMR–GPF, 3 mL (p/n 5610-2090) – Agilent Bond Elut EMR–Lipid polish pouch (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	1 to 10 ng/g for all pesticides
Recovery	– 60 to 120% recovery for > 96% of pesticides – Failure rate of pesticides recovery was 3.7%, 6.5%, and 1.8% in blackberry, blueberry, and raspberry, respectively
Relative Standard Deviation	< 20% RSD
Method Calibration	– 1 to 10 to 500 ng/g for all pesticides – Exception: 50 to 500 ng/g calibration range for malathion – R^2 > 0.99
Matrix Removal or Matrix Effect	> 99% pigment removal

Application highlights

- Developed and validated a fast, reliable method for analyzing 108 GC-amenable pesticides in berries using Agilent Bond Elut QuEChERS EN extraction and Captiva EMR–GPF cleanup with GC/MS/MS.
- Captiva EMR–GPF cartridges offer simplified passthrough cleanup, superior pigment removal, better target recovery, and improved reproducibility over traditional dSPE.
- Quantitation showed > 93% pass rate in blueberry and > 99% in blackberry and raspberry for recovery, RSD, and linearity.
- Cleanup yielded colorless extracts with > 99% UV absorbance reduction, confirming effective pigment removal.

Application note highlight

Determination of Multiclass, Multiresidue Pesticides in Spring Leaf Mix

Application note: 5994-4765EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	138 Pesticides (LC-amenable)
Sample Matrix	Spring leaf mix
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6490 LC/TQ – Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959753-902) – Agilent ZORBAX Eclipse Plus C18, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901)
Sample Preparation Method	1) QuEChERS AOAC extraction using ACN with 1% AA, 15 g sample for extracton 2) Mixed-mode passthrough cleanup on EMR–HCF1 or EMR–HCF2 cartridges for sample crude extract 3) Post-treatment by further dilution with water
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–HCF1, 3 mL (p/n 5610-2088) – Agilent Captiva EMR–HCF2, 3 mL (p/n 5610-2089)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.5 to 10 ng/g for all pesticides
Recovery	– 70 to 120% recovery for > 96% of pesticides – 50 to 70% recovery for acidic pesticides, including 2,4-D, 2,4,5-TP, MCPA, 2,4,5-T, dichlorprop, and mecoprop
Relative Standard Deviation	< 20% RSD
Method Calibration	– 0.5 to 10 to 500 ng/g for all pesticides – R^2 > 0.99
Matrix Removal or Matrix Effect	98% pigment removal by EMR–HCF1, and 97% pigment removal by EMR–HCF2

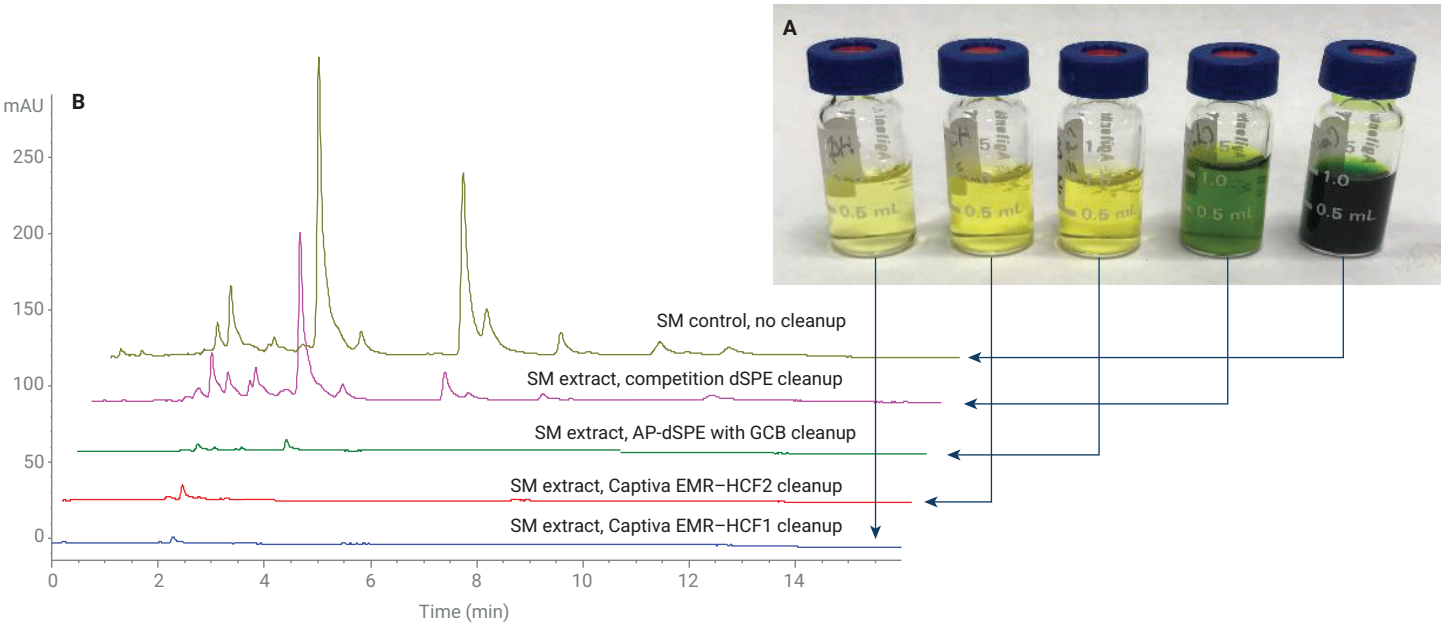


Figure 2-17. Spring mix (SM) matrix sample pigment removal efficiency demonstration. (A) Extracted samples color comparison. (B) LC/UV (λ = 450 nm) stacked chromatograms for extracted spring mix samples.

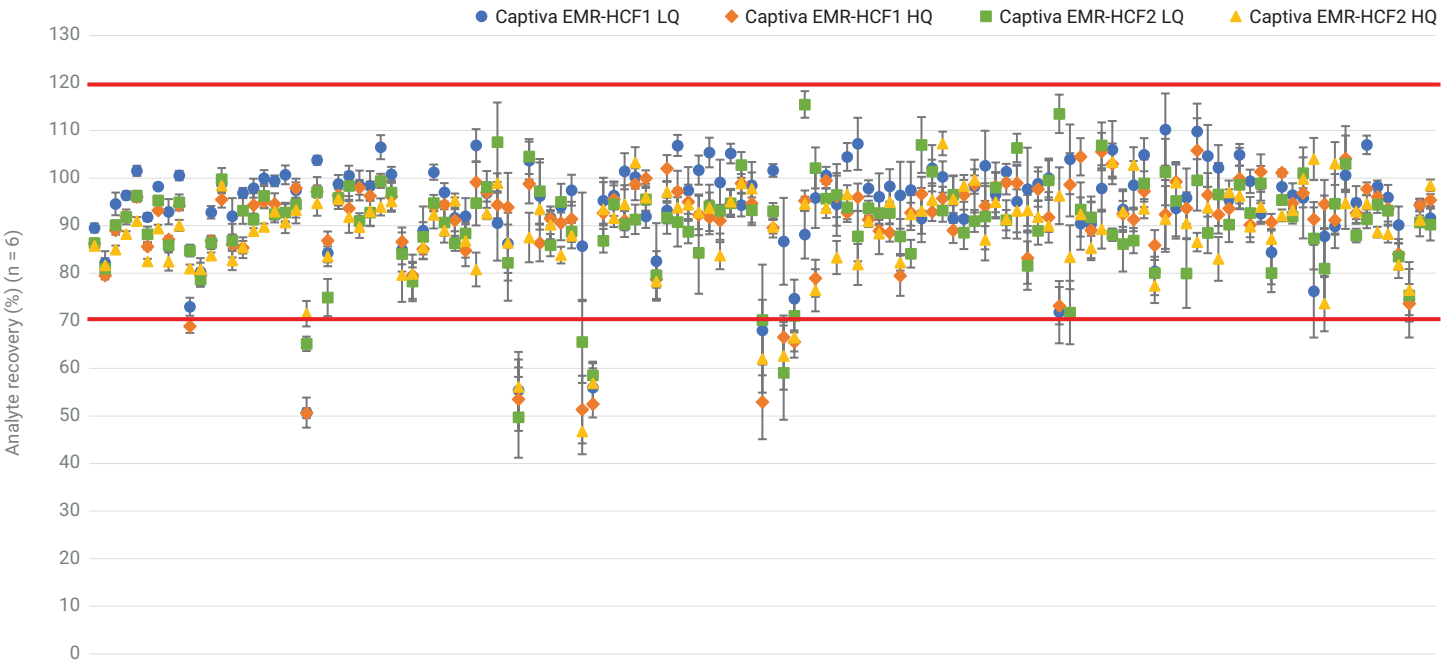


Figure 2-18. Target accuracy and precision results in spring mix. Two levels of prespiking include 10 ng/g for LQ and 100 ng/g for HQ. Samples were prepared using Agilent Bond Elut QuEChERS AOAC extraction kit followed by Agilent Captiva EMR–HCF1 and Captiva EMR–HCF2 cleanup, respectively.

Application highlights

- Two simple, rapid, and reliable methods using Agilent Bond Elut QuEChERS AOAC extraction, followed by either Agilent Captiva EMR–HCF1 or Captiva EMR–HCF2 cartridge passthrough cleanup, were developed and verified for 138 LC-amenable pesticides in spring mix by LC/MS/MS.
- Two types of Captiva EMR–HCF (EMR–HCF1 with NH2 and EMR–HCF2 with PSA) were designed for high-chlorophyll leafy vegetable matrix with optimized formula and bed mass.
- Methods provided highly efficient chlorophyll pigment removal and significantly reduced unwanted interactions with targets, especially for sensitive compounds such as planar pesticides.
- Pigment removal assessment by LC/UV confirmed that > 96% of green/yellow pigment interferences were removed by the EMR–HCF cleanup.
- Over 96% of the pesticides were identified with 70 to 120% recovery, RSD < 20%, and calibration curves with R^2 > 0.99, within the calibration range.
- The same sample preparation approach for the analysis of GC-amenable pesticides in spinach was employed in the demonstration of a fast and robust 10-minute method on Agilent 7000E and 7010C GC/MS/MS systems in application note 5994-4967EN.
- The same sample preparation approach was further employed for hydrogen carrier gas for analyzing pesticides in high-chlorophyll leafy vegetables using an Agilent HydroInert source on an Agilent 7000E GC/TQ system, as shown in application note 5994-6505EN.
- The QuEChERS extraction followed with EMR mixed-mode passthrough cleanup using Captiva EMR with Carbon S approach was demonstrated as one of five keys to unlock maximum performance in the analysis of over 200 pesticides in challenging foods by GC/MS/MS in application note 5994-4965EN.

Application note highlight

Determination of Multiclass, Multiresidue Pesticides in Bell Peppers

Application note: 5994-4767EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	240 Pesticides (LC- and GC-amenable)
Sample Matrix	Bell peppers
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6490 LC/TQ – Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959753-902) – Agilent ZORBAX Eclipse Plus C18, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901) – Agilent 8890 GC with an Agilent 7000D GC/TQ – Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI) – Agilent Ultra Inert liner single taper with wool (p/n 5190-2293)
Sample Preparation Method	1) QuEChERS AOAC extraction using ACN with 1% AA, 15 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–GPF 3) One sample preparation method for both LC/TQ and GC/TQ analysis
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–GPF, 3 mL (p/n 5610-2090) – Agilent Bond Elut EMR–Lipid polish pouch (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– LC/TQ: 0.5 ng/g – GC/TQ: 1 ng/g
Recovery	– 70 to 120% recovery for > 98% of pesticides – Exceptions: low recovery for quinmerac, bifenazate, and dichlofluanid
Relative Standard Deviation	– < 20% RSD for 99% of targets – Exceptions: higher RSDs for quinmerac and 2,4-D
Method Calibration	– LC/TQ: 0.5 to 500 ng/g – GC/TQ: 1 to 500 ng/g – $R^2 > 0.99$ – Exceptions: $0.98 < R^2 < 0.99$ for dimethomorph I, spinosad D, prochloraz, moxidectin, metribuzin, malathion, triadimefon, zoxamide, azamethiphos, coumaphos, and pyraclostrobin
Matrix Removal or Matrix Effect	> 95% pigment removal, but UV detection

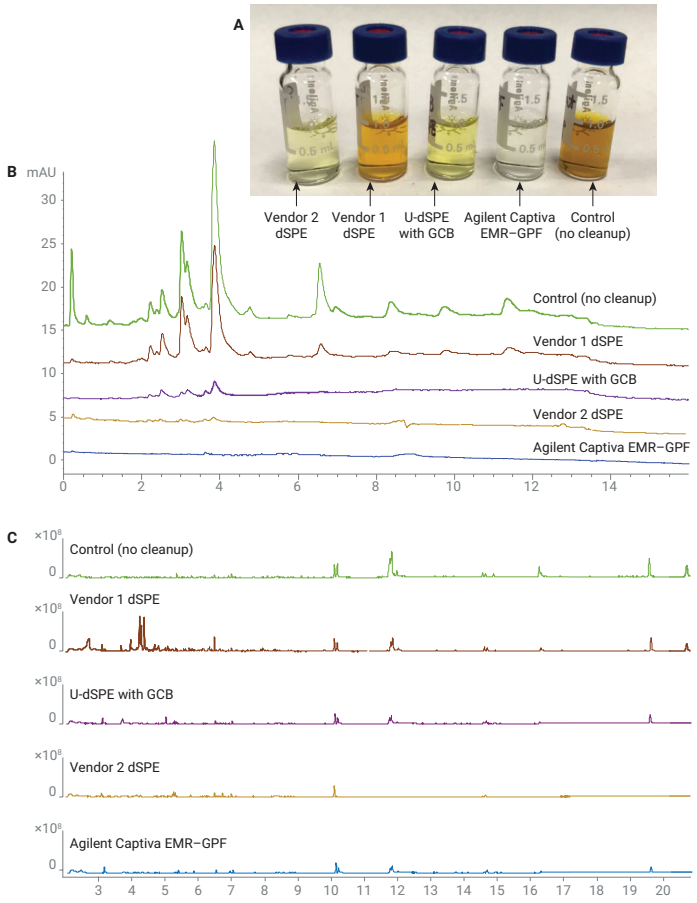


Figure 2-19. Mixed bell pepper matrix sample cleanliness evaluation. (A) Extracted samples color comparison. (B) LC/UV (λ = 450 nm) stacked chromatograms for samples' UV adsorption. (C) GC/MS full scan background chromatogram.

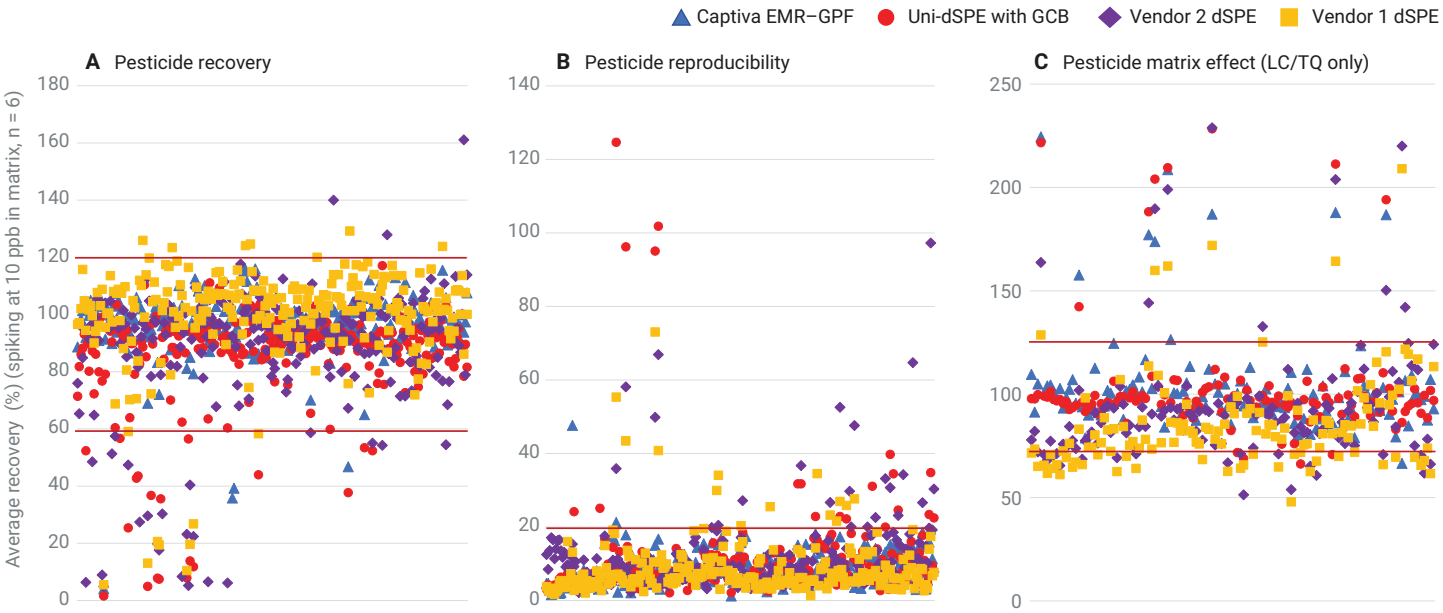


Figure 2-20. Quantitative analysis of 230 pesticides in mixed bell pepper at 10 ng/g fortification level (n = 6) based on (A) targets recovery, (B) targets reproducibility (RSD%), and (C) matrix effect (LC/TQ only), using different cleanup methods.

Application highlights

- A fast, simple, and validated method was developed for analyzing 230 LC- and GC-amenable pesticides in mixed bell pepper using LC/MS/MS and GC/MS/MS.
- The workflow uses the Agilent Bond Elut QuEChERS AOAC extraction kit followed by Captiva EMR–GPF passthrough cleanup.
- Compared to traditional dSPE with GCB and two other vendor dSPE cleanups, this method offers a more convenient and streamlined sample preparation.
- It provides selective and efficient pigment and matrix removal, improving target recovery and reproducibility.
- The method reduces matrix effects and interferences, enhancing overall data quality.
- As a result, it improves quantitation pass rates for large pesticide panels in fresh produce matrices.
- The same sample preparation method was applied for pesticides analysis in celery in application note 5944-4766EN.

Application note highlight

Analysis of 510 Pesticides in Black Pepper

Application note: 5994-4768EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	510 Pesticides (LC-amenable)
Sample Matrix	Black pepper
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6470B LC/TQ – Agilent ZORBAX RRHD Eclipse Plus C18 column, 2.1 × 150 mm, 1.8 µm (p/n 959759-902)
Sample Preparation Method	1) QuEChERS EN extraction using ACN with 1% AA, 10 g sample for extraction 2) Mixed-mode passthrough cleanup on consecutive EMR–GPD and EMR–GPF cartridges
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN kit (p/n 5982-5650CH) – Agilent Captiva EMR–GPD cartridge (p/n 5610-2091) – Agilent Captiva EMR–GPF (EMR–GPF) cartridge (p/n 5610-2090)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	10 to 25 µg/kg, meeting MRL requirements for compounds in pepper matrix established in the EU pesticides database
Recovery	– 40 to 120% recovery for 75% of pesticides – Failure rate of pesticides recovery was 3.7%, 6.5%, and 1.8% in blackberry, blueberry, and raspberry, respectively
Relative Standard Deviation	< 20% RSD
Method Calibration	– 0.25 to 100 µg/L in black pepper extract – R^2 > 0.99 for > 85% of targets
Matrix Removal or Matrix Effect	– 85% of analytes with matrix effect of 40 to 120% – Improved matrix cleanup efficiency compared to traditional techniques

Application highlights

- The workflow enables selective and sensitive quantitation of 510 pesticide residues in black pepper.
- Sample preparation using Bond Elut QuEChERS EN extraction and Captiva EMR–GPD/GPF cleanup effectively removes matrix interferences and reduces matrix effects.
- The method yields acceptable quantitation for 75% of pesticides, outperforming traditional preparation techniques.
- Cleaner extracts reduce LC/MS contamination and carryover, lowering maintenance needs and boosting long-term robustness.
- Sub-1 and 10 ng/mL LODs were achieved for 81% and 97% of targets, meeting EU MRLs for pepper.
- The workflow shows high reproducibility and potential for use with other complex dry food matrices rich in pigment and fat.

