Application Note

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

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Abstract

This Application Note presents the development and validation of a multiresidue method for the analysis of pesticide residues in olive oil. Olive oil samples were extracted using liquid-liquid extraction (LLE) followed by Captiva EMR—Lipid cleanup. The cleaned sample eluent was then dried with anhydrous $MgSO_4$ prior to GC/MS/MS analysis. 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, and the developed method was verified in olive oil. The results showed that all of the pesticides achieved satisfactory recovery results (recoveries of 60 to 120 %). Over 96 % of the pesticides were identified with 70 to 120 % accuracy, RSD <20 %, and calibration curves from 1 to 500 ng/g in olive oil with R^2 >0.99. The matrix co-extractive residue removal efficiency measured by gravity was 85 % in olive oils. This was significantly higher than that obtained using conventional PSA/C18 dispersive solid phase extraction (dSPE) cleanup (55 %).

Introduction

Olive oil is one of the important cooking oils used in daily life. Modern agricultural practices use many pesticides to control pests and diseases and increase crop yields. Olive crops are usually treated with insecticides such as organophosphorus, organochlorine, carbamate, and so forth, or fungicides such as phthalimides, triazines, sulphamide, and so forth. Thus, pesticide residues may occur in the final olive oil products. Pesticide use is subject to strict regulations, especially concerning residual levels in commercial goods. The European Union (EU)[1], the Codex Alimentarius Commission of the Food and Agriculture Organization (FAO), and the World Health Organization (WHO)[2] have established maximum residue limits (MRLs) in oils for a large number of pesticides. The MRL list can be searched online at https://www.globalmrl.com. The MRLs in olive and olive oil are different for various pesticides, but are usually higher than 10 µg/kg. In addition, the new emerging field of organic food requires the testing methods to analyze pesticide residue at very low levels (such as 0.5–2 µg/kg)[3].

The development of multiresidue methods for the determination of fat-soluble pesticides in edible oil at low levels is still a challenging issue. Much effort has been invested in extraction of hydrophobic pesticides from sample matrices and cleanup of lipids prior to analysis. Matrix co-extractives deposit on the instrument flowpath, decreasing analyte sensitivity by degrading the flowpath inertness. An efficient cleanup of the oil extract is necessary to improve column lifetime and reduce instrument maintenance frequency. It can also be particularly challenging to remove interfering lipids without losing lipophilic pesticide classes such as organochlorine and pyrethyroid.

Agilent Enhanced Matrix Removal—Lipid (EMR—Lipid) dSPE cleanup has gained much attention since it was introduced in 2015. The EMR—Lipid dSPE sorbent selectively interacts with the unbranched hydrocarbon chains of lipids, leaving bulky target analytes in solution for subsequent analysis. This makes it ideal for multiclass, multiresidue analysis. Previous studies investigating multiclass pesticides analysis and veterinary drugs in fatty matrices provided high matrix cleanup as well as high analyte recovery and precision[4-6] in samples including edible oils[7]. The Captiva EMR—Lipid sorbent reduces the water percentage needed for sorbent activation, and reduce the difficulty of water removal after EMR—Lipid cleanup. This simplifies the workflow, and improves the solubility of hydrophobic compounds during cleanup[8].

This study investigates sample preparation using Captiva EMR—Lipid cartridge pass-through cleanup for the analysis of 26 representative pesticides in olive oils by GC/MS/MS. Table 1 shows the classification, Log P value, retention time, MS/MS transitions, and collection window for the pesticides tested.

Experimental

Chemicals and reagents

All pesticide compounds and IS chemicals were purchased from Sigma-Aldrich (St Louis, MO, USA). HPLC grade acetonitrile (ACN), methanol (MeOH), and ethyl acetate (EtOAc) were from Honeywell (Muskegon, MI, USA). Reagent grade dimethyl sulfoxide (DMSO) was from Sigma-Aldrich (St. Louis, MO, USA).

Solutions and standards

The standard and IS stock solutions were prepared in MeOH or ACN or DMSO at 2.0 mg/mL, and stored in amber glass vials.

The individual stock solutions were stored at –20 °C in a freezer for three months. The stock solutions were warmed to room temperature, sonicated before use, and returned after use.

A combined standard working solution containing 26 pesticide compounds was prepared in acetone at 20 µg/mL. A combined IS working solution containing three IS compounds was prepared in EtOAc at 25 µg/mL. Both working solutions were stored in amber glass vials in a refrigerator at 4 °C for one month.

The 20:80 EtOAc/ACN extraction solvent was prepared by mixing 100 mL of EtOAc with 400 mL of ACN, and stored at room temperature. The 80:20 ACN/water elution solution was prepared by mixing 200 mL of ACN and 50 mL of water, and stored at room temperature.

Equipment and materials

The study was performed using an Agilent 7890B GC coupled with an Agilent 7010A triple quadrupole GC/MS. The GC system was equipped with an electronic pneumatic control (EPC), a multimode inlet (MMI) with air cooling, an Agilent 7693A automatic liquid sampler, and a backflushing system based on a purged ultimate union controlled by an AUX EPC module. Agilent MassHunter workstation software was used for data acquisition and analysis.