Application note highlight

Determination of Multiclass, Multiresidue Pesticides in Tree Nuts by Captiva EMR–LPD Passthrough Cleanup and LC/MS/MS

Application note: 5994-5129EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	125 Pesticides (LC-amenable)
Sample Matrix	Tree nuts (almonds, pecans, cashews, and hazelnuts)
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6490 LC/TQ – Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959753-902) – Agilent ZORBAX Eclipse Plus C18, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901)
Sample Preparation Method	1) QuEChERS AOAC extraction using ACN with 1% AA, 3 to 7.5 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–LPD
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–LPD, 6 mL (p/n 5610-2092)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– 1 ng/g in almond, pecan, and cashew – 2.5 ng/g in hazelnut
Recovery	– > 90% average recovery – < 5% failure rate on recovery, RSD, or matrix effect in four types of tree nuts
Relative Standard Deviation	< 10% RSD
Method Calibration	– 1 to 1,000 ng/g in almond, pecan, and cashew – 2.5 to 2,500 ng/g in hazelnut – R^2 > 0.98 for all targets
Matrix Removal or Matrix Effect	– > 64% of matrix removal based on matrix residue removal – > 56% matrix background cleanup based on GC/MS full scan

Application highlights

- A simple and rapid method was developed for analyzing 125 LC-amenable pesticides in tree nuts using LC/MS/MS.
- The workflow uses Agilent Bond Elut QuEChERS AOAC extraction followed by Captiva EMR–LPD passthrough cleanup.
- This method simplifies sample preparation while efficiently removing matrix interferences from fatty tree nuts.
- It delivers acceptable pesticide recovery, reproducibility, and matrix effect, with over 95% of targets meeting SANTE guidelines.
- Compared to Captiva EMR–Lipid cleanup, the EMR–LPD cartridge achieves more complete lipid removal, especially in fatty acid-rich matrices.
- Acidified ACN extraction with 10% water addition prevents loss of sensitive pesticides by minimizing unwanted sorbent interactions.
- The streamlined workflow saves time and effort, enhancing lab productivity and long-term performance.
- The sample preparation method was applied to walnut as demonstration of a new GC/MS/MS workflow for pesticides analysis in application note 5994-4965EN.

Application note highlight

Determination of Over 300 Pesticides in Cayenne Pepper

Application note: 5994-5630EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	> 300 Pesticides (LC- and GC-amenable)
Sample Matrix	Cayenne pepper
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6490 LC/TQ – Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959753-902) – Agilent ZORBAX Eclipse Plus C18, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901) – Agilent 8890 GC with an Agilent 7000E GC/TQ – Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI-KEY) – Agilent Ultra Inert 2 mm dimpled liner (p/n 5190-2297)
Sample Preparation Method	1) QuEChERS AOAC extraction using ACN with 1% AA, 3 g sample for extracton 2) Mixed-mode passthrough cleanup on EMR–GPD 3) One sample preparation for both LC/TQ and GC/TQ analysis
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–GPD, 6 mL (p/n 5610-2091) – Agilent Bond Elut EMR–Lipid polish pouch (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	2.5 ng/g for all pesticides
Recovery	70 to 120% average recovery achieved for > 92% of targets
Relative Standard Deviation	< 20% average RSD for > 97% targets in cayenne pepper
Method Calibration	– 2.5 to 2,500 ng/g for all pesticides – R^2 > 0.99 was achieved for 86% of targets for full dynamic range with linear regression – R^2 > 0.99 was achieved for 6% of targets for full dynamic range with quadratic regression – R^2 > 0.99 was achieved for 8% of targets using a modified range with either linear or quadratic regression due to either the lack of sensitivity or selectivity at low calibration levels or matrix positive contribution
Matrix Removal or Matrix Effect	~ 60% of cayenne pepper co-extractives were removed

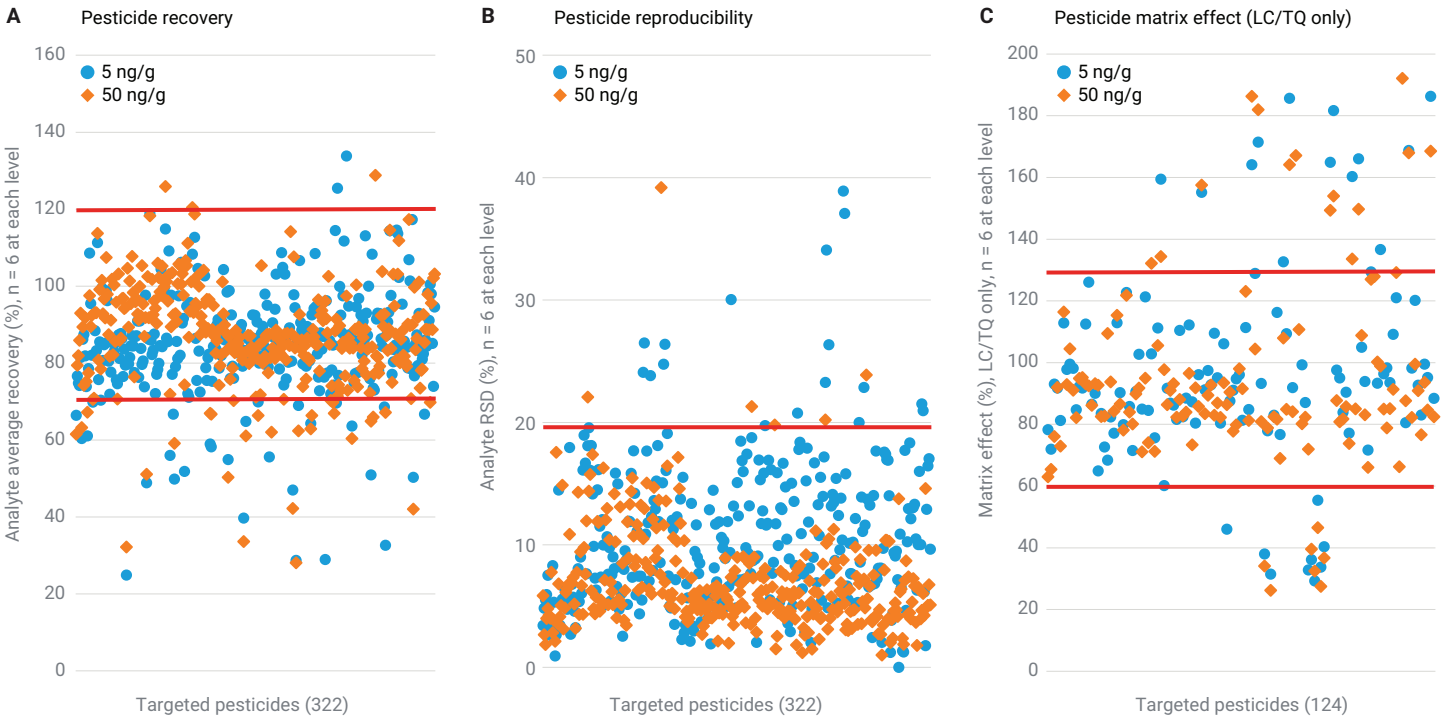


Figure 2-21. Method quantitation individual target results at 5 and 50 ng/g level in cayenne pepper for (A) pesticide recoveries, (B) pesticide reproducibility, and (C) pesticide matrix effect (LC/TQ only).

Application highlights

- A simple, rapid, and reliable method using Agilent Bond Elut QuEChERS AOAC extraction followed by Agilent Captiva Enhanced Matrix Removal–General Pigment Dry (EMR–GPD) cartridge passthrough cleanup was developed and verified for over 300 pesticides in cayenne pepper by LC/MS/MS and GC/MS/MS.
- The method provides convenient and simplified sample passthrough cleanup, selective and efficient matrix removal for cayenne pepper powder, and acceptable pesticide recovery, reproducibility, and matrix effect.
- 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 sample preparation method was applied to cayenne pepper powder as demonstration of a new GC/MS/MS workflow for pesticides analysis in application note 5994-4965EN.

Application note highlight

Determination of Over 300 Pesticides in Cinnamon

Application note: 5994-5671EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	> 300 Pesticides (LC- and GC-amenable)
Sample Matrix	Cinnamon
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6490 LC/TQ – Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959753-902) – Agilent ZORBAX Eclipse Plus C18, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901) – Agilent 8890 GC with an Agilent 7000E GC/TQ – Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI-KEY) – Agilent Ultra Inert 2 mm dimpled liner (p/n 5190-2297)
Sample Preparation Method	1) QuEChERS AOAC extraction using ACN with 1% AA, 1.5 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–GPD 3) One sample preparation method for both LC/TQ and GC/TQ analysis
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–GPD, 6 mL (p/n 5610-2091) – Agilent Bond Elut EMR–Lipid polish pouch (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	5 ng/g
Recovery	70 to 120% average recovery achieved for > 95% of targets
Relative Standard Deviation	< 20% average RSD for > 97% of targets in cinnamon
Method Calibration	– 5 to 5,000 ng/g in cinnamon – R^2 > 0.99 for 88% of targets over full dynamic range with linear regression – R^2 > 0.99 for 6% of targets over full dynamic range with quadratic regression – R^2 > 0.99 for 6% of targets over modified dynamic range with either linear or quadratic regression
Matrix Removal or Matrix Effect	~ 60% of cinnamon co-extractives were removed according to matrix removal assessment by dried residue weight

Application highlights

- A fast and reliable method was developed for analyzing over 300 pesticides in cinnamon bark powder using LC/MS/MS and GC/MS/MS.
- The workflow uses Agilent Bond Elut QuEChERS AOAC extraction followed by Captiva EMR–GPD passthrough cleanup.
- This method simplifies sample preparation while effectively removing complex matrix interferences from cinnamon powder.
- It delivers acceptable pesticide recovery, reproducibility, and matrix effect, ensuring reliable quantitation.
- The Captiva EMR–GPD cleanup offers a convenient and efficient solution for challenging spice matrices.

Application note highlight

Determination of Over 300 Pesticides in Tobacco

Application note: 5994-5777EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	> 300 Pesticides (LC- and GC-amenable)
Sample Matrix	Tobacco
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with an Agilent 6490 LC/TQ – Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959753-902) – Agilent ZORBAX Eclipse Plus C18, UHPLC guard, 2.1 × 5 mm, 1.8 µm (p/n 821725-901) – Agilent 8890 GC with an Agilent 7000E GC/TQ – Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI-KEY) – Agilent Ultra Inert 2 mm dimpled liner (p/n 5190-2297)
Sample Preparation Method	1) QuEChERS EN extraction using ACN with 1% AA, 1 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–LPD 3) One sample preparation for both LC/TQ and GC/TQ analysis
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–LPD, 6 mL (p/n 5610-2092) – Agilent Bond Elut EMR–Lipid polish pouch (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	10 ng/g for all pesticides
Recovery	70 to 120% average recovery achieved for > 95% of targets
Relative Standard Deviation	< 20% average RSD for > 98% targets in tobacco
Method Calibration	– 10 to 5,000 ng/g in tobacco – R^2 > 0.99 for 84% of targets using full dynamic calibration range with linear regression – R^2 > 0.99 for 4% of targets using full dynamic range with quadratic regression – R^2 > 0.99 for 34 out of 325 targets using a modified range with either linear or quadratic regression due to either the lack of sensitivity or selectivity at low calibration levels or matrix positive occurrence resulting in a raised LOQ
Matrix Removal or Matrix Effect	~ 60% of tobacco co-extractives were removed

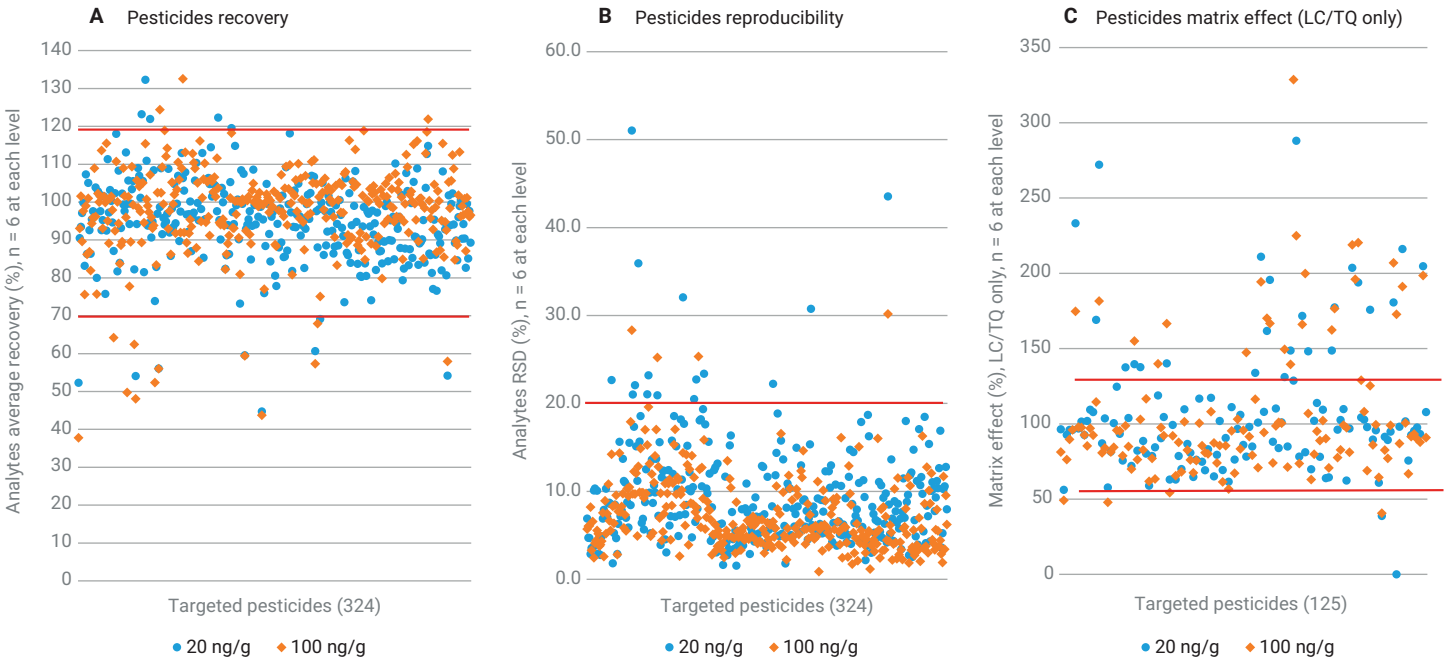
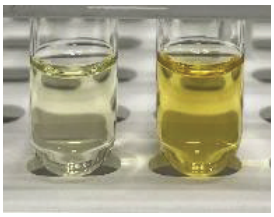


Figure 2-22. Method quantitation individual target results at 20 and 100 ng/g level in tobacco for (A) pesticides recovery, (B) pesticides reproducibility, and (C) pesticides matrix effect (LC/TQ only).

A

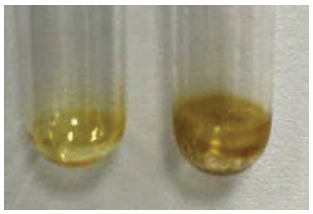
Tobacco

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 Tobacco extract GC/MS full scan background

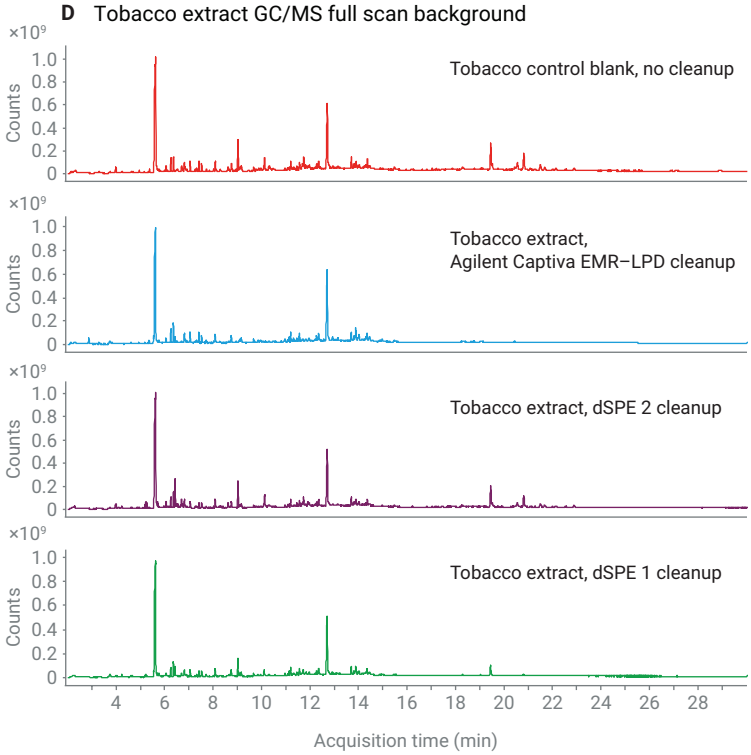


Figure 2-23. Preliminary study on tobacco matrix. (A) Typical tobacco dry leaf, (B) tobacco extract after QuEChERS extraction with and without Agilent Captiva EMR–LPD cleanup, (C) tobacco extract dried residue with and without Captiva EMR–LPD cleanup, (D) tobacco extract GC/MS full scan chromatographic background.

Application highlights

- A simple, rapid, and reliable method using Agilent Bond Elut QuEChERS EN extraction followed by Agilent Captiva Enhanced Matrix Removal–Low Pigment Dry (EMR–LPD) cartridge passthrough cleanup was developed and verified for over 300 pesticides in tobacco by LC/MS/MS and GC/MS/MS.
- The 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.
- 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% RSD for 98% targets in tobacco.
- A similar method was applied for over 300 LC- and GC-amenable pesticides analysis in cumin in application note 5994-6882EN.

Application note highlight

Comparison of Sample Preparation Methods for Pesticide Analysis in Botanical Dietary Supplement Materials

Application note: 5994-7361EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	> 440 Pesticides (LC- and GC-amenable)
Sample Matrix	Botanical dietary supplement (BDS) materials
Instrument, Detection, and Critical Consumables	– LC/MS/MS method was for 220 pesticides for analysis – GC/MS/MS method was for 227 pesticides for analysis
Sample Preparation Method	1) QuEChERS AOAC extraction using ACN, 0.5 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–GPD, EMR–LPD, or EMR–GPD plus EMR–GPF, depending on matrix 3) One sample preparation for both LC/TQ and GC/TQ analysis 4) Collaboration work with customer for the demonstration of EMR sample preparation workflow adoption by the front-end testing labs in industry 5) Customer comparison with the traditional approach demonstrates the EMR method as a more one-size-fits-most approach for various botanical matrices, providing increased instrument uptime and reducing both consumables and labor resources
Sample Preparation Products	– Agilent Bond Elut QuEChERS AOAC extraction kit (p/n 5982-5755CH) – Agilent Captiva EMR–GPD, 6 mL (p/n 5610-2091) – Agilent Captiva EMR–LPD, 6 mL (p/n 5610-2092) – Agilent Captiva EMR–GPF, 3 mL (p/n 5610-2090)

Results summary

Evaluation Parameter	Results
Recovery	70 to 120% recovery in representative BDS samples for over 82% of the total 447 pesticides analyzed
Relative Standard Deviation	< 20% RSD in representative BDS samples for over 82% of the total 447 pesticides analyzed
Matrix Removal or Matrix Effect	New method provided a cleaner sample matrix that reduced the matrix impact on the pesticide analysis

Application highlights

- This method was collaborative work, demonstrated by customers in a third-party laboratory.
- Developed a fast, reliable method for analyzing > 440 pesticides in botanical dietary supplements (BDS) using Agilent QuEChERS extraction and Captiva EMR with Carbon S cartridges, followed by LC/MS/MS and GC/MS/MS.
- Cartridge selection was tailored to sample matrix complexity and pigment intensity using EMR–GPD for green tea and peppermint tea, EMR–LPD for barberry root, and EMR–GPD plus EMR–GPF for curcumin complex.
- The simplified cleanup process enables selective and efficient matrix removal with good recovery and reproducibility.
- A single method reduces preparation time, solvent use, and consumables—making it more sustainable and ecofriendly.
- Consolidated sample preparation simplifies both results interpretation and documentation.
- The method demonstrates acceptable performance with recovery of 70 to 120% and RSDs < 20% in representative BDS samples for over 82% of the total 447 pesticides analyzed.

Application note highlight

Brewing Excellence: Quantitating Over 200 Pesticides in Black Tea with Steady Performance and Maximized Uptime by GC/MS/MS

Application note: 5994-7436EN

Regulation or official guideline? SANTE 11312/2021 guidelines

Method summary

Method Parameter	Setting
Analytes	> 200 Pesticides (GC-amenable)
Sample Matrix	Black tea
Instrument, Detection, and Critical Consumables	– Agilent 8890 GC with an Agilent 7010D GC/TQ – Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm film thickness (p/n 19091S-431UI-KEY) – Agilent Ultra Inert 2 mm dimpled liner (p/n 5190-2297)
Sample Preparation Method	1) QuEChERS EN extraction using ACN with 2% FA, 2 g sample for extraction 2) Mixed-mode passthrough cleanup on EMR–GPD; crude tea extract was mixed with 2% acidic buffer before loading
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit (p/n 5982-5650CH) – Agilent Captiva EMR–GPD, 6 mL (p/n 5610-2091) – Agilent Bond Elut EMR–Lipid polish pouch (p/n 5982-0102)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.01 ppb for 34% of the targets, at or below 0.1 ppb for 74% of compounds, and below 2 ppb for 96%
Recovery	40 to 120% recovery for > 98% pesticides, including for particularly challenging pesticides such as captan and folpet
Relative Standard Deviation	< 20% RSD for 176 compounds
Method Calibration	– Matrix-matched calibrations over a wide dynamic range, up to five orders of magnitude – 0.01 to 1,000 ppb in complex black tea extract
Matrix Removal or Matrix Effect	– Extract cleanup reduces matrix background and improves in-source loading, enhances selectivity, sensitivity, dynamic range, and system uptime. – Analyte response (normalized by ISTD) stayed consistent over 800 injections and 400+ hours.