Other equipment used for sample preparation included:

- Centra CL3R centrifuge (Thermo IEC, MA, USA)
- Multi Reax Test Tube Shaker (Heidolph, Schwabach, Germany)
- Pipettes and repeater (Eppendorf, NY, USA)
- Agilent positive pressure manifold 48 processor (PPM-48) (p/n 5191-4101)
- 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)

Table 1. List of selected pesticides for analysis, pesticide class, Log P value, retention time (RT), and MS/MS conditions.

Pesticide	Class	Log P	RT (min)	First MRM transition (*m/z*)	CE (V)	Second MRM transition (*m/z*)	CE (V)
Dichlorvos	Organophosphate	1.9	4.77	184.9 → 93.0	10	109.0 → 79.0	5
Trichlorfon	Organophosphate	0.4	6.00	110.8 → 47	30	81.8 → 47.0	50
2- Phenylphenol	Phenol	3.2	6.37	170.0 → 141.1	25	169.0 → 115.1	25
Ethalfluralin	Dinitroaniline	5.1	7.25	275.9 → 202.1	15	315.9 → 275.9	10
Sulfotep	Organophosphate	4.0	7.50	237.8 → 145.9	10	201.8 → 145.9	10
Atrazine-D_5 (IS)	N/A	N/A	7.98	219.9 → 200.2	5	219.9 → 58.1	10
Lindane	Organochlorine	3.5	8.28	181.0 → 145.0	15	216.9 → 181.0	5
Diazinon	Organophosphate	3.7	8.41	199.1 → 93.0	20	137.1 → 54.0	20
Chlorothalonil	Chloronitrile	2.9	8.72	265.8 → 231.0	20	263.8 → 168.0	25
Chlorpyriphos-Me	Organophosphate	4.0	9.28	285.9 → 92.9	20	124.9 → 47.0	15
Dichlofluanid	Sulphamide	3.7	9.91	223.9 → 123.1	20	123.0 → 77.0	20
Aldrin	Organochlorine	6.5	10.09	262.9 → 192.9	35	254.0 → 220.0	35
Parathion	Organophosphate	3.8	10.11	290.9 → 109.0	10	138.9 → 109.0	5
Tolylfluanid	Sulphamide	3.9	10.78	136.9 → 91.1	20	136.9 → 65.0	30
Procymidone	Dicarboximide	3.3	10.98	284.9 → 96.0	10	282.8 → 96.0	10
Folpet	Phthalimide	3.0	10.99	259.8 → 130.1	15	261.8 → 130.1	15
Endosulfan	Organochlorine	3.7	11.43	206.9 → 172	15	194.9 → 160.0	5
Bupirimate	Pyrimidinol	3.7	11.99	272.9 → 193.1	15	272.9 → 108.0	5
Endrin	Organochlorine	3.2	12.28	316.7 → 280.8	5	244.8 → 173.0	30
DDT-D_8 (IS)	N/A	N/A	13.15	245.0 → 173.1	20	243.0 → 173.1	20
DDT	Organochlorine	6.9	13.19	235.0 → 165.2	20	237.0 → 165.2	20
TPP (IS)	N/A	N/A	13.51	325.9 → 233.0	27	325.9 → 169.0	30
Captan	Phthalimide	2.5	13.70	151.0 → 79.1	15	149.0 → 79.1	10
Captafol	Phthalimide	3.8	13.60	183.0 → 79.0	10	150.0 → 79.0	5
Iprodione	Dicarboximide	3.1	13.86	313.8 → 55.9	20	187.0 → 124.0	25
Phosmet	Organophosphate	3.0	14.06	160.0 → 133.1	20	160.0 → 77.1	20
Permethrin	Pyrethroid	6.1	15.86	183.1 → 153.1	15	183.1 → 168.1	10
Coumaphos	Organophosphate	3.9	15.99	361.9 → 109.0	10	210.0 → 182.0	10
Pyraclostrobin	Strobilurin	4.0	17.58	164.0 → 132.0	15	132.0 → 77.0	20
Deltamethrin	Pyrethroid	4.6	18.26	252.9 → 93.1	15	181.0 → 152.1	25

Instrument conditions

The GC/MS/MS instrument conditions were established based on previous published methods using equivalent instruments[9,10]. Table 2 lists the conditions of GC/MS/MS operation.

Figure 1 shows typical MRM chromatograms for multiple classes of pesticides in the fortified olive oil samples at the level of 100 ng/g using the above GC/MS/MS conditions.

Table 2. 7890B GC and 7010A GC/MS/MS conditions.