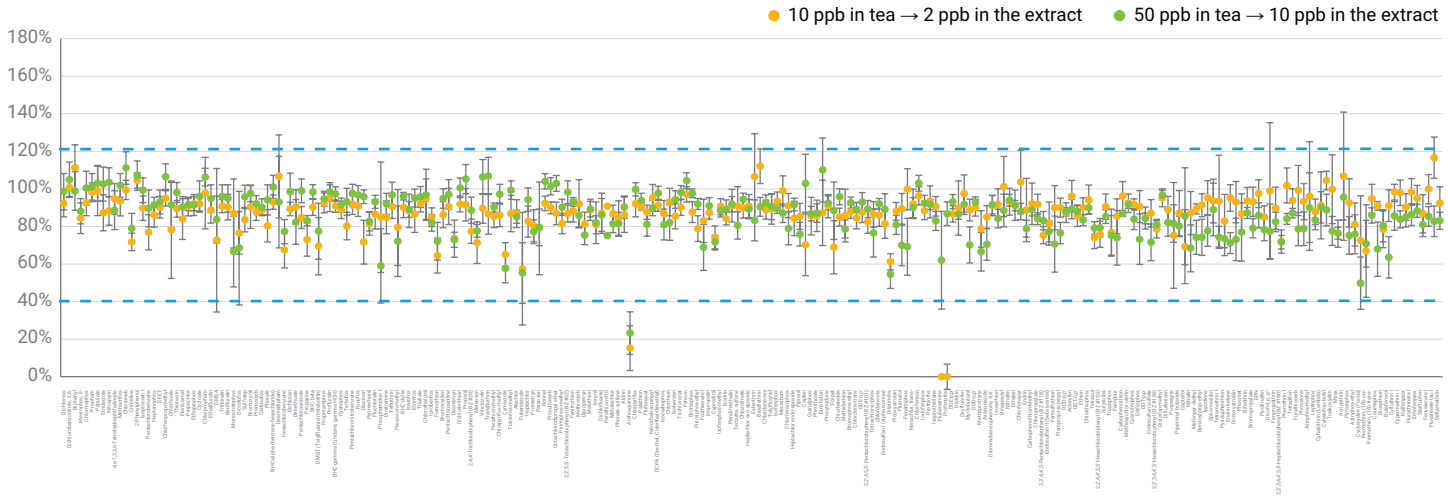


Figure 2-24. Pesticide recoveries in black tea at 10 and 50 ppb shown for all 244 pesticides.

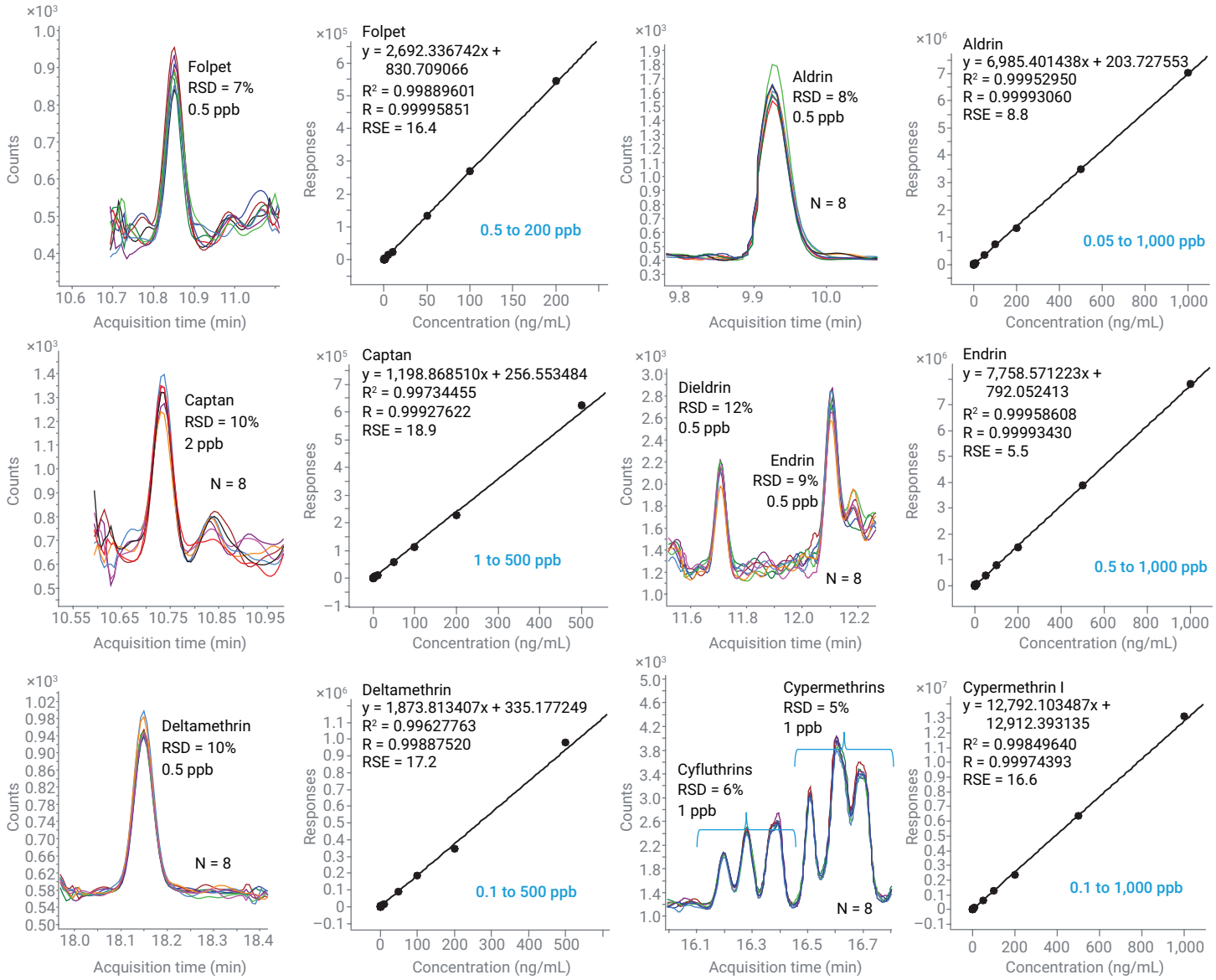


Figure 2-25. MRM chromatograms with eight replicate injections for the selected challenging pesticides. Included are LOQ levels and their calibration curves.

Application highlights

- This workflow solution analyzed 246 pesticides in black tea by GC/TQ at trace levels.
- Matrix-matched calibration allowed for excellent accuracy over a wide dynamic range, spanning up to five orders of magnitude in a complex black tea extract.
- LOQs were as low as 0.01 ppb for over a third of evaluated compounds, and the calibration range spanned up to five orders of magnitude while meeting SANTE 11312/2021 guidelines.
- Method demonstrated exceptional ruggedness and robustness over 800 consecutive injections of a black tea extract spiked with pesticides at 2 ppb with high precision and low RSDs, ensuring prolonged instrument uptime and maximum throughput.

Multilcass Multiresidue Veterinary Drugs Analysis in Food

Introduction

Veterinary drugs are widely used in animal food production, and trace-level residues may remain in meat, milk, eggs, and other animal-derived foods. To meet global regulatory requirements and protect consumer health, laboratories must reliably detect a broad range of chemically diverse veterinary drugs in complex, high-protein and high-fat matrices.

The applications in this compendium demonstrate EMR-based sample preparation strategies that support robust LC/MS/MS analysis for key veterinary drug workflows, including:

- Multiclass multiresidue analysis of veterinary drugs across meat and fish, milk, eggs, and other animal-origin matrices
- Highly selective and effective lipids removal to reduce matrix effects while maintaining analyte recovery
- Improved elution flow under gravity to enhance the product usability
- 10 application notes summarized for multiclass multiresidue veterinary drug analysis

Analyte – Vet Drugs		
Sample Category	**Sample Matrix**	**Application Highlight**
Eggs	Egg	5994-2007EN
		5994-3124EN
Dairy	Milk	5994-3124EN
		5994-7372EN
Fish, Shellfish	Salmon	5994-1124EN
Meat	Beef	5991-8598EN
	Pork	5994-2007EN
	Chicken, pork, beef	5994-1932EN
	Pork, beef, lamb, chicken	5994-8233EN
Edible Offal	Chicken kidney, liver	5994-3680EN

Application note highlight

Analyze 193 Veterinary Drug Residues in Livestock and Poultry Meat

Application note: 5994-8233EN

Regulation or official guideline? China's National Food Safety Standard GB 31650-2019

Method summary

Method Parameter	Setting
Analytes	193 veterinary drugs, 15 classes
Sample Matrix	Pork, beef, lamb, chicken
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495C LC/TQ – Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
Sample Preparation Method	Solvent extraction, then passthrough cleanup on Captiva EMR–Lipid HF cartridge, gravity elution, filter before injection
Sample Preparation Product	Agilent Captiva EMR–Lipid HF cartridges, 3 mL (p/n 5610-2235)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	5 µg/kg for all 193 targets
Recovery	– 50 to 120% recovery for most drugs – Exceptions: < 50% recovery for cefdinir, amprolium, doxycycline, chlorpromazine
Relative Standard Deviation	< 20% RSD for all targets in four meat matrices
Method Calibration	– 0.5 to 100 ng/mL for most targets, with a few exceptions at 0.5 to 50 ng/mL – Matrix-matched calibration curves – $R^2 > 0.99$

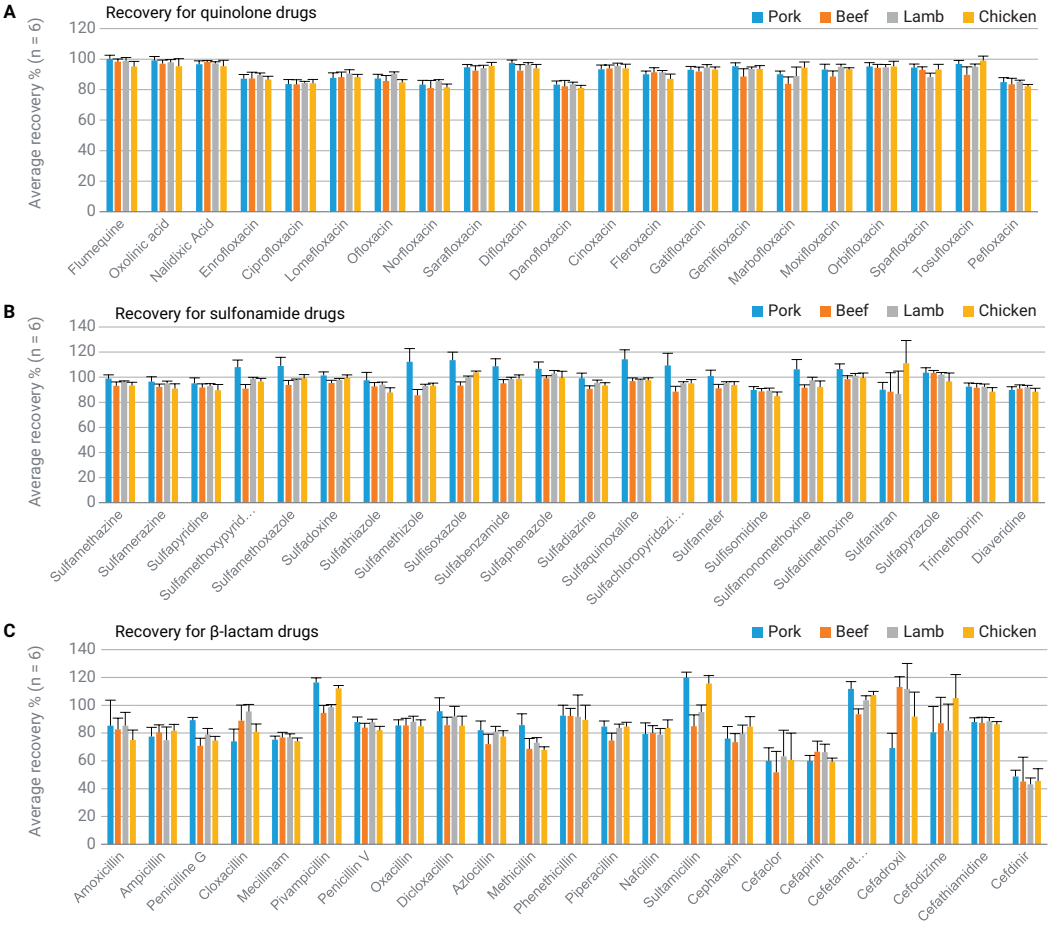


Figure 3-10. Recoveries of three typical vet drug classes, (A) quinolone, (B) sulfonamide, and (C) β-lactam in pork, beef, lamb, and chicken. The sample spiking level was 5 µg/kg.

Application highlights

- A method using Agilent Captiva EMR–Lipid HF cartridges was validated for the analysis of 193 veterinary drugs in pork, beef, lamb, and chicken matrices.
- Captiva EMR–Lipid HF cartridges provided highly efficient and selective matrix removal without compromising veterinary drug targets recovery.
- This simplified sample preparation method saves time and effort.
- The method provides good linearity, precision, and accuracy and could be used to meet the China National Food Safety Standard GB 31650 maximum residue limits for veterinary drugs in food.

Application note highlight

An End-to-End Workflow for Quantitative Screening of Multiclass Multiresidue Veterinary Drugs in Meat Using the Agilent 6470 Triple Quadrupole LC/MS

Application note: 5994-1932EN

Regulation or official guideline? SANTE Guideline

Method summary

Method Parameter	Setting
Analytes	210 veterinary drugs, 28 classes
Sample Matrix	Chicken, pork, beef
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6470 LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 3.0 × 100 mm, 2.7 µm (p/n 695575-302)
Sample Preparation Method	Solvent extraction, then passthrough cleanup on Agilent Captiva EMR–Lipid cartridge
Sample Preparation Product	Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.25 to 25 µg/kg
Recovery	60 to 120% recovery for 206 out of 210 targets in chicken < 60% recovery for four targets
Relative Standard Deviation	< 20% RSD > 20% RSD for four targets
Method Calibration	– Various LOQs to 100 µg/kg – Matrix-matched calibration curves – R^2 > 0.99
Matrix Removal or Matrix Effect	> 75% matrix removal for > 93% of targets

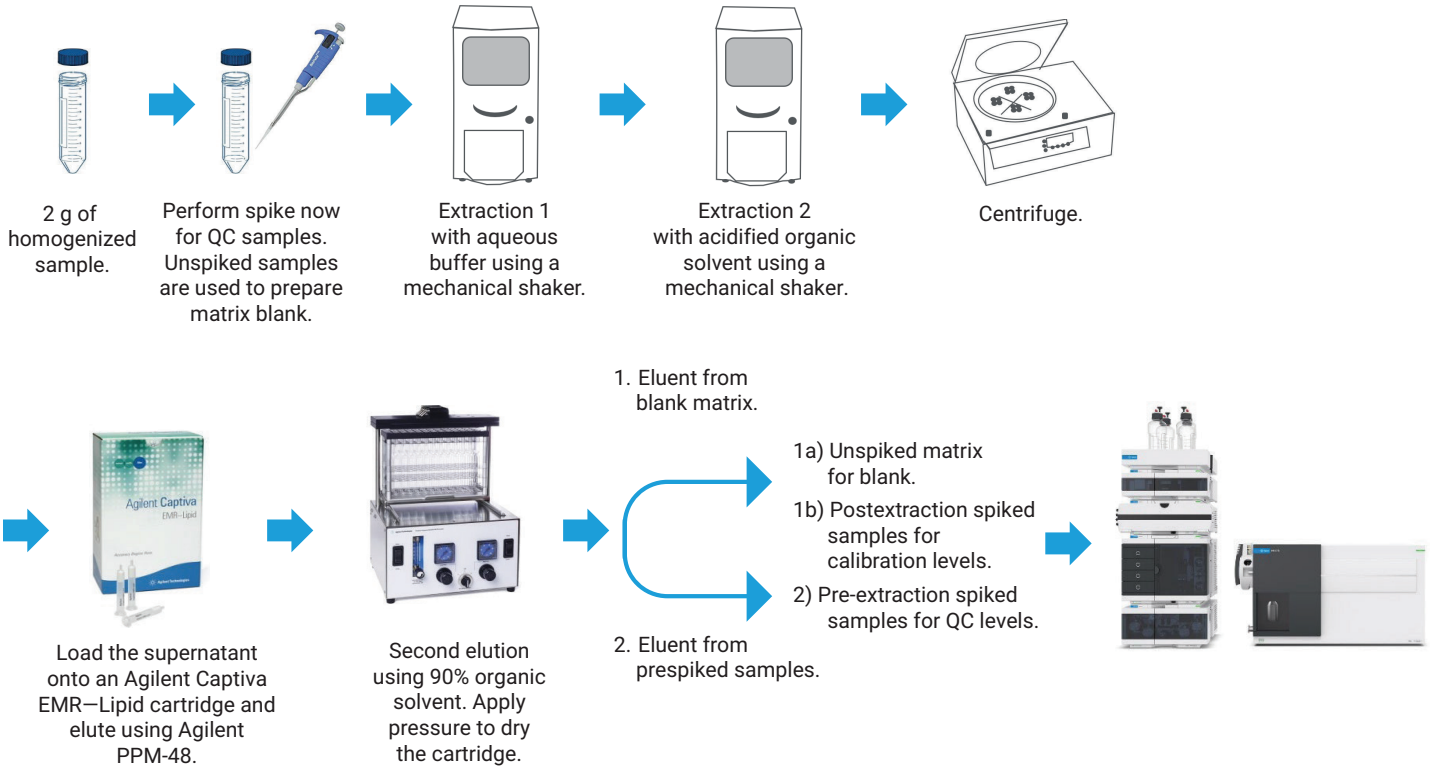


Figure 3-11. Flowchart of sample extraction and Agilent Captiva EMR—Lipid cleanup protocol. (The size of images is not to any scale.)

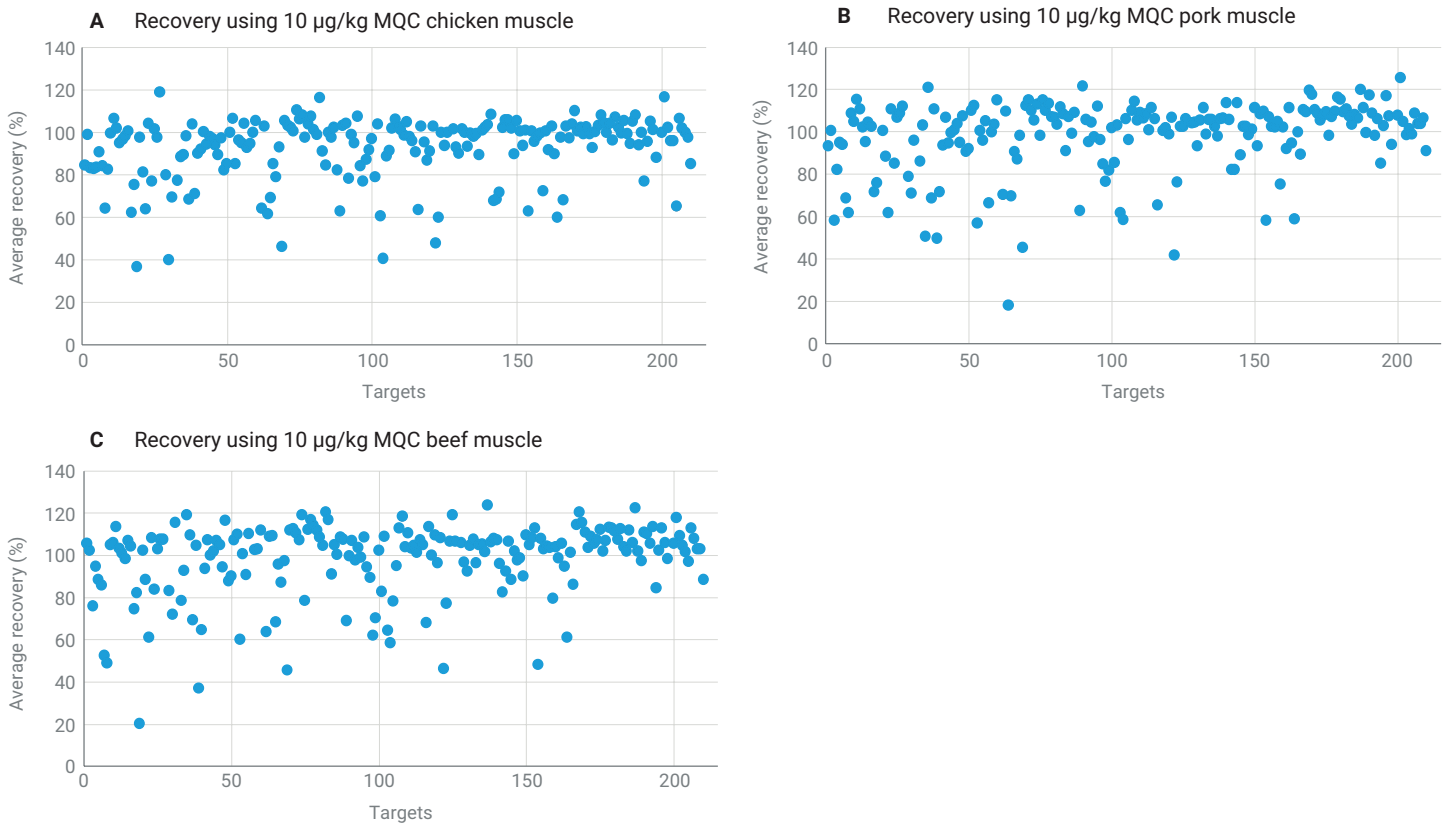


Figure 3-12. Target recovery from (A) chicken, (B) pork, and (C) beef muscle matrices using 10 µg/kg prespiked MQC samples.

Application highlights

- This highly sensitive and reproducible workflow provides fast and reliable screening and quantitation of 210 multiclass veterinary drugs in meat using a solid-liquid extraction with Captiva EMR–Lipid sample cleanup.
- Workflow applicability for routine veterinary drug screening analysis was demonstrated by performing screening of AOAC-listed targets in chicken matrix.
- The simple sample preparation protocol provides highly efficient, selective, and reproducible matrix/lipid removal without impacting the target analyte recoveries.
- Method sensitivity achieved sub-5 ng/mL LODs for most analytes.
- Workflow applicability was also demonstrated in beef and pork matrices.

Application note highlight

Determination of 58 Glucocorticoids in Milk

Application note: 5994-7372EN

Regulation or official guideline? China's National Food Safety Standard GB 31650-2019

Method summary

Method Parameter	Setting
Analytes	58 Glucocorticoids
Sample Matrix	Milk
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6470B LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 3.0 × 100 mm, 2.7 µm (p/n/ 695975-302)
Sample Preparation Method	QuEChERS extraction using salts for veterinary drugs, then passthrough cleanup on Agilent Captiva EMR–Lipid HF cartridge, gravity elution, filter before injection
Sample Preparation Products	– Agilent Bond Elut QuEChERS extraction kit for veterinary drugs (p/n 5982-0032 (50/pk) or 5982-6032 (no centrifuge tubes, 50/pk)) – Agilent Captiva EMR–Lipid HF cartridges, 6 mL (p/n 5610-2236)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.2 to 2 µg/kg
Recovery	90 to 109% recovery for all 58 drugs
Relative Standard Deviation	1.2 to 14% RSD for all targets
Method Calibration	– 0.1 to 20 ng/mL – Matrix-matched calibration curves – $R^2 > 0.99$
Matrix Removal or Matrix Effect	55 to 90% for eight fatty food matrices

Application highlights

- A simplified, rapid, and reliable method using QuEChERS extraction followed by Agilent Captiva EMR–Lipid HF passthrough cleanup was developed and validated for 58 glucocorticoids in cow milk and goat milk.
- Captiva EMR–Lipid HF cartridges demonstrated improved usability for complex food sample elution under gravity within an acceptable elution time window, without compromising matrix removal and target recovery.
- The validated method delivers acceptable LOQs at 0.2 µg/kg for 47 targets and 2 µg/kg for 11 targets, recovery (90 to 109%), and RSD (1.2 to 14.0%) for 58 glucocorticoids in cow milk and goat milk.
- Method could be used to meet China's National Food Safety Standard GB 31650-2019 maximum residue limits (MRLs) for betamethasone and dexamethasone (0.3 µg/kg) in milk.

Application note highlight

Multiclass Multiresidue Veterinary Drug Analysis in Beef Using Agilent Captiva EMR–Lipid Cartridge Cleanup and LC/MS/MS

Application note: 5991-8598EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	39 Veterinary drugs
Sample Matrix	Beef
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity LC with Agilent 6490 LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 2.1 × 150 mm, 2.7 µm (p/n 693775-902)
Sample Preparation Method	Solvent extraction, then passthrough cleanup on Agilent Captiva EMR–Lipid cartridge
Sample Preparation Products	Agilent Captiva EMR–Lipid cartridge, 6 mL, 600 mg (part number 5190-1004) and 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	5 ng/g for 24 drugs and 1 ng/g for 15 drugs
Recovery	– 60 to 120% recovery for 37 out of 39 drugs – Exceptions: acetopromazine and chlorpromazine with < 60% recovery
Relative Standard Deviation	< 20% RSD
Method Calibration	– 5 to 1,000 ng/g for 24 drugs and 1 to 200 ng/g for 15 drugs – Matrix-matched calibration curves – R^2 > 0.98 for all targets
Matrix Removal or Matrix Effect	> 40% matrix co-extractives residues remove

Application highlights

- A rapid, reliable, and robust method using solid-liquid extraction followed by Agilent Captiva EMR–Lipid cartridge cleanup was developed and optimized for the analysis of veterinary drug multiresidues in beef.
- This method is the first application for using EMR–Lipid passthrough cleanup for vet drugs analysis with more details on method development.
- EMR–Lipid sorbent provides highly selective lipids removal and does not cause unwanted target analytes loss.
- Compared to other cartridge passthrough cleanup products, the Captiva EMR–Lipid cartridge provided more efficient matrix cleanup and better recovery of hydrophobic analytes.
- This cleanup method provides superior matrix cleanup, excellent recovery, and high precision.

Application note highlight

Quantitative Screening of Multiresidue Veterinary Drugs in Milk and Egg Using the Agilent 6495C Triple Quadrupole LC/MS

Application note: 5994-3124EN

Application note highlight

Quantitative Analysis of 210 Veterinary Drugs in Organ Meat Using the Agilent 6470 Triple Quadrupole LC/MS

Application note: 5994-3680EN

Regulation or official guideline? SANTE Guideline

Method summary

Method Parameter	Setting
Analytes	210 Veterinary drugs, 28 classes
Sample Matrix	Milk, egg, and chicken kidney and liver
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495C or 6470 LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 3.0 × 100 mm, 2.7 µm (p/n 695575-302)
Sample Preparation Method	Solvent extraction, then passthrough cleanup on Agilent Captiva EMR–Lipid cartridge
Sample Preparation Product	Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.25 to 25 µg/kg
Recovery	– Milk, egg: 60 to 120% recovery for 189 out of 210 targets – Chicken kidney, liver: 60 to 120% recovery for 195 out of 210 targets
Relative Standard Deviation	< 20% RSD for all targets in all matrices
Method Calibration	– Various LOQs to 100 µg/kg – Matrix-matched calibration curves – $R^2 > 0.99$
Matrix Removal or Matrix Effect	– Milk, egg: > 75% matrix removal for > 95% of targets – Chicken kidney, liver: > 75% matrix removal for > 92% of targets

Application highlights

- Quantitative analysis of 210 multiclass veterinary drugs in milk, egg, and chicken kidney and liver matrices was achieved using the Agilent Comprehensive Veterinary Drug dMRM Solution.
- Sample preparation was efficient for target extraction, matrix cleanup, and analysis.
- The method offered sub-1 µg/kg sensitivity for most analytes in milk and eggs and sub-5 µg/kg sensitivity for most analytes in kidney and liver.
- More than 90% targets were within the average recovery of 60 to 120%.
- Linearity, precision, accuracy, recovery, and repeatability results confirmed the method reliability for regulatory-based routine analysis of veterinary drug residues in animal muscle tissue and organ meat.
- Results demonstrated the method reliability for routine screening of over 98% of AOAC-listed veterinary drug targets from the milk matrix, and 100% of AOAC-listed targets from the egg matrix.

Application note highlight

A Fast Sample Preparation Workflow for Veterinary Drugs Analysis in Salmon

Application note: 5994-1124EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	53 Veterinary drugs
Sample Matrix	Salmon
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity LC with Agilent 6495B LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 3.0 × 100 mm, 2.7 µm (p/n 695975-302)
Sample Preparation Method	Solvent extraction, then passthrough cleanup on Agilent Captiva EMR–Lipid cartridge
Sample Preparation Product	Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (part number 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.1 ng/g for all 53 veterinary drugs
Recovery	60 to 120% recovery for all 53 drug targets
Method Calibration	– 0.1 to 100 ng/g – Matrix-matched calibration curves – $R^2 > 0.99$ for all targets

Application highlights

- A simple, fast, robust sample preparation workflow and analytical method were developed for the analysis of seven classes of 53 veterinary drugs in salmon.
- The workflow only contains two steps: a one-step acetonitrile solvent extraction with 5% formic acid, and a gravity passthrough cleanup with Agilent Captiva EMR–Lipid.
- This method provides acceptable recoveries and relative standard deviations for commonly tested veterinary drug classes for seafood.

Application note highlight

Analysis of Acetyl Progesterones in Pork, Eggs, and Milk Using the Agilent Captiva EMR–Lipid by LC/MS/MS

Application note: 5994-2007EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	Acetyl progesterones: flurogestone acetate, megestrol acetate, melengestrol acetate, chlormadinone acetate
Sample Matrix	Pork, kidney, liver, eggs, milk
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6470 LC/TQ – Agilent InfinityLab Poroshell 120 SB-C18, 3.0 × 100 mm, 2.7 µm (p/n 685975-302)
Sample Preparation Method	QuEChERS extraction using salts for veterinary drugs, then passthrough cleanup on Agilent Captiva EMR–Lipid cartridge, filter before injection
Sample Preparation Products	– Agilent Bond Elut QuEChERS extraction kit, veterinary drugs, nonbuffered (p/n 5982-0032) – Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	1 ng/g for the four acetyl progesterones in five matrices
Recovery	80 to 114% recovery
Relative Standard Deviation	0.4 to 9.3% RSD
Method Calibration	– 0.1 to 100 ng/g – Matrix-matched calibration curves
Matrix Removal or Matrix Effect	~ 50 to 190% matrix effect for four targets in five matrices

Application highlights

- A method using an Agilent QuEChERS extraction kit followed by Agilent Captiva EMR–Lipid cleanup is established for the fast and reliable analysis of four acetyl progesterone compounds in meat, eggs, and milk.
- The method provided excellent analyte recovery and reproducibility, efficient matrix removal, and a simplified workflow.

Multiclass Multiresidue PFAS Analysis in Food, Feed, and Other Complex Matrices

Introduction

PFAS have emerged as contaminants of increasing regulatory concern in food, feed, and other complex matrices, requiring laboratories to reliably detect and quantify target analytes at ultra-low part per trillion (ppt) levels across diverse and complex sample types. LC/MS/MS is the method of choice for PFAS analysis, but effective sample preparation remains critical to achieving required sensitivity, controlling contamination risk, and minimizing matrix effects.

The applications in this compendium highlight EMR-based sample preparation approaches that support robust PFAS analysis by simplifying cleanup and improving matrix removal for challenging samples, including:

- Trace-level determination of regulated and emerging PFAS in food, feed, and other complex matrices
- Improved matrix cleanup to support low limit of quantitation (LOQs) while maintaining method efficiency and reproducibility
- Streamlined sample preparation workflows saving time and effort
- 18 application notes for multiclass multiresidue PFAS analysis

Analyte – PFAS		
Sample Category	**Sample Matrix**	**Application Highlight**
Fresh Fruits, Vegetables	Grape, lettuce, mushroom, carrot, tomato, orange juice	5994-7369EN
Eggs	Egg	5994-7366EN
Dairy	Milk	5994-7366EN
Infant Formula, Baby Food	Infant formula	5994-7366EN
	Baby food	5994-7367EN
Dry Plant Material	Dry soybeans	5994-7371EN
	Coffee powder, protein powder	5994-8610EN
Edible Oil	Fish oil	5994-8610EN
Fish, Shellfish	Tuna, shrimp	5994-7368EN
	Tilapia	5994-8232EN
Meat	Beef	5994-7368EN
	Beef	5994-0553EN
Edible Offal	Bovine kidney	5994-7370EN
Beverages	Orange Juice	5994-7369EN
	Beer, wine	5994-8813EN
Environmental	Biosolids	5994-8777EN
	Soil	5994-8778EN
Cosmetics, Personal Care Products	Sunscreen, lotion, foundation, lipstick, eyeshadow	5994-9111EN

Application note highlight

Determination of 30 Per- and Polyfluoroalkyl Substances in Infant Formula, Milk, and Eggs

Application note: 5994-7366EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS targets based on AOAC SMPR mandatory targets list
Sample Matrix	Infant formula, milk, eggs

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 5 or 10 g sample, dry and recon post-treatment
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– LOQ met AOAC SMPR criteria (0.01 µg/kg and above) – **Exception:** Higher LOQ for PFOA in infant formula and 6:2 FTS in milk, due to the positive detection in matrix blank
Recovery	– 65 to 135% for all PFAS targets – 80 to 120% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Relative Standard Deviation	– < 13.9% RSD in salmon – < 11.1% RSD in beef
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.002 to 1 µg/kg range for milk and eggs – 0.004 to 2 µg/kg for infant formula – R^2 > 0.99 for all 30 PFAS targets
Matrix Removal or Matrix Effect	Demonstrated with > 90% reduced GC/MS full scan background and > 60% reduced LC/Q-TOF background

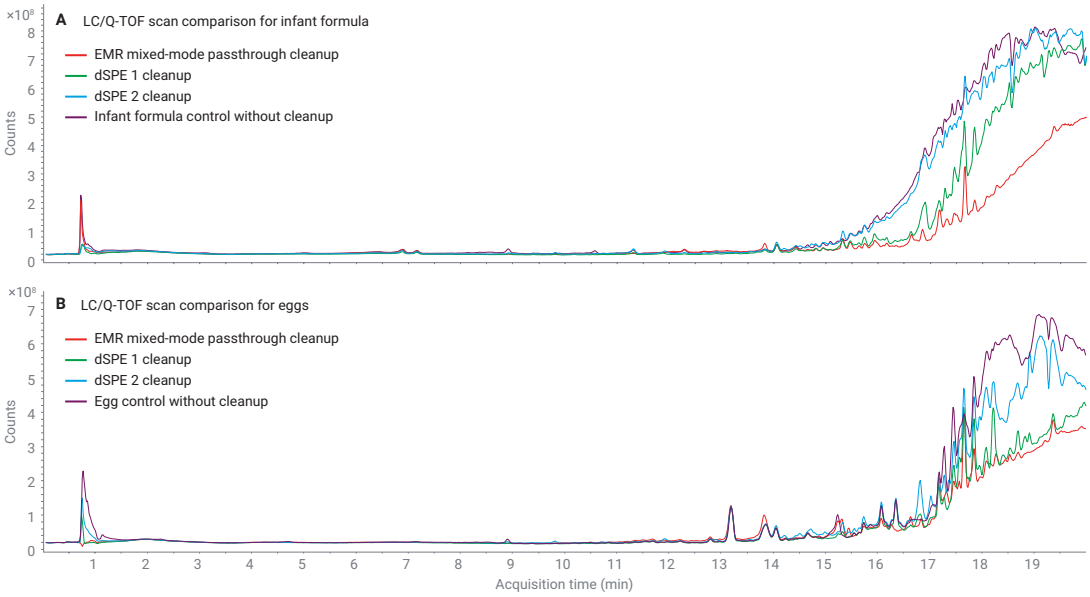


Figure 4-10. Food matrix removal comparison between EMR mixed-mode passthrough cleanup versus traditional dSPE cleanups using LC/Q-TOF TIC (+) scan for (A) infant formula sample and (B) egg sample.

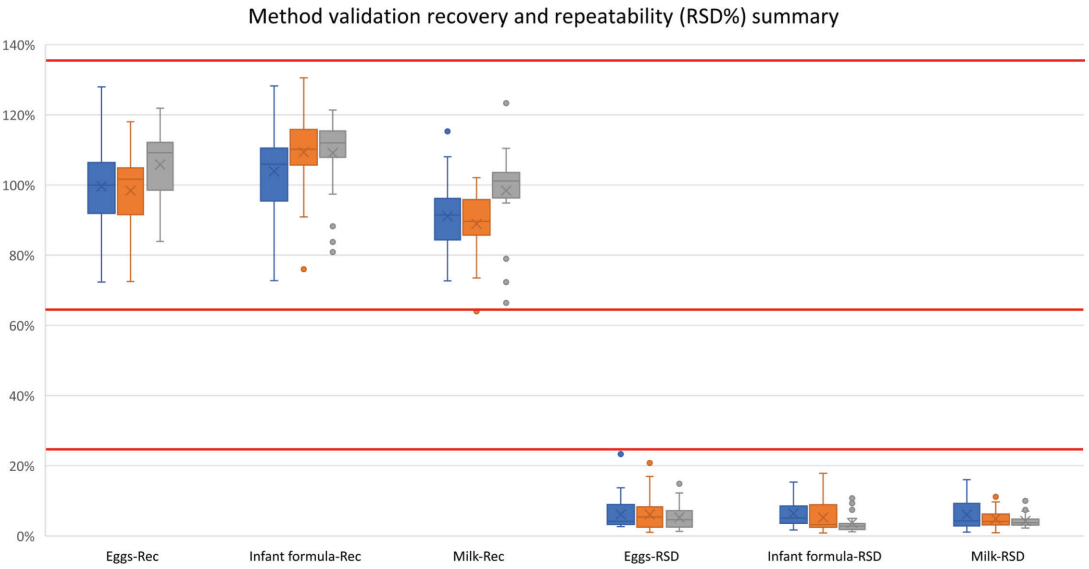


Figure 4-11. Method validation recovery (Rec) and repeatability (RSD%) summary for PFAS analysis in infant formula, milk, and eggs.

Application highlights

- A rapid LC/MS/MS method was developed for 30 PFAS targets in infant formula, milk, and eggs.
- The method uses QuEChERS extraction and passthrough cleanup with the Agilent Captiva EMR PFAS Food II cartridge.
- It provides better matrix removal, PFAS recovery, and sample volume recovery than traditional dSPE cleanup.
- The workflow is simpler, saving time and improving laboratory productivity.
- The method was validated and meets AOAC SMPR 2023.003 performance requirements.
- This workflow is extendable to other animal-origin food liquid, semi-liquid, and milk powder.

Application note highlight

Determination of 30 Per- and Polyfluoroalkyl Substances in Baby Food

Application note: 5994-7367EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS targets based on AOAC SMPR mandatory targets list
Sample Matrix	Baby food

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food I, 340 mg, 10 g sample, dry and recon post-treatment
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food I cartridges, 6 mL, 340 mg (p/n 5610-2230)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	LOQ met AOAC SMPR criteria (0.001 µg/kg and above)
Recovery	– 65 to 135% for all PFAS targets – 80 to 120% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Relative Standard Deviation	– < 25% for all PFAS targets – < 20% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.001 to 0.5 µg/kg range – R^2 > 0.99 for all 30 PFAS targets
Matrix Removal or Matrix Effect	Demonstrated with > 60% reduced GC/MS full scan background, and > 50% reduced LC/Q-TOF background

Application highlights

- A rapid LC/MS/MS method was developed for 30 PFAS targets in baby food.
- The method uses QuEChERS extraction and passthrough cleanup with the Agilent Captiva EMR PFAS Food I cartridge.
- It provides significant improvements in matrix removal, PFAS recovery, and sample volume recovery compared to traditional dSPE cleanup.
- The simplified cleanup process saves time and effort, improving overall laboratory productivity.
- The method was validated and meets AOAC SMPR 2023.003 performance requirements.
- This workflow is extendable to other plant-origin fresh and processed food matrices.

Application note highlight

Determination of 30 Per- and Polyfluoroalkyl Substances in Fruits, Vegetables, and Juices

Application note: 5994-7369EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS targets based on AOAC SMPR mandatory targets list
Sample Matrix	Grape, lettuce, mushroom, carrot, tomato, orange juice

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food I, 340 mg, 10 g sample, dry and recon post-treatment
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food I cartridges, 6 mL, 340 mg (p/n 5610-2230)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– LOQ met AOAC SMPR criteria (0.002 µg/kg and above) – **Exceptions:** Higher LOQs for 4:2 FTS, 6:2 FTS, and PFOS in carrot, PFNA in mushroom
Recovery	– 65 to 135% for all PFAS targets – 80 to 120% for four core PFAS, PFHxS, PFOA, PFOS, PFNA – **Exceptions:** Higher recoveries for PFBA and 4:2 FTS in tomato, 4:2 FTS, PFHxA, 6:2 FTS and PFOSA in lettuce, 4:2 FTS, DONA and 6:2 FTS in carrot, and 4:2 FTS in grape at one or two spiking levels – All exceptions are related to the positive detection in matrix blanks or high matrix background
Relative Standard Deviation	– < 25% for all PFAS targets – < 20% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.001 to 0.5 µg/kg range – R^2 > 0.99 for all 30 PFAS targets
Matrix Removal or Matrix Effect	Demonstrated efficient pigment removal for fresh plant matrices

Application highlights

- A rapid LC/MS/MS method was developed for 30 PFAS targets in six produce and juice matrices.
- The method uses QuEChERS extraction and passthrough cleanup with the Agilent Captiva EMR PFAS Food I cartridge.
- It provides significant improvements in matrix removal, PFAS recovery, and sample volume recovery compared to traditional dSPE cleanup.
- The simplified cleanup process saves time and effort, improving overall laboratory productivity.
- The method was validated and meets AOAC SMPR 2023.003 performance requirements.
- This workflow is extendable to other fresh produce or processed produce food matrices.

Application note highlight

Determination of 30 Per- and Polyfluoroalkyl Substances in Beef, Tuna, and Shrimp

Application note: 5994-7368EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS targets based on AOAC SMPR mandatory targets list
Sample Matrix	Beef, tuna, shrimp

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 5 g sample, dry and recon post-treatment
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– LOQs met AOAC SMPR criteria (0.02 µg/kg and above) – **Exception:** Higher PFNA LOQ in beef
Recovery	– 65 to 135% for all PFAS targets – 80 to 120% for four core PFAS, PFHxS, PFOA, PFOS, PFNA – **Exception:** High recoveries for PFNA in beef and PRTrDA in shrimp, due to positive detection in matrix blank
Relative Standard Deviation	– < 25% for all PFAS targets – < 20% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.004 to 2 µg/kg range – R^2 > 0.99 for all 30 PFAS targets
Matrix Removal or Matrix Effect	Demonstrated with > 90% reduced GC/MS full scan background, and > 60% reduced LC/Q-TOF background

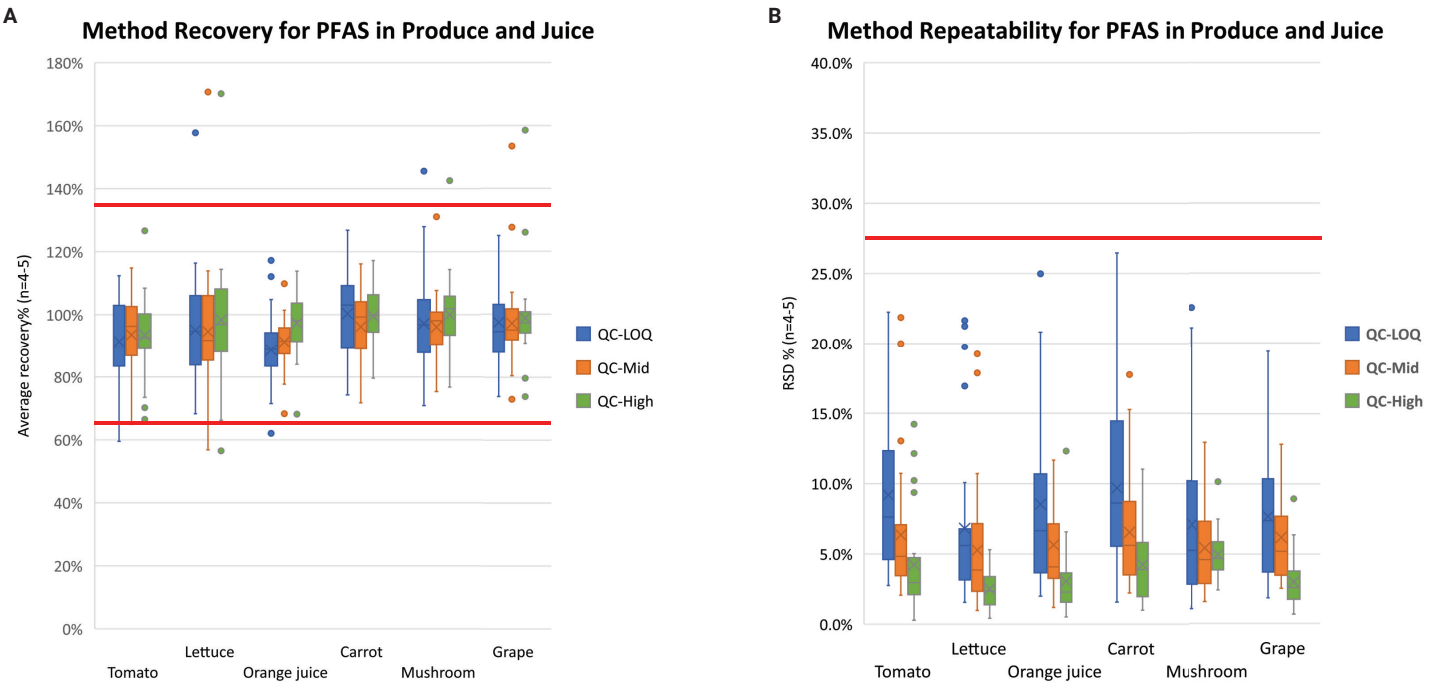


Figure 4-12. Method (A) recovery and (B) repeatability results summary for 30 PFAS in produce and juice.

Application highlights

- A rapid LC/MS/MS method was developed for 30 PFAS targets in beef, tuna, and shrimp.
- The method uses QuEChERS extraction and passthrough cleanup with the Agilent Captiva EMR PFAS Food II cartridge.
- It delivers significant improvements in matrix removal, PFAS recovery, and sample volume recovery compared to traditional dSPE cleanup.
- The simplified workflow saves time and effort, enhancing overall laboratory productivity.
- The method was validated and meets AOAC SMPR 2023.003 performance requirements.
- This workflow is extendable to other similar biological tissue matrices.

Application note highlight

Determination of 30 Per- and Polyfluoroalkyl Substances in Bovine Kidney

Application note: 5994-7370EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS targets based on AOAC SMPR mandatory targets list
Sample Matrix	Bovine kidney

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 2 g sample, dilution with water at 1:1 post-treatment
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	LOQs met AOAC SMPR criteria (0.2 µg/kg and above)
Recovery	– 65 to 135% for all PFAS targets – 80 to 120% for four core PFAS, PFHxS, PFOA, PFOS, PFNA – **Exception:** High recovery for 4:2 FTS and PFPeS at one spiking level, due to matrix effect
Relative Standard Deviation	– < 25% for all PFAS targets – < 20% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.02 to 100 µg/kg range – R^2 > 0.99 for all 30 PFAS targets
Matrix Removal or Matrix Effect	Demonstrated efficient fat/lipids removal for fatty edible offal matrix

Application highlights

- A rapid LC/MS/MS method was developed for 30 PFAS targets in bovine kidney.
- The method uses QuEChERS extraction and passthrough cleanup with the Agilent Captiva EMR PFAS Food II cartridge.
- The workflow is simplified, saving time and improving overall laboratory productivity.
- The method was validated and meets AOAC SMPR 2023.003 performance requirements.
- This workflow is extendable to other edible offal food matrices.

Application note highlight

Determination of 30 Per- and Polyfluoroalkyl Substances in Dry Soybeans

Application note: 5994-7371EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS targets based on AOAC SMPR mandatory targets list
Sample Matrix	Dry soybeans
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program – Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902) – Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006) – PP containers for sample extraction and storage, including: – PP vials and caps (p/n 5182-0567 and 5182-0542) – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)
Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 5 g sample, dry and recon post-treatment
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	LOQ met AOAC SMPR criteria (0.05 µg/kg and above)
Recovery	– 65 to 135% for all PFAS targets – 80 to 120% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Relative Standard Deviation	– < 25% for all PFAS targets – < 20% for four core PFAS, PFHxS, PFOA, PFOS, PFNA
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.05 to 10 µg/kg range – R^2 > 0.99 for all 30 PFAS targets
Matrix Removal or Matrix Effect	Demonstrated with > 80% reduced LC/Q-TOF background

Application highlights

- A rapid LC/MS/MS method was developed for 30 PFAS targets in soybeans.
- The method uses QuEChERS extraction and passthrough cleanup with the Agilent Captiva EMR PFAS Food II cartridge.
- The workflow is simplified, saving time and improving overall laboratory productivity.
- The method was validated and meets AOAC SMPR 2023.003 performance requirements.
- This workflow is extendable to other similar plant-origin dried food matrices.

Application note highlight

Determination of 30 PFAS in Fish Oil, Coffee Powder, and Protein Powder

Application note: 5994-8610EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	30 PFAS
Sample Matrix	Fish oil, coffee powder, protein powder

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ, feed injection on Hybrid Multisampler
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 1 or 2 g sample, direct injection
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– LOQs met AOAC SMPR criteria (0.01 µg/kg and above) – Higher LOQ for 6:2 FTS in protein powder due to positive detection in matrix blank
Recovery	– 65 to 135% for all PFAS targets – Four critical PFAS met 80 to 120% recovery
Relative Standard Deviation	– 25% RSD – Four critical PFAS met < 20% RSD
Method Calibration	– Neat calibration curve with the use of 18 ISTDs – 0.02 to 50 µg/kg range for protein powder and coffee powder – 0.01 to 25 µg/kg range for fish oil

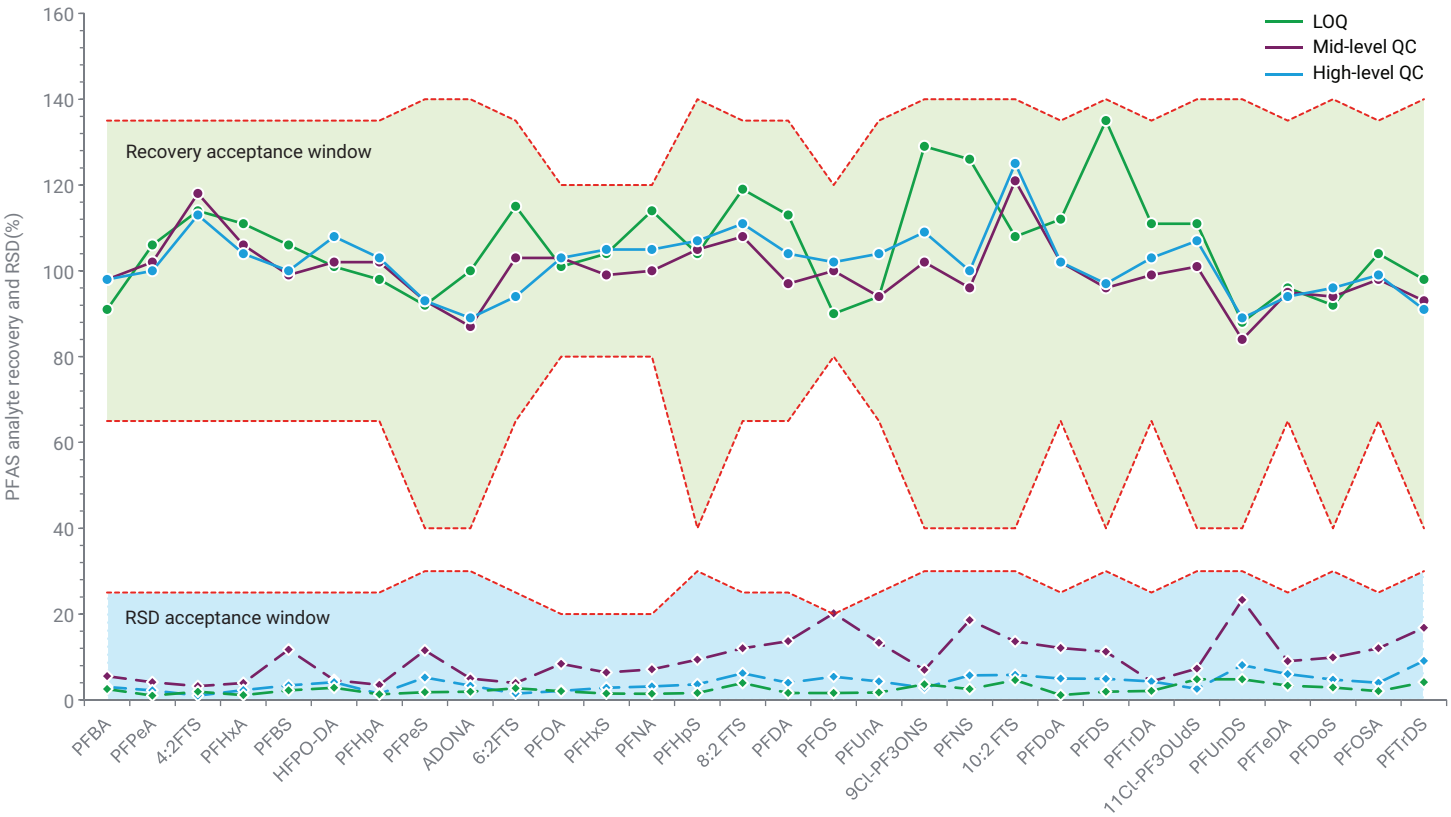


Figure 4-13. Validation results summary for 30 PFAS in coffee powder. Acceptance criteria based on AOAC SMPR 2023.003.

Application highlights

- A simplified and reliable method was developed for quantifying 30 PFAS compounds in coffee powder, protein powder, and fish oil using QuEChERS extraction and Captiva EMR PFAS Food II cleanup, followed by LC/TQ analysis.
- Feed injection program on Hybrid Multisampler allows direct injection of sample eluent after EMR cleanup.
- The sample preparation workflow is characterized by its simplicity, robustness, and cost-effectiveness, offering significant savings in time and resources.
- The method was validated according to AOAC SMPR 2023.003 guidelines, confirming its suitability for regulatory and quality control applications.

Application note highlight

Determination of 43 PFAS in Beer and Wine

Application note: 5994-8813EN

Regulation or official guideline? AOAC SMPR 2023.003

Method summary

Method Parameter	Setting
Analytes	43 PFAS
Sample Matrix	Beer and wine

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using feed injection
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	Solvent extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food I, 680 mg, 2 g sample, direct injection
Sample Preparation Product	Agilent Captiva EMR PFAS Food I cartridges, 6 mL, 750 mg (p/n 5610-2231)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	LOQ met AOAC SMPR criteria (0.01 µg/kg and above)
Recovery	– 65 to 135% recovery – Four critical PFAS met 80 to 120% recovery – **Exception:** Higher accuracy for 3-3 FTCA due to matrix effect and lack of corresponding ISTD
Relative Standard Deviation	– 25% RSD – Four critical PFAS met < 20% RSD
Method Calibration	– Neat calibration curve with the use of 24 ISTDs – 0.01 to 8 µg/kg dynamic range or higher
Matrix Removal or Matrix Effect	Demonstrated with acceptable matrix cleanup by visual comparison of sample color and separated matrix residue layer

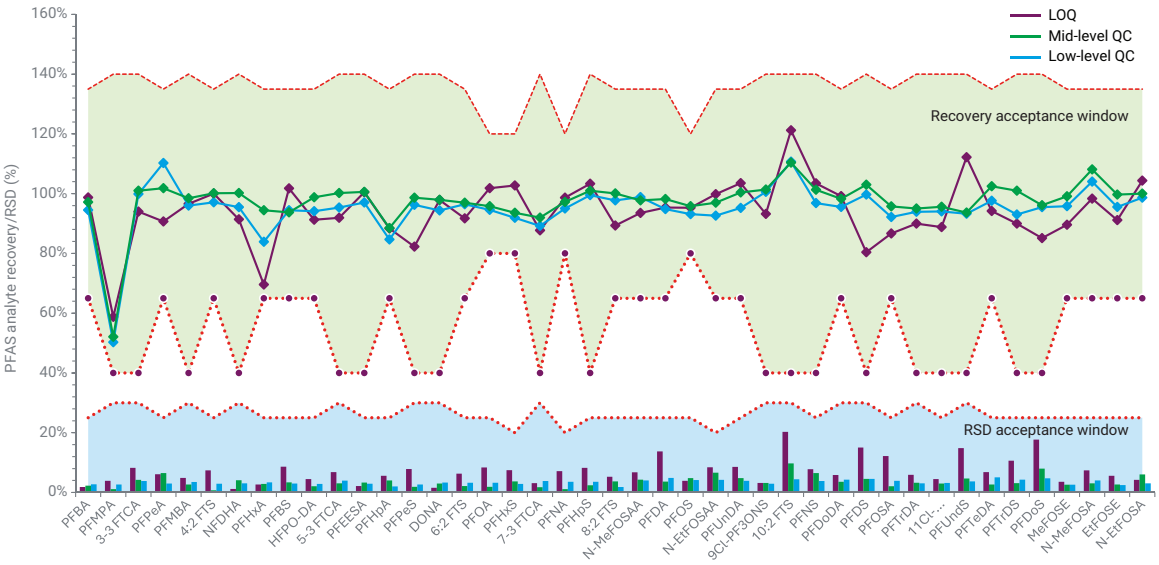


Figure 4-14. Validation results summary for 43 PFAS in red wine. The three lines in the middle show analyte recovery results, and the three sets of columns at the bottom represent RSD values at three spiking levels. Results are color-coded by spiking levels: purple for LOQ, blue for low, and green for mid-level.

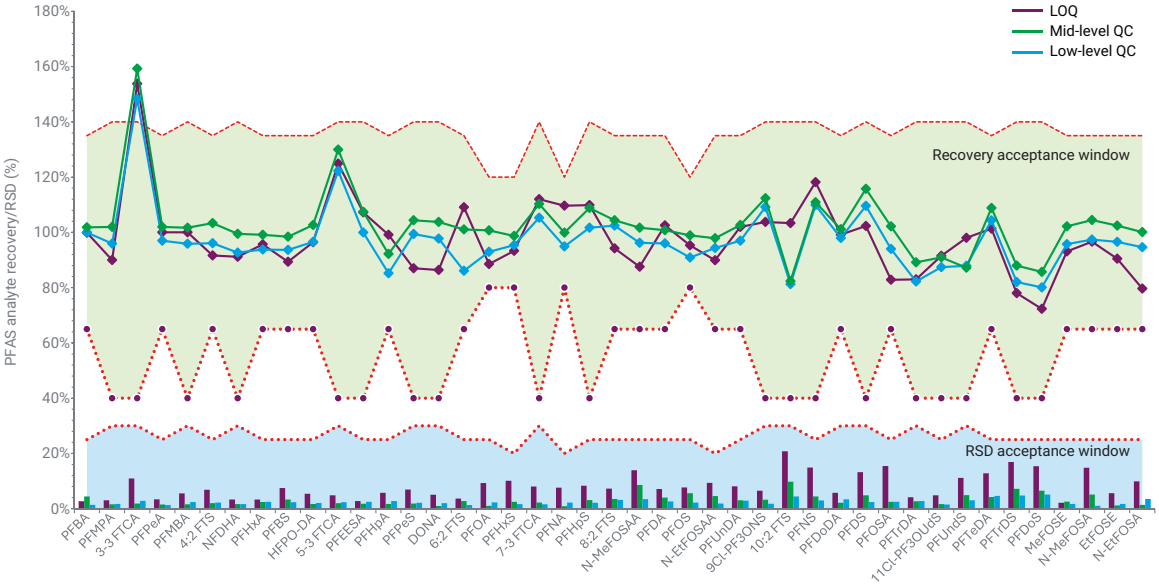


Figure 4-15. Validation results summary for 43 PFAS in light beer. The three lines in the middle show analyte recovery results, and the three sets of columns at the bottom represent RSD values at three spiking levels. Results are color-coded by spiking levels: purple for LOQ, blue for low, and green for mid-level.

Application highlights

- A simplified and reliable method was developed for the quantitative determination of 43 PFAS compounds in alcoholic beverages using solvent extraction and Captiva EMR PFAS Food I cleanup, followed by LC/MS/MS detection.
- The sample preparation workflow is simple, robust, and cost-effective, resulting in significant savings in both time and resources.
- The method was validated according to AOAC SMPR guidelines, confirming its suitability for high-quality PFAS analysis in alcoholic beverage matrices.

Application note highlight

Determination of 40 PFAS in Tilapia Tissue Following EPA 1633 Method Guidance

Application note: 5994-8232EN

Regulation or official guideline? EPA Method 1633 quality control guidance

Method summary

Method Parameter	Setting
Analytes	40 PFAS (EPA Method 1633 list)
Sample Matrix	Tilapia

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 5 g sample, direct injection
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– LOQ met EPA 1633 QC criteria for all 40 targets (0.05 µg/kg and above) – 27 analytes in the AOAC list also met SMPR criteria on LOQ
Recovery	– Recoveries (89 to 151%), met EPA Method 1633 QC acceptance criteria except 3:3 FTCA at LOQ level – **Exception:** 3:3 FTCA at LOQ spiking level
Relative Standard Deviation	RSD (0.7 to 12.2%), met EPA Method 1633 QC acceptance criteria
Method Calibration	– Neat calibration curve with the use of 24 EIS and seven NIS – 0.05 to 20 µg/kg dynamic range or higher
Matrix Removal or Matrix Effect	Demonstrated with ~ 100% matrix effect for all NIS compounds

Application highlights

- A simplified and rapid method using QuEChERS extraction followed by Agilent Captiva EMR PFAS Food II passthrough cleanup was developed and validated for 40PFAS targets in fish tissue.
- The method was validated under EPA 1633 guidelines using LC/MS/MS with acceptable recoveries for both EIS and NIS standards, good accuracy and precision, and acceptable method detection limits (MDLs) and limits of quantitation (LOQs).
- Compared to traditional EPA 1633 SPE-based sample preparation, the new method saved over 80% in preparation time, reduced solvent and chemical consumption by approximately 80%, and used fewer consumables.
- The new method demonstrated improved performance, efficiency, and cost-effectiveness for PFAS analysis in tissue samples compared to the traditional EPA 1633 method.

Table 4-4. Comparison of QuEChERS$_{ext}$-EMR method quantification results with published EPA 1633 results.[1]

Target	RT (min)	Quantification Reference IS	MDL-EMR (µg/kg)	MDL-EPA (µg/kg)	LOQ-EMR (µg/kg)	LOQ-EPA (µg/kg)
PFHxA	5.56	$^{13}C_5$-PFHxA	0.002	0.111	0.05	0.4 to 0.5
PFHpA	6.94	$^{13}C_4$-PFHpA	0.006	0.099	0.05	0.4 to 0.5
PFOA isomers	8.5	$^{13}C_8$-PFOA	0.005	0.105	0.05	0.4 to 0.5
PFNA isomers	10.1	$^{13}C_9$-PFNA	0.009	0.119	0.05	0.4 to 0.5
PFDA	11.34	$^{13}C_6$-PFDA	0.006	0.149	0.05	0.4 to 0.5
PFUnA	11.99	$^{13}C_7$-PFUdA	0.007	0.125	0.05	0.4 to 1.0
PFDoA	12.51	$^{13}C_2$-PFDoA	0.007	0.101	0.05	0.4 to 0.5
PFTrDA	12.96	$^{13}C_2$-PFDoA	0.009	0.142	0.05	0.4 to 0.5
PFTeDA	13.36	$^{13}C_2$-PFTeDA	0.009	0.159	0.05	0.4 to 1.0
PFOSA isomers	12.88	$^{13}C_8$-PFOSA	0.005	0.069	0.05	0.4 to 0.5
N-MeFOSA isomers	14.19	D_3-N-MeFOSA	0.011	0.162	0.05	0.4 to 0.5
N-EtFOSA isomers	14.45	D_5-N-EtFOSA	0.008	0.163	0.05	0.4 to 1.0
N-MeFOSAA isomers	11.32	D_3-N-MeFOSAA	0.016	0.145	0.05	0.4 to 0.5
N-EtFOSAA isomers	11.63	D_5-N-EtFOSAA	0.01	0.148	0.05	0.4 to 0.5
PFBS	5.76	$^{13}C_3$-PFBS	0.007	0.097	0.05	0.4 to 0.5
PFPeS	7.36	$^{13}C_4$-PFHpA	0.004	0.076	0.05	0.4 to 0.5
PFHxS isomers	9.09	$^{13}C_3$-PFHxS	0.013	0.081	0.05	0.4 to 0.5
PFHpS	10.82	$^{13}C_9$-PFNA	0.004	0.119	0.05	0.4 to 0.5
PFOS isomers	11.74	$^{13}C_8$-PFOS	0.005	0.145	0.05	0.4 to 2.0
PFNS	12.32	$^{13}C_7$-PFUdA	0.009	0.108	0.05	0.4 to 0.5
PFDS	12.81	$^{13}C_2$-PFDoA	0.018	0.114	0.05	0.4 to 0.5
PFDoS	13.61	$^{13}C_8$-PFOS	0.021	0.153	0.05	0.4 to 0.5
PFPeA	4.5	$^{13}C_5$-PFPeA	0.005	0.155	0.1	0.8 to 1.0
PFEESA	6.47	$^{13}C_4$-PFHpA	0.007	0.123	0.1	0.8 to 1.0
PFMPA	3.95	$^{13}C_4$-PFBA	0.007	0.273	0.1	0.8 to 2.0
PFMBA	4.79	$^{13}C_5$-PFPeA	0.005	0.168	0.1	0.8 to 1.0
NFDHA	5.43	$^{13}C_5$-PFHxA	0.01	0.216	0.1	0.8 to 1.0
PFBA	3.47	$^{13}C_4$-PFBA	0.019	0.208	0.2	1.6 to 4.0
HFPO-DA	6.05	$^{13}C_2$-HFPO-DA	0.014	0.339	0.2	1.6 to 2.1
4:2 FTS	5.14	$^{13}C_2$-4:2 FTS	0.024	0.369	0.2	1.6 to 2.0
6:2 FTS	7.82	$^{13}C_2$-6:2 FTS	0.019	0.537	0.2	1.6 to 2.0
8:2 FTS	10.87	$^{13}C_2$-8:2 FTS	0.039	0.378	0.2	1.6 to 2.0
ADONA	7.52	$^{13}C_8$-PFOA	0.013	0.274	0.2	1.6 to 2.0
9Cl-PF3ONS	12.2	$^{13}C_7$-PFUdA	0.019	0.362	0.2	1.6 to 2.0
11Cl-PF3OUdS	13.15	$^{13}C_8$-PFOS	0.021	0.352	0.2	1.6 to 2.0
N-MeFOSE isomers	14.09	D_7-N-MeFOSE	0.046	0.832	0.5	4.0 to 5.0
N-EtFOSE isomers	14.37	D_9-N-EtFOSE	0.028	1.77	0.5	4.0 to 5.0
3:3 FTCA	4.07	$^{13}C_5$-PFPeA	0.034	0.716	0.25	2.0 to 4.0
5:3 FTCA	6.17	$^{13}C_4$-PFHpA	0.12	2.38	1.25	10 to 20
7:3 FTCA	9.26	$^{13}C_3$-PFHxS	0.199	2.02	1.25	10 to 12.5

Application note highlight

Determination of 40 PFAS in Biosolids Following EPA Method 1633 Quality Control Guidance

Application note: 5994-8777EN

Regulation or official guideline? EPA Method 1633 quality control guidance

Method summary

Method Parameter	Setting
Analytes	40 PFAS
Sample Matrix	Biosolids

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 0.5 g sample, direct injection
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– Most analytes (were below EPA Method 1633 published LOQs – Higher LOQs and MDLs for N-MeFOSAA, N-EtFOSAA, PFOS, PFTrDA, and N-MeFOSE, due to significant high positive detection in matrix reference material
Recovery	– EIS and NIS recoveries (36 to 206%), met EPA Method 1633 acceptance range – Native accuracy was impacted by the significant positive detections in matrix reference material – The detected PFAS compounds' concentration were comparable with NIST non-certified concentration with < 25% difference
Relative Standard Deviation	Targets RSD (0.6 to 13.4%), met EPA Method 1633 acceptance criteria
Method Calibration	– Neat calibration curve with the use of 24 EIS and seven NIS – 0.16 to 200 µg/kg dynamic range or higher
Matrix Removal or Matrix Effect	Demonstrated with acceptable matrix effect for NIS compounds

Table 4-5. Proficiency test results and comparison. All concentrations reported in µg/kg.

Analytes	NIST 2781 Non-Certified Conc.	Detection in the Study (n = 7)			Reference Report (n = 6)		
		Ave. Conc.	RSD	Diff.	Ave. Conc.	RSD%	Diff. (%)
PFHxA	13.0 ± 2.0	11.48	1.1%	–11.7%	12.45	4.6%	–4.20%
PFHpA	7.96 ± 1.5	7.00	2.4%	–12.1%	7.64	4.2%	–4.00%
PFOA	28.5 ± 3.3	28.11	0.9%	–1.4%	27.17	2.3%	–4.70%
PFHxS	9.39 ± 1.76	7.07	3.6%	–24.7%	3.37	109.7%	–64.1%
PFOS	225 ± 41	197.52	3.0%	–12.2%	244.3	4.6%	8.60%
PFOSA	6.31 ± 0.97	5.97	1.3%	–5.4%	1.85	49.9%	–70.70%

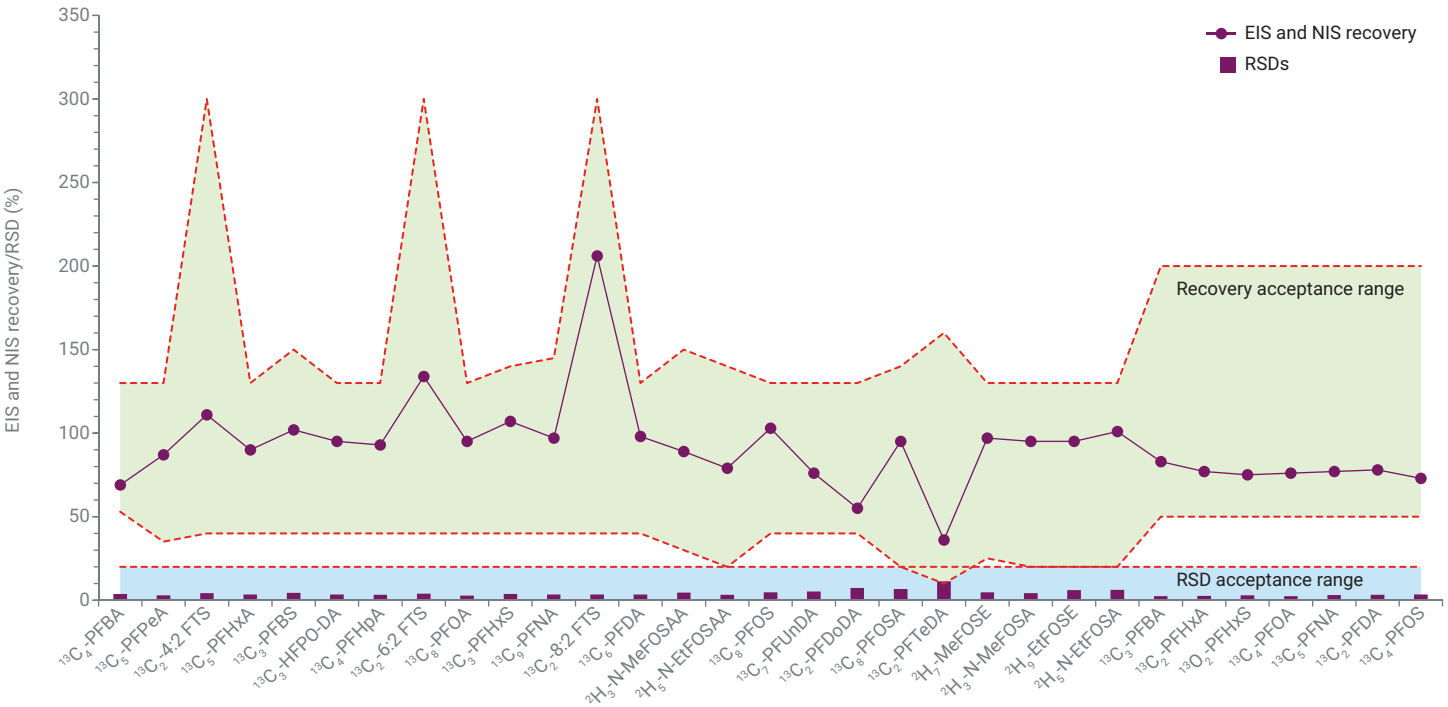


Figure 4-16. Average recoveries of EIS and NIS compounds in the biosolids validation batch using the QuEChERS-EMR method. The purple line in the middle exhibits the EIS and NIS recovery, while the columns at the bottom present the RSDs.

Application highlights

- A simplified and rapid method was developed for quantifying 40 PFAS compounds in biosolid/sludge using QuEChERS extraction and Agilent Captiva EMR PFAS Food II cleanup, followed by LC/MS/MS analysis.
- Compared to traditional SPE-based sample preparation outlined in EPA Method 1633, this new approach reduces preparation time by over 50%, lowers solvent consumption by approximately 80%, and minimizes the use of consumables.
- The method met EPA Method 1633 validation criteria, demonstrating acceptable recoveries for both isotopically labeled (EIS) and non-isotopically labeled (NIS) compounds, accurate quantitation of native PFAS analytes, and lower MDLs and LOQs than estimated values from solid sample levels and EPA guidance—except in a few cases due to significant matrix-related detections.
- A proficiency test using selected PFAS analytes in the NIST 2781 Standard Reference Material confirmed acceptable quantitation with excellent reproducibility.
- This approach provides a cost-effective, high-performance alternative to conventional PFAS sample preparation methods and is well-suited for biosolid/sludge matrices.

Application note highlight

Determination of 40 PFAS in Soil Following EPA Method 1633 Quality Control Guidance

Application note: 5994-8778EN

Regulation or official guideline? EPA Method 1633 quality control guidance

Method summary

Method Parameter	Setting
Analytes	40 PFAS
Sample Matrix	Soil and sediment

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6495D LC/TQ using a sandwiched injection program
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004-0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food I, 680 mg, 5 g sample, direct injection
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food I cartridges, 680 mg (p/n 5610-2231)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	Targets LOQs 0.006 to 1.25 µg/kg, all below EPA Method 1633 published LOQs
Recovery	– EIS and NIS recoveries (88 to 139%), met EPA Method 1633 QC acceptance criteria – Native PFAS accuracy (83 to 147%) at three spiking levels, met EPA Method 1633 QC acceptance criteria – **Exception:** Higher accuracy for PFHpA at LOQ level
Relative Standard Deviation	Native RSD (0.8 to 18.4%), met EPA Method 1633 acceptance criteria
Method Calibration	– Neat calibration curve with the use of 24 EIS and 7 NIS – 0.02 to 20 µg/kg dynamic range or higher
Matrix Removal or Matrix Effect	Demonstrated with acceptable matrix effect for NIS compounds

Application highlights

- A simplified and rapid method was successfully developed for quantifying 40 PFAS compounds in soil using QuEChERS extraction and Captiva EMR PFAS Food I cleanup, followed by LC/MS/MS analysis.
- Compared to traditional SPE-based preparation methods, this new approach reduces sample preparation time by over 50%, decreases solvent usage by approximately 80%, and minimizes the use of consumables.
- The method adheres to EPA method 1633 quality control guidelines and demonstrated acceptable recoveries for both EIS and NIS compounds, accurate quantitation of native PFAS analytes, and lower method detection limits (MDLs) and limits of quantitation (LOQs) than those reported in EPA method 1633 guidance.
- This approach offers a cost-effective, high-performance alternative to conventional PFAS sample preparation methods and is also applicable to sediment matrices.

Application note highlight

Determination of 41 PFAS in Cosmetics

Application brief: 5994-9111EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	41 PFAS
Sample Matrix	Sunscreen, lotion, foundation, lipstick, eyeshadow

Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity III LC with Agilent 6495D LC/TQ using standard injection
	– Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (p/n 959758-902)
	– Agilent InfinityLab PFC-free HPLC conversion kit (p/n 5004 0006)
	– PP containers for sample extraction and storage, including:
	  – PP vials and caps (p/n 5182-0567 and 5182-0542)
	  – Extraction tubes, 15 mL and 50 mL (p/n 5610-2049 and 5610-2039)

Sample Preparation Method	QuEChERS extraction (acidified) + passthrough cleanup on Captiva EMR PFAS Food II, 1 g sample, standard injection with 0.5 µL volume
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts (p/n 5982-5650CH) – Agilent Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (p/n 5610-2232)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	LOQ: 2.1 ng/g (average for PFAS analytes in tested cosmetics)
Recovery	– 70 to 120% recovery for >97% of analytes in four types of cosmetics matrices – Exception: 6-2 FTSA, EtFOSE, N-EtFOSA, PFBA, PFTrDA in Lipstick matrix
Relative Standard Deviation	Not reported
Method Calibration	– For analytes with an internal standard (ISTD), an internal-standard calibration method was applied. For compounds without an ISTD, absolute calibration was used – 0.05 to 10 ppb (ng/g) dynamic range
Matrix Removal or Matrix Effect	Acceptable matrix effect using the final optimized method

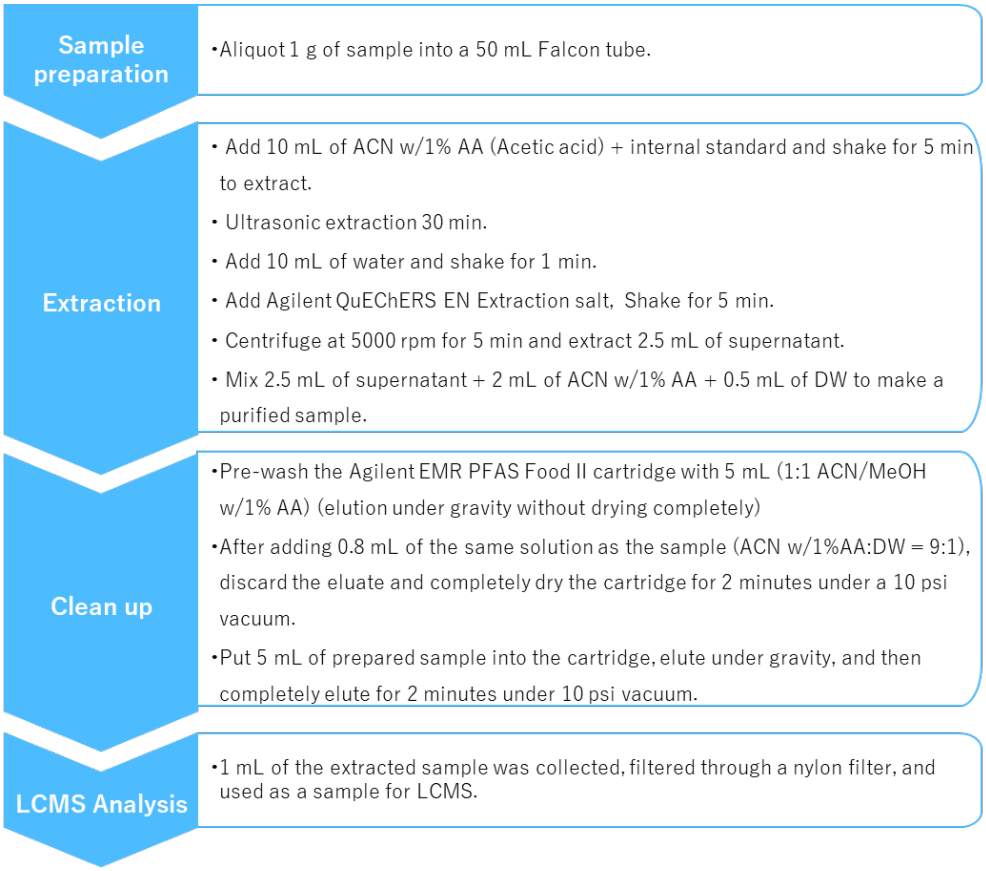


Figure 4-17. Sample preparation procedure for preparing cosmetic products using QuEChERS-EMR workflow.

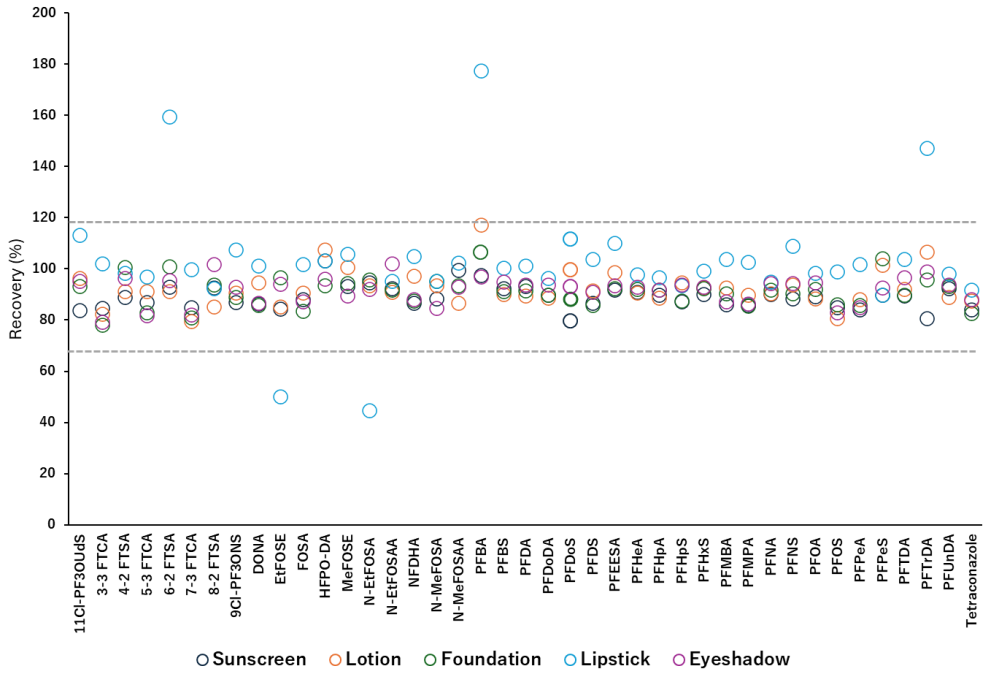


Figure 4-18. Recovery of 41 PFAS analytes in five types of cosmetic products. PFAS standard was fortified at 1 ng/g in matrix blanks and tested with 15 samples (5 types × 3 manufacturers per type).

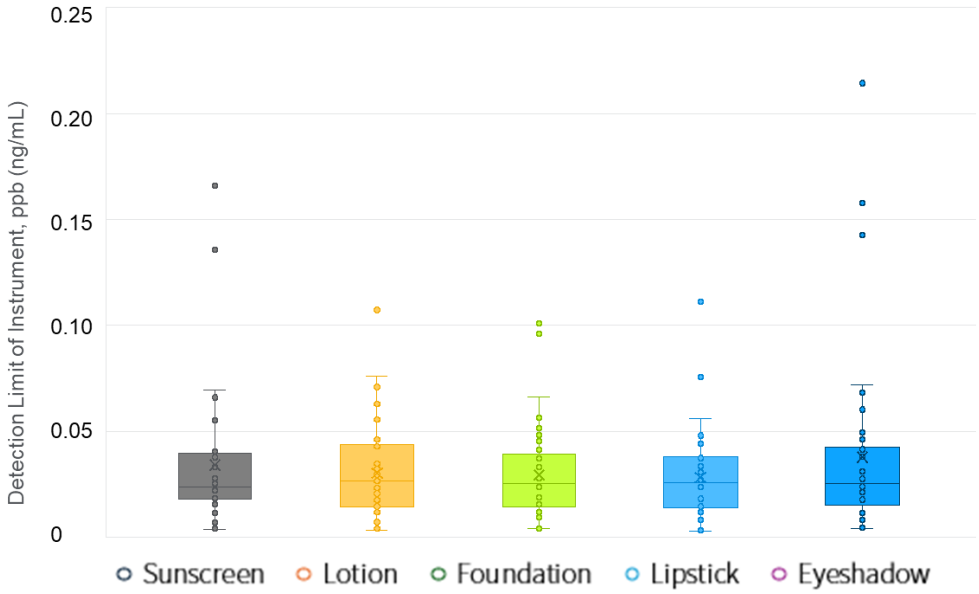


Figure 4-19. Detection limits range for PFAS targets in five types of cosmetic.

Application highlights

- A simplified and reliable method was developed for the quantitative determination of 41 PFAS compounds in five types of cosmetics using QuEChERS extraction and Captiva EMR PFAS Food II cleanup, followed by LC/MS/MS detection.
- The sample preparation workflow is simple, robust, and cost-effective, resulting in significant savings in both time and resources.
- The method demonstrates acceptable targets recovery and method detection limit in five common cosmetics matrices–sunscreen, lotion, foundation, lipstick and eyeshadow.

Multiclass Multiresidue Mycotoxins Analysis in Food and Feed

Introduction

Mycotoxins are toxic fungal metabolites that can contaminate a wide range of crops, foods, and feeds, posing significant risks to human and animal health. Regulatory agencies worldwide enforce strict limits, often at low-ppb levels, requiring laboratories to reliably quantify multiple chemically diverse mycotoxins across complex matrices.

The applications in this chapter demonstrate EMR-based sample preparation strategies that support robust LC/MS/MS analysis by improving matrix cleanup while maintaining recovery for challenging mycotoxin classes, including:

- Multiclass multiresidue analysis of regulated mycotoxins in food and feed matrices
- Reduced matrix effects and improved extract cleanliness for sensitive and structurally diverse mycotoxins
- Six application notes for multiclass multiresidue mycotoxins analysis

Analyte - Mycotoxins		
Sample Category	**Sample Matrix**	**Application Highlight**
Fresh Fruits, Vegetables	Olives	5994-6101EN
Dairy	Cheese	5991-8694EN
Infant Formula, Baby Food	Infant formula	5994-0365EN
Dry Plant Material	Dry corn, dry soybean	5994-7373EN
Nuts, Nut Butter	Peanut butter	5994-0366EN
Pet Food	Dog food, cat food	5994-7471EN

Application note highlight

Determination of Multiclass Multiresidue Mycotoxins in Dry Corn Kernels and Soybeans

Application note: 5994-7373EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	21 mycotoxins
Sample Matrix	Corn, soybean
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6475 LC/TQ – Agilent ZORBAX Rapid Resolution High Definition (RRHD) Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm column (p/n 959758-902)
Sample Preparation Method	QuEChERS extraction (acidified), plus mixed-mode passthrough cleanup on Agilent Captiva EMR Mycotoxins cartridge
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR Mycotoxins cartridges, 3 mL cartridges, 300 mg (p/n 5610-2233)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– For corn: 0.01 to 20 ng/g with exceptions: – 75 ng/g for DON, FS-X, 15-ADON – 50 ng/g for NEO, 3-ADON, DAS – For soybean: 0.04 to 20 ng/g with exceptions: – 75 ng/g for FS-X and 15-ADON
Recovery	– For corn: 79 to 121% accuracy with exceptions: 50% for AB2 in corn due to the noncorresponding ISTD used for quantitation – For soybean: 73 to 122% accuracy with exceptions: 150 to 200% for HT-2, FB3, and OTA in soybean due to the noncorresponding ISTD used for quantitation
Relative Standard Deviation	– For corn: < 18% RSD – For soybean: < 14% RSD
Method Calibration	– Neat calibration curve, stable labeled ISTD used – 500× dynamic calibration range with variations on a few targets: – 250× dynamic calibration range for 15-ADON and AG2 – 100× dynamic calibration range for FB1 and FB3 – $R^2 > 0.99$
Matrix Removal or Matrix Effect	> 90% of matrix co-extractives reduction compared to traditional method

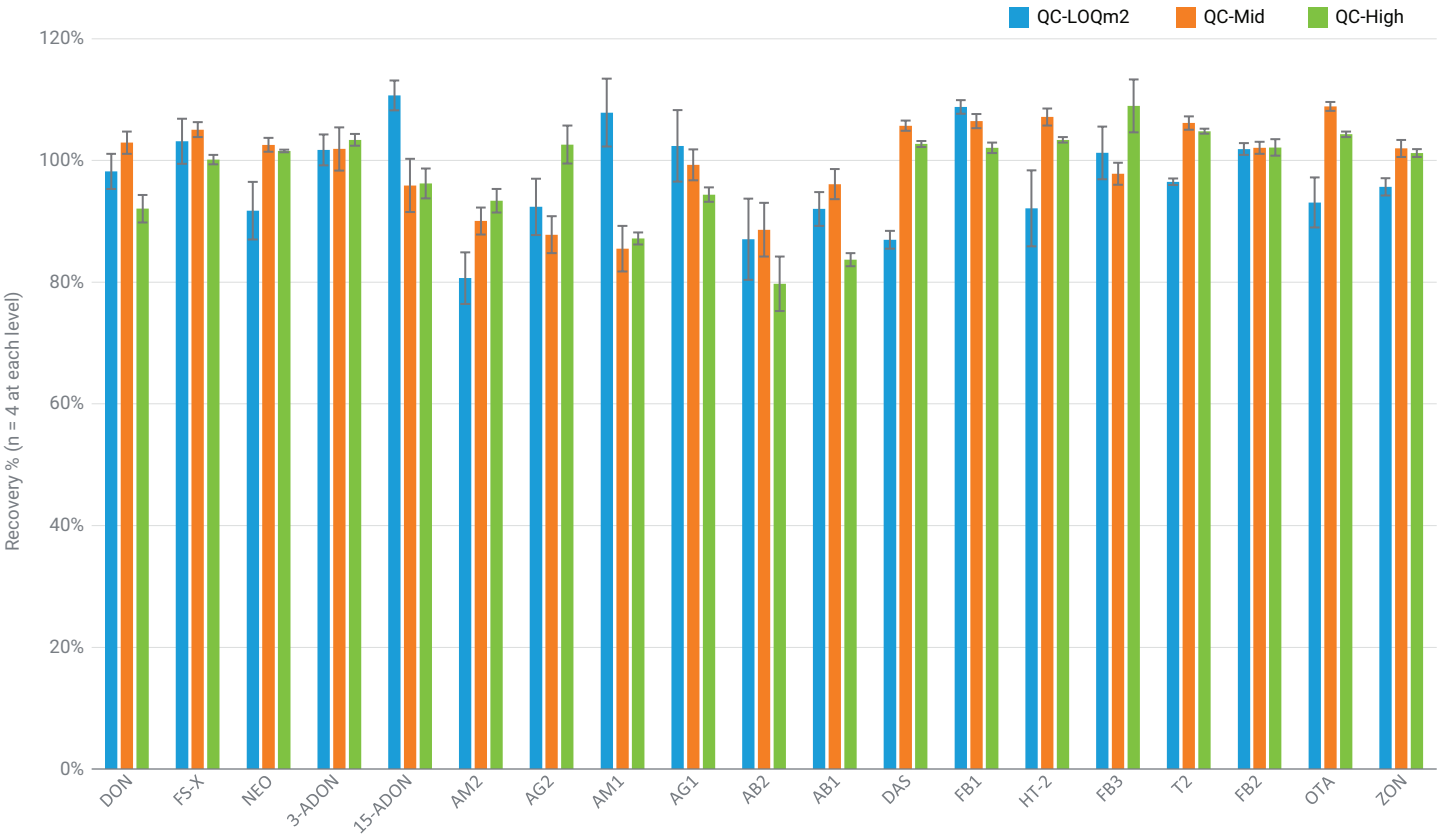


Figure 5-10. Mycotoxin recovery in dry corn extracted by QuEChERS extraction followed with EMR mixed-mode passthrough cleanup using Agilent Captiva EMR Mycotoxins cartridge.

Note: Refer to Table 1 in Agilent application note 5994-7373EN for sample QC concentrations.

Application highlights

- Simple, rapid, and reliable method using QuEChERS extraction followed by cleanup with an Agilent Captiva EMR Mycotoxins cartridge was developed and validated for 21 mycotoxins in dry corn kernels and soybeans.
- The method provides significant improvement over the SIDA method for matrix removal and mycotoxins recovery compared to other commercial SPE and dSPE products.
- The method saves time and effort, improving overall lab productivity.
- The sample preparation procedure is extendable to mycotoxins analysis in other seeded dry feed and complex processed food matrices.

Application note highlight

Determination of Multiclass Multiresidue Mycotoxins in Pet Food

Application note: 5994-7471EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	21 mycotoxins
Sample Matrix	Pet foods
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6475 LC/TQ – Agilent ZORBAX Rapid Resolution High Definition (RRHD) Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm column (p/n 959758-902)
Sample Preparation Method	QuEChERS extraction (acidified), plus mixed-mode passthrough cleanup on Agilent Captiva EMR Mycotoxins cartridge
Sample Preparation Products	– Agilent Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (p/n 5982-5650CH) – Agilent Captiva EMR Mycotoxins cartridges, 6 mL cartridges, 600 mg (p/n 5610-2234)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– For cat food: 0.04 to 20 ng/g with a few exceptions: – 375 ng/g for DON – 75 ng/g for 15-ADON – For dog food: 0.1 to 20 ng/g with a few exceptions: – 75 ng/g for DON and FS-X – 375 ng/g for 15-ADON – 100 ng/g for FB1
Recovery	– For cat food: 79 to 134% accuracy – 177% for OTA due to the noncorresponding ISTD used for quantitation – For dog food: 71 to 128% accuracy – 45% for FB3 due to the noncorresponding ISTD used for quantitation
Relative Standard Deviation	– For cat food: < 14% RSD – For dog food: < 12% RSD
Method Calibration	– Neat calibration curve, stable labeled ISTD used – 500× dynamic calibration range with variations on a few targets: – 250× dynamic calibration range for 15-ADON and AG2 – 100× dynamic calibration range for FB1 and FB3 – $R^2 > 0.99$
Matrix Removal or Matrix Effect	> 90% of matrix co-extractives reduction compared to traditional method

Application highlights

- A simple, rapid, and reliable method using QuEChERS extraction followed by cleanup with an Agilent Captiva EMR Mycotoxins cartridge was developed and validated for 21 mycotoxins in pet food.
- The method offers significant improvement over the SIDA method for matrix removal and mycotoxins recovery and repeatability.
- The method saves time and effort, improving overall lab productivity.
- The sample preparation procedure is extendable to mycotoxins analysis in other seeded dry feed and complex processed food matrices.

Application note highlight

Multiclass Mycotoxin Analysis in Cheese Using Agilent Captiva EMR–Lipid Cleanup and LC/MS/MS

Application note: 5991-8694EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	13 mycotoxins
Sample Matrix	Blue and parmesan cheese
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6460 LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 2.1 × 100 mm, 2.7 µm column (p/n 695775-902)
Sample Preparation Method	QuEChERS extraction (acidified), plus mixed-mode passthrough cleanup on Agilent Captiva EMR Mycotoxins cartridge
Sample Preparation Products	– Agilent QuEChERS original extraction salts (p/n 5982-5555) – Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.5 to 5 ng/g in cheese matrix for all targets
Recovery	71 to 112% recovery
Relative Standard Deviation	– For cat food: < 14% RSD – For dog food: < 12% RSD
Method Calibration	– Matrix-matched calibration curve, no stable labeled ISTD used – 200× dynamic range with variation on different targets – R^2 > 0.990 for all targets
Matrix Removal or Matrix Effect	– 60% reduced GC/MS full scan background – 90% reduced phospholipids background – 50 to 70% less matrix residue

Application highlights

- Agilent Captiva EMR–Lipid provides an easy and effective cleanup option for multiclass mycotoxins analysis in high-fat cheese matrix.
- Significantly higher recovery achieved using Captiva EMR–Lipid than other commercially available cleanup products.
- Method validation in blue cheese and parmesan cheese gave excellent recovery (70.7 to 111.8%), precision (< 20%), and sensitivity down to 0.5 ng/g.
- The method is ideal for laboratories looking to simplify sample preparation while improving method performance.

Table 5-2. Recovery and precision results for 13 mycotoxins in blue and parmesan cheeses (n = 5).

Analyte	LQ		MQ		HQ	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Parmesan Cheese						
AF-M1	111.8	1.5	95.6	5.9	96.3	1.7
AF-G2	101.8	2.2	98.5	3.8	104.6	3.2
AF-G1	102.2	2.8	89.1	2.2	93.9	6.6
AF-B2	108.5	1.5	101.5	4.2	103.5	2.4
AF-B1	103.2	5.1	84.9	2.7	90.3	2.9
FB1	79.4	6.7	71.3	3.2	74.2	2.2
OTB	109.6	1.7	98.5	7.2	106.0	1.8
MPA	111.3	8.6	103.6	2.1	107.5	4.6
FB3	98.2	7.1	90.6	8.1	92.0	5.0
ZON	98.0	7.8	85.8	4.0	88.2	2.8
FB2	101.9	5.5	92.4	7.8	95.6	3.8
OTA	104.7	10.4	89.4	5.7	92.6	2.5
STC	85.4	3.4	70.7	2.3	75.7	2.5
Blue Cheese						
AF-M1	97.0	17.8	101.2	8.8	107.9	5.8
AF-G2	88.6	12.4	96.1	6.3	98.3	8.6
AF-G1	91.8	9.1	97.5	2.5	105.5	3.2
AF-B2	98.2	13.8	99.7	8.8	108.5	8.1
AF-B1	91.8	7.9	93.5	5.7	102.4	6.2
FB1	103.9	7.9	83.5	5.4	85.3	5.8
OTB	81.5	7.1	79.9	3.9	89.0	5.8
MPA	92.4	10.3	95.0	1.8	95.4	8.0
FB3	101.9	5.7	93.9	5.0	94.3	7.7
ZON	76.1	3.9	83.3	9.6	90.2	9.3
FB2	102.0	4.7	100.6	5.9	99.4	3.9
OTA	89.0	3.4	82.5	7.9	84.9	5.5
STC	100.0	3.0	74.3	13.4	70.9	6.8

Note: Refer to Table 1 in Agilent application note 5991-8694EN for sample QC concentrations.

Application note highlight

Mycotoxin Analysis in Infant Formula Using Captiva EMR–Lipid Cleanup and LC/MS/MS

Application note: 5994-0365EN

Application note highlight

Mycotoxin Analysis in Peanut Butter Using Captiva EMR–Lipid Cleanup and LC/MS/MS

Application note: 5994-0366EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	13 mycotoxins
Sample Matrix	Infant formula, peanut butter
Instrument, Detection, and Critical Consumables	– Agilent 1290 Infinity II LC with Agilent 6490 LC/TQ – Agilent InfinityLab Poroshell 120 EC-C18, 2.1 × 100 mm, 2.7 µm column (p/n 695775-902)
Sample Preparation Method	QuEChERS extraction (acidified), plus mixed-mode passthrough cleanup on Agilent Captiva EMR Mycotoxins cartridge
Sample Preparation Products	– Agilent QuEChERS original extraction salts (p/n 5982-5555) – Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	1 to 10 ng/g in infant formula and peanut butter matrices
Recovery	– 70 to 107% for all targets in infant formula – 79 to 119% for all targets in peanut butter
Relative Standard Deviation	– < 18% RSD for infant formula – < 17% RSD for peanut butter
Method Calibration	– Matrix-matched calibration curve, no stable labeled ISTD used – 200× dynamic range with variation on different targets – $R^2 > 0.992$
Matrix Removal or Matrix Effect	– > 90% GC/MS full scan background reduction for infant formula matrix – > 60% GC/MS full scan background reduction for peanut butter matrix

Application highlights

- Agilent Captiva EMR–Lipid is an easy and effective cleanup option for multiclass mycotoxins analysis in high-fat infant formula and peanut butter matrices.
- Efficient cleanup was demonstrated through GC/MS full scans.
- Significantly higher recovery of mycotoxins was possible using Captiva EMR–Lipid compared to other commercially available cleanup products.
- The method is ideal for laboratories looking to simplify sample preparation while improving method performance.
- The method is extendable to other high-fat food matrices such as olives, as shown in Agilent application note 5994-6101EN.

Multiresidue PAHs Analysis in Food and Other Applications

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are toxic, hydrophobic compounds that can be introduced into foods through environmental exposure or thermal processing and tend to accumulate in fatty matrices such as fish, meat, oils, and dairy products. Regulatory limits at low-ppb levels require sensitive GC/MS or GC/MS/MS analysis supported by highly effective lipid and fat removal during sample preparation.

The applications in this compendium demonstrate EMR-based sample preparation approaches that support reliable analysis of PAHs and other challenging hydrophobic contaminants, including:

- Low-level determination of regulated PAHs in fatty and processed food matrices
- Efficient lipid and fat removal to improve extract cleanliness and protect GC/MS system performance
- Four Application notes for multiresidue PAHs analysis
- Other applications include THC and CBD analysis in chocolate and UV filters from personal care products

Analyte – PAHs and Other Applications			
Sample Category	**Analyte**	**Sample Matrix**	**Application Highlight**
Infant Formula, Baby Food	PAHs	Infant formula	5994-5560EN
Edible Oil	PAHs	Pumpkin seed, olive, avocado, almond, grape seed oil	5994-1483EN
Fish, Shellfish	PAHs	Salmon	5994-0553EN
Meat	PAHs	Beef	5994-0553EN
Confectionery	CBD, THC	Chocolate	5994-2873EN
Cosmetics, Personal Care Products	UV filters	Sunscreen, lipstick and lotion	5994-1611EN

Application note highlight

Determination of 19 Polycyclic Aromatic Hydrocarbon Compounds in Salmon and Beef

Application note: 5994-0553EN

Regulation or official guideline? European Commission Regulation (EU) No. 836/2011

Method summary

Method Parameter	Setting
Analytes	19 PAHs
Sample Matrix	Salmon, beef
Instrument, Detection, and Critical Consumables	– Agilent 7890B GC with Agilent 7000D GC/TQ – Agilent J&W DB-EUPAH, 30 m × 0.25 mm, 0.25 µm (p/n 122-9632) – Agilent Ultra Inert liner, 4 mm id, single taper with wool (p/n 5190-2293)
Sample Preparation Method	Solvent extraction with 20:80 EtOAc:ACN, followed by Captiva EMR–Lipid passthrough cleanup, then isooctane back-extraction
Sample Preparation Product(s)	Agilent Captiva EMR–Lipid cartridge, 3 mL, 300 mg (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	– 1 ng/g in salmon and beef for all targets – Lowest tested concentration was 1 ng/g; high S/N ratios indicate lower LOQs are possible
Recovery	– 57 to 103% recovery in salmon – 64 to 107% recovery in beef
Relative Standard Deviation	– < 13.9% RSD in salmon – < 11.1% RSD in beef
Method Calibration	– Neat calibration standard curves with isotopic ISTDs – 1 (2) to 500 ng/g in salmon and beef – R^2 > 0.993 for all 19 PAH targets
Matrix Removal or Matrix Effect	– > 60% of matrix residue removal for salmon – > 90% of matrix residue removal for beef

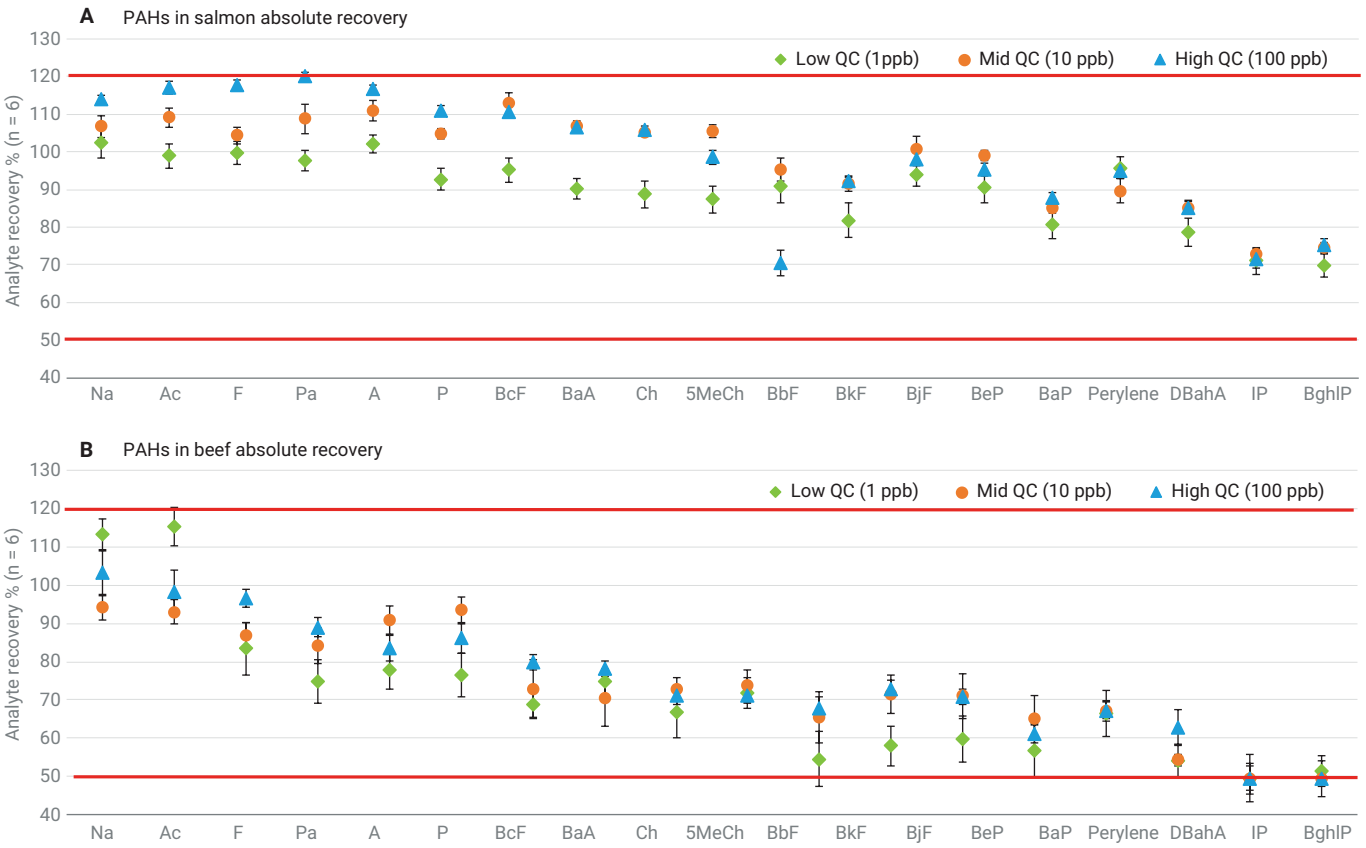


Figure 6-8. PAH recovery in (A) salmon and (B) beef using the optimized sample preparation procedure.

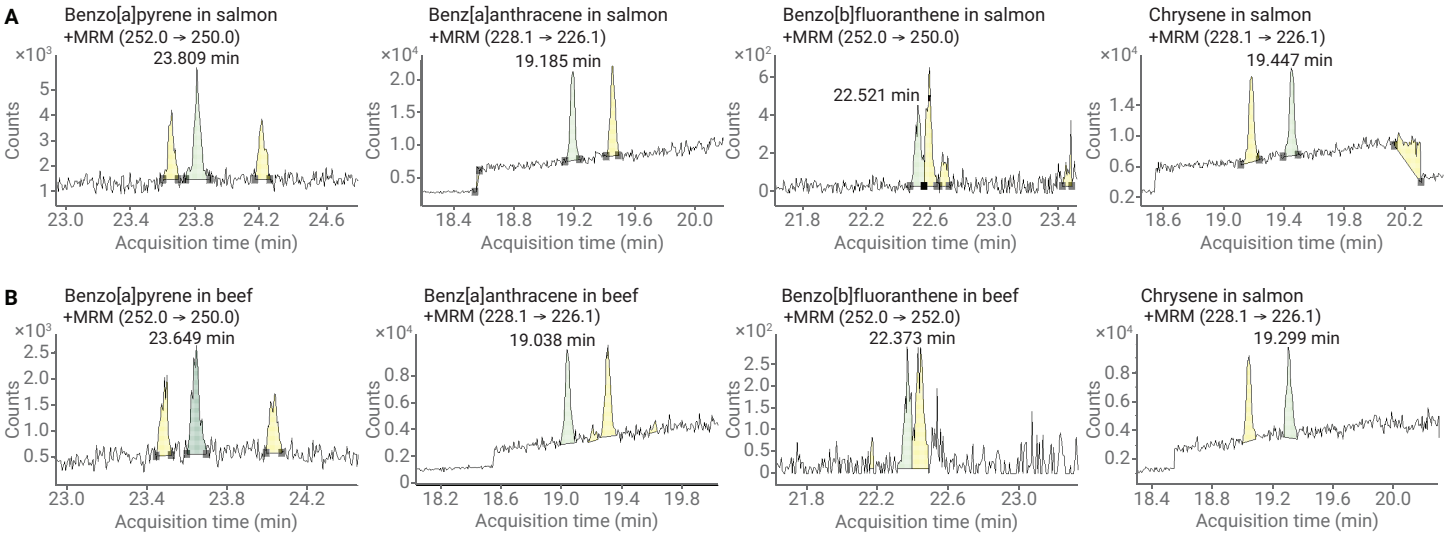


Figure 6-9. Critical PAH targets chromatograms at LOQ of 1 ng/g in (A) salmon and (B) beef.

Application highlights

- A simple, rugged, and reliable method was developed and validated for the multiresidue analysis of PAHs in salmon and beef.
- The method was optimized to improve the extraction efficiency and complete elution on an Agilent Captiva EMR–Lipid cartridge, followed by an isooctane back-extraction for water removal and solvent swamping.
- Results showed that all the tested PAH compounds achieved acceptable recovery results based on European Commission regulation (recoveries of 50 to 120%), RSD < 20 %, and calibration curves from 1 to 500 ng/g in salmon and beef with $R^2 > 0.99$.

Application note highlight

Determination of 14 Polycyclic Aromatic Hydrocarbon Compounds in Edible Oil

Application note: 5994-1483EN

Regulation or official guideline? European Commission Regulation (EU) No. 836/2011

Method summary

Method Parameter	Setting
Analytes	14 Heavy PAHs
Sample Matrix	Edible oils: pumpkin seed oil, olive oil, avocado oil, almond oil, and grape seed oil
Instrument, Detection, and Critical Consumables	– Agilent 7890B GC with Agilent 7000D GC/TQ – Agilent J&W DB-EUPAH, 30 m × 0.25 mm, 0.25 µm (p/n 122-9632) – Agilent Ultra Inert liner, 4 mm id, single taper with wool (p/n 5190-2293)
Sample Preparation Method	Solvent extraction with 20:80 EtOAc:ACN, followed by hyphenated Captiva EMR–Lipid and Bond Elut Jr PSA passthrough cleanup, then isooctane back-extraction
Sample Preparation Products	– Agilent Captiva EMR–Lipid cartridge, 6 mL, 600 mg (p/n 5190-1004) – Agilent Bond Elut Jr PSA, 500 mg (p/n 12162042B)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.9 ng/g in edible oil for all targets
Recovery	84 to 113% Recovery in edible oil
Relative Standard Deviation	< 15% RSD in edible oil
Method Calibration	– Neat calibration standard curves with isotopic ISTDs – 0.9 to 200 ng/g in edible oil – R^2 > 0.987 for all 14 PAH targets
Matrix Removal or Matrix Effect	> 95% Matrix residue removal

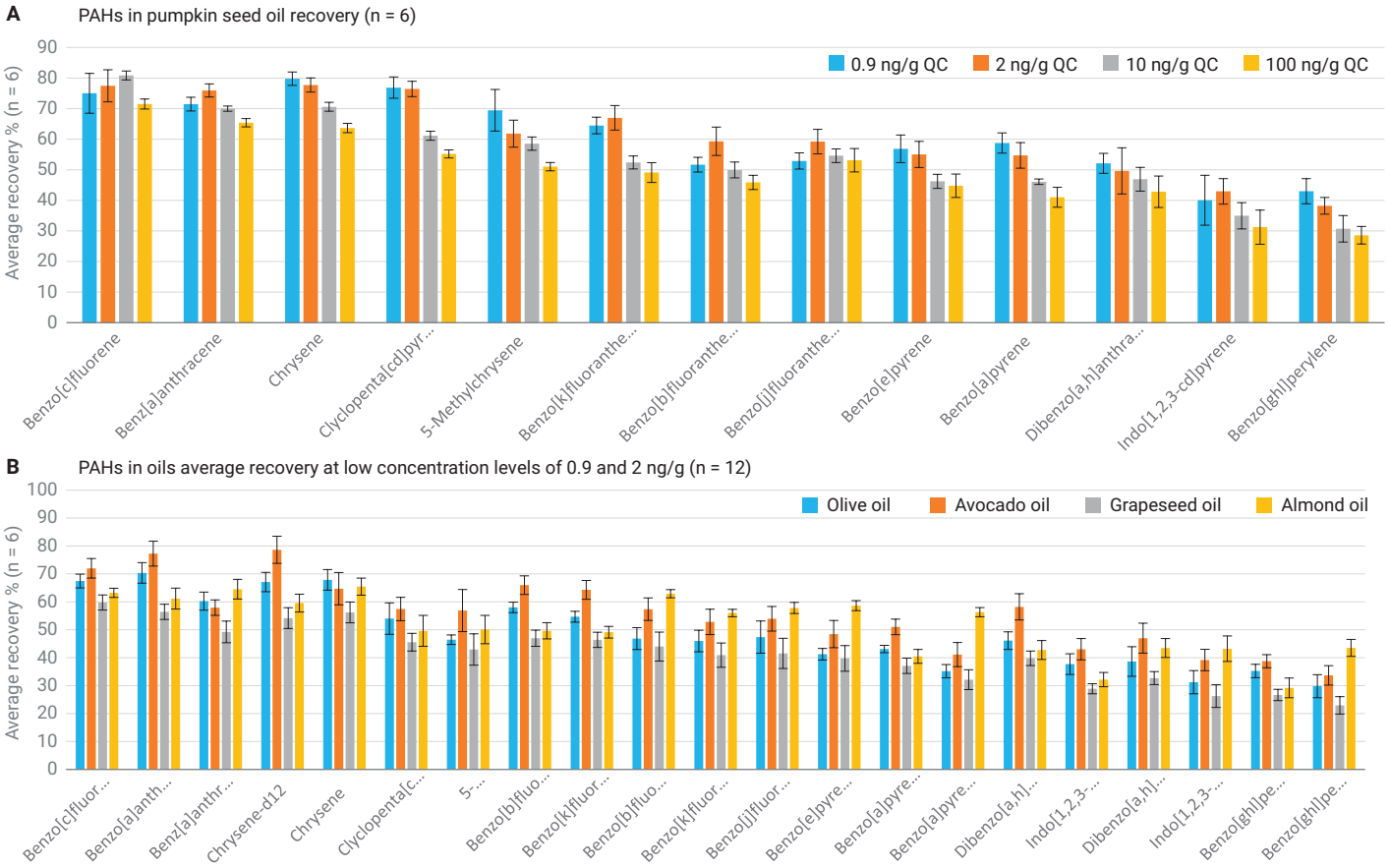


Figure 6-10. Heavy PAH compounds recoveries from edible oils. (A) recovery data in pumpkin seed oil at four different spiking levels, (B) average recovery data in other four oils at two low spiking levels.

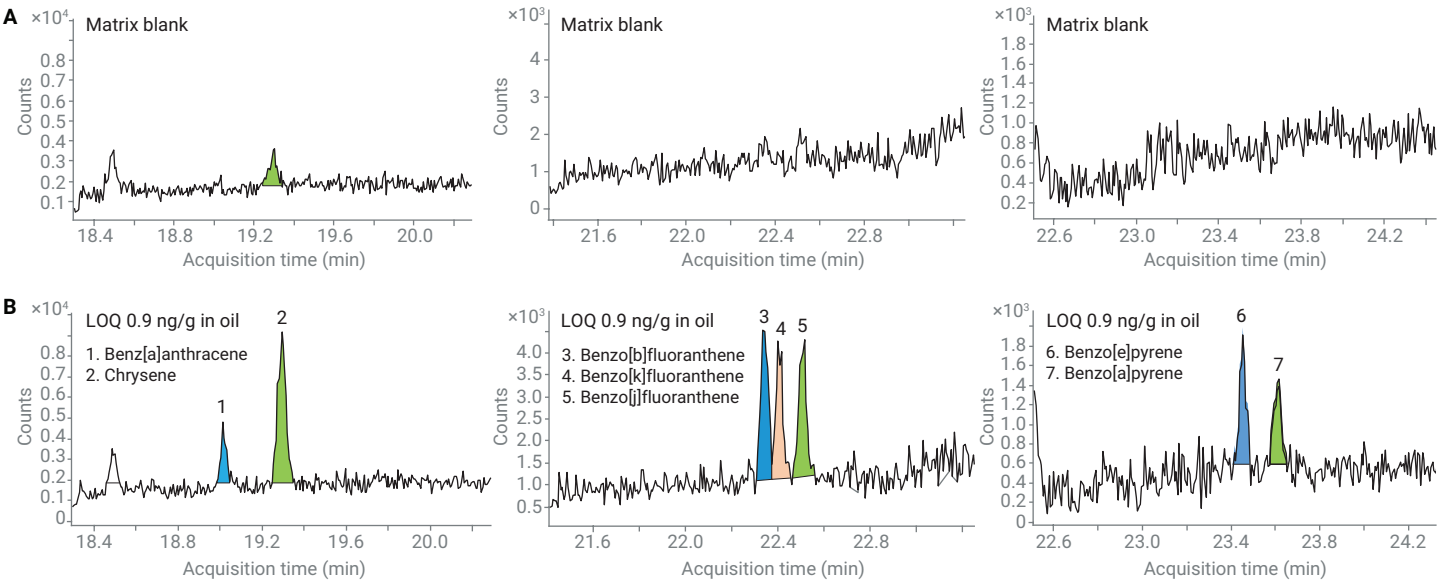


Figure 6-11. Separation observed for EU Commission-monitored PAHs. MRM chromatograms are for (A) pumpkin seed oil matrix blanks, and (B) LOQ sample at 0.9 ng/g in pumpkin seed oil.

Application highlights

- A simple, rugged, and reliable method using liquid-liquid extraction followed by Agilent Captiva EMR–Lipid hyphenated with Bond Elut Jr PSA cartridge cleanup was developed and validated for the analysis of heavy PAHs in edible oil.
- Oil matrix co-extractive residue provided > 95% removal with significantly cleaner chromatographic backgrounds.
- The method provided the desired limit of quantitation (LOQ) (0.9 to 2 ng/g) required by the European Commission regulation, with acceptable quantitation, accuracy, and precision results.

Application note highlight

Extraction and Analysis of Polycyclic Aromatic Hydrocarbons in Infant Formula

Application note: 5994-5560EN

Regulation or official guideline? European Commission Regulation (EU) No. 836/2011

Method summary

Method Parameter	Setting
Analytes	8 PAHs
Sample Matrix	Infant formula
Instrument, Detection, and Critical Consumables	– Agilent 8890 GC with Agilent 5977C Inert Plus GC/MSD – Agilent J&W DB-EUPAH GC column, 20 m × 0.18 mm, 0.14 µm, 7-inch cage (p/n 121-9627) – Agilent HydroInert source with 9 mm HydroInert extraction lens (p/n G7078-67930) Hydrogen carrier gas
Sample Preparation Method	Solvent extraction with 20:80 EtOAc:ACN, followed by Captiva EMR–Lipid passthrough cleanup, then isooctane back-extraction
Sample Preparation Product	Agilent Captiva EMR–Lipid 3 mL cartridge (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.5 ng/g in infant formula
Recovery	– 65 to 95% for all PAH targets except benzo[k]fluoranthene – Benzo[k]fluoranthene at 1 ng/g (54% recovery)
Relative Standard Deviation	– < 20% except for all PAH targets except benzo[ghi]perylene – Benzo[ghi]perylene (34.6% RSD)
Method Calibration	– Neat calibration standard curves – 0.5 to 100 ng/g in infant formula – R^2 > 0.99 for all PAH targets

Application highlights

- A sample preparation method for the extraction and cleanup of polycyclic aromatic hydrocarbons (PAHs) from infant formula.
- Agilent Captiva Enhanced Matrix Removal–Lipid (EMR–Lipid) provides highly selective, efficient lipid removal from the infant formula with acceptable analyte recoveries.
- Hydrogen (H_2) carrier gas is used with the Agilent HydroInert source on the 5977C GC/MSD.
- The H_2 carrier provides a more sustainable, economical alternative to helium carrier gas, while the HydroInert source prevents hydrogenation and dichlorination reactions to preserve GC/MSD performance.

Application note highlight

Simple and Accurate Quantification of THC and CBD in Cannabis-Infused Chocolate Edibles

Application note: 5994-2873EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	CBD, THC
Sample Matrix	Chocolate (milk, dark, and white)
Instrument, Detection, and Critical Consumables	– Agilent 1260 Infinity II LC with Agilent 6545 LC/Q-TOF – Agilent InfinityLab Poroshell 120 EC-C18, 3.0 × 50 mm, 2.7 µm (p/n 699975-302)
Sample Preparation Method	Solvent extraction with cold ACN with 2% formic acid, followed by Agilent Captiva EMR–lipid passthrough cleanup, with additional elution by 80:20 ACN:water
Sample Preparation Product	Agilent Captiva EMR–Lipid, 3 mL (p/n 5190-1003)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.5 µg/mL in chocolate extract
Recovery	104 to 108% for both targets
Relative Standard Deviation	< 10% RSD
Method Calibration	– Neat calibration standard curves – 0.5 to 100 µg/mL in chocolate extract – R^2 > 0.999 for CBD and THC in chocolate
Matrix Removal or Matrix Effect	Cleaner chromatographic background on LC/Q-TOF when using Agilent Captiva EMR–Lipid cleanup

Application highlights

- Fast sample preparation resulting in increased sample throughput.
- Agilent Captiva EMR–Lipid provided high lipid removal from chocolate matrices compared to other common preparation techniques for high lipid content, resulting in a higher LC/UV signal for THC and CBD.
- A short LC gradient reduced solvent consumption.
- Cleaner samples resulted in less lipid accumulation on the analytical column.
- The method provides increased system uptime, lab productivity, and profitability.
- Potency testing in fatty edibles using this approach is simple, accurate, and reliable.

Application note highlight

Determination of UV Filters in Sunscreens Using Agilent Captiva EMR–Lipid Cleanup by HPLC

Application note: 5994-1611EN

Regulation or official guideline? Not mentioned

Method summary

Method Parameter	Setting
Analytes	5 UV filters: octocrylene, octinoxate, avobenzone, homosalate, and octisalate
Sample Matrix	Sunscreen, lip balm
Instrument, Detection, and Critical Consumables	– Agilent 1260 Infinity II LC – Agilent InfinityLab Poroshell 120 EC-C18 column, 3.0 mm × 50 mm, 2.7 µm (p/n 699975-302) – Agilent InfinityLab Poroshell 120 EC-C18 guard column, 2.1 mm × 5 mm, 2.7 µm (p/n 821725-911)
Sample Preparation Method	Solvent extraction with hot water, then IPA, followed by Agilent Captiva EMR–Lipid passthrough cleanup
Sample Preparation Product	Agilent Captiva EMR–Lipid, 6 mL, cartridges (p/n 5190-1004)

Results summary

Evaluation Parameter	Results
Limits of Quantitation	0.125 mg/mL in sunscreen and lip balm
Recovery	95 to 104%
Relative Standard Deviation	< 2.19%
Method Calibration	– Neat calibration standard curves – 0.125 to 2 mg/mL – R^2 > 0.999 for all targets
Matrix Removal or Matrix Effect	LC/UV chromatography was significantly improved by Agilent Captiva EMR–Lipid cleanup, including RT consistency, baseline, and target response repeatability

Table 6-3. Average recovery (%), standard deviation, and %RSD of UV filters extracted from seven different OTC product samples using Agilent Captiva EMR–Lipid cleanup (n = 6 per product).

Sunscreen Lotion A	Octocrylene	Octinoxate	Avobenzone	Homosalate	Octisalate
Average Recovery (%)	98.67	97.63	100.22	98.92	97.63
Standard Deviation	2.16	1.94	2.20	2.12	1.97
%RSD	2.18	1.99	2.19	2.15	2.02
Sunscreen Lotion B	**Octocrylene**	**Octinoxate**	**Avobenzone**	**Homosalate**	
Average Recovery (%)	97.25	97.71	99.39	100.46	
Standard Deviation	1.58	1.47	1.48	1.62	
%RSD	1.63	1.50	1.49	1.62	
Sunscreen Lotion C	**Octocrylene**	**Avobenzone**	**Homosalate**	**Octisalate**	
Average Recovery (%)	98.55	97.5	95.58	100.13	
Standard Deviation	0.60	0.62	0.58	0.49	
%RSD	0.60	0.64	0.61	0.49	
Sunscreen Lotion D	**Octocrylene**	**Octinoxate**	**Avobenzone**	**Homosalate**	
Average Recovery (%)	99.46	98.76	100.14	101.01	
Standard Deviation	0.72	1.26	1.35	1.48	
%RSD	0.73	1.28	1.34	1.46	
Sunscreen Lotion E	**Octocrylene**	**Avobenzone**	**Homosalate**	**Octisalate**	
Average Recovery (%)	100.46	100.28	99.04	100.63	
Standard Deviation	1.52	1.82	1.17	0.95	
%RSD	1.51	1.81	1.19	0.94	

Sunscreen Lotion F	Octinoxate	Octisalate
Average Recovery (%)	98.18	95.25
Standard Deviation	0.26	0.20
%RSD	0.26	0.21
Lip Balm	**Octinoxate**	**Avobenzone**
Average Recovery (%)	95.68	99.52
Standard Deviation	0.70	0.71
%RSD	0.73	0.72

Application highlights

- A robust, validated method was developed to prepare samples followed by quantitative analysis of the active UV filter ingredients in suncare products.
- Agilent Captiva EMR–Lipid cleanup selectively removed matrix lipids from the crude extracts of six different sunscreen lotions and a lip balm without interfering with the recovery of UV-filtering agents.
- Captiva EMR–Lipid cleanup sample preparation enabled the sequential, robust analysis of multiple sunscreen and lip balm samples on the same column and increased the total number of effective injections by > 200%.
- The method can be used to improve experimental efficiency, data reproducibility, and cost-effectiveness of OTC testing in cosmetics research, product development, and quality control.

Disclaimer

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

Learn more: For comprehensive EMR methodology guidance—including method development principles, product selection, performance evaluations, and practical tips—refer to the Captiva EMR Reference Guide, the technical companion to the applications presented in this compendium.

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