Parameter	Value
Columns	Agilent HP-5ms UI, 15 m × 0.25 mm, 0.25 µm film thickness (two) (p/n 19091S-431UI)
Carrier gas	Helium
Column 1 flow	1.0 mL/min
Column 2 flow	1.2 mL/min
Injection volume	2 µL cold splitless
Inlet liner	4 mm id Ultra Inert liner single taper with wool (p/n 5190-2293)
MMI temperature program	60 °C for 0.2 minutes, 600 °C/min to 300 °C, and hold
Oven temperature program	60 °C for 1 minute, 40 °C/min to 170 °C, 10 °C/min to 310 °C, Hold for 3 minutes
Run time	20.75 minutes
Backflush conditions	3 minutes post run 310 °C oven temperature 50 psi aux EPC pressure, and 2 psi inlet pressure
Transfer line temperature	280 °C
Source temperature	EI source, 280 °C
Quadrupole temperature	150 °C
Data monitoring	Dynamic MRM mode
Gain factor	5
Solvent delay	3 minutes

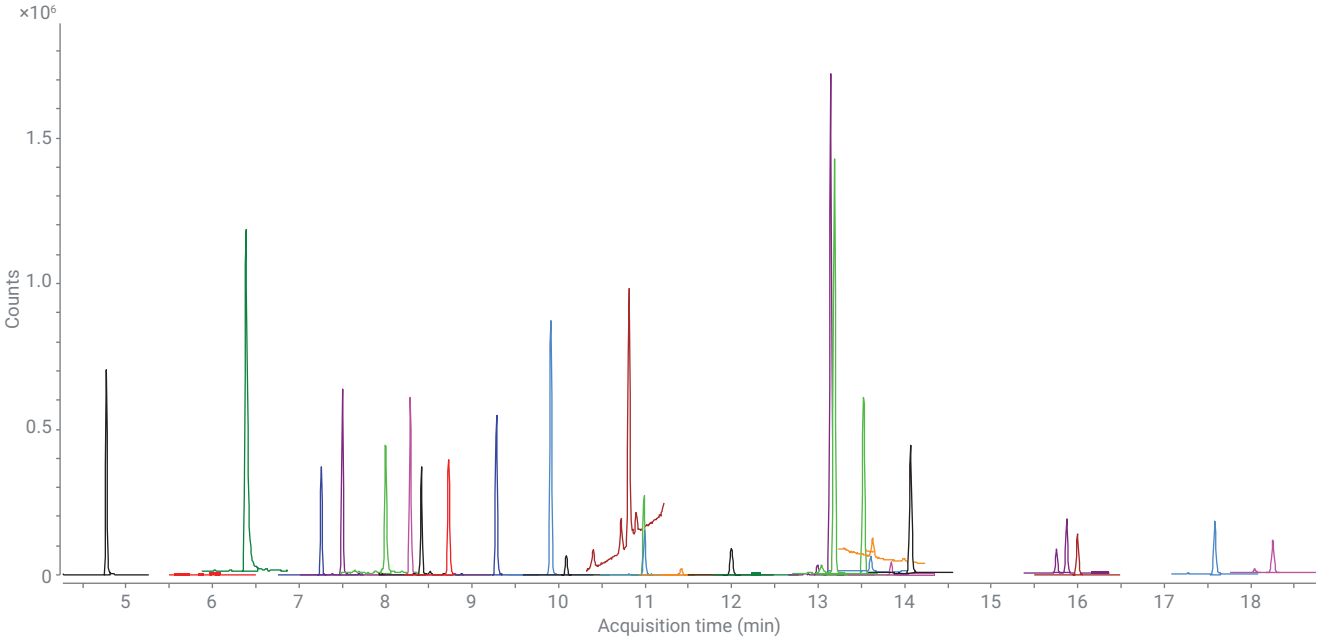

Figure 1. GC/MS/MS MRM chromatogram of pesticides in the fortified olive oil sample at the level of 100 ng/g using above GC/MS/MS conditions.

Sample preparation

The organic olive oil was purchased from local grocery stores and stored at room temperature. Olive samples were prepared using the developed sample preparation method. Figure 2 shows the step-by-step procedure, featuring three major parts: sample extraction using a two-step LLE, sample extract cleanup using Captiva EMR—Lipid pass-through cleanup, and post treatment for water removal using anhydrous $MgSO_4$ salt partition. The entire workflow introduced a five-fold dilution of the original sample concentration.

Matrix co-extractives removal evaluation

Matrix removal was investigated by a gravimetric determination of sample co-extractives residue. The amount of co-extractives residue was determined by gravimetric measurement[11] to study matrix removal after the sample extraction and cleanup procedure.

Sample extract (1 mL) was collected after sample extraction without further cleanup, and referred as the *no cleanup* sample. Sample extracts after Captiva EMR—Lipid cleanup were used as the *EMR—Lipid cleanup* sample, where an aliquot of 1.25 mL of sample extract was collected after the drying step. The additional 0.25 mL was to correct for dilution introduced through secondary elution. Samples were collected in replicates of two (n = 2), and the average weight was used to determine the matrix removal %. An experimental comparison evaluated the matrix removal with QuEChERS C18 dSPE cleanup using the same sample matrix blank.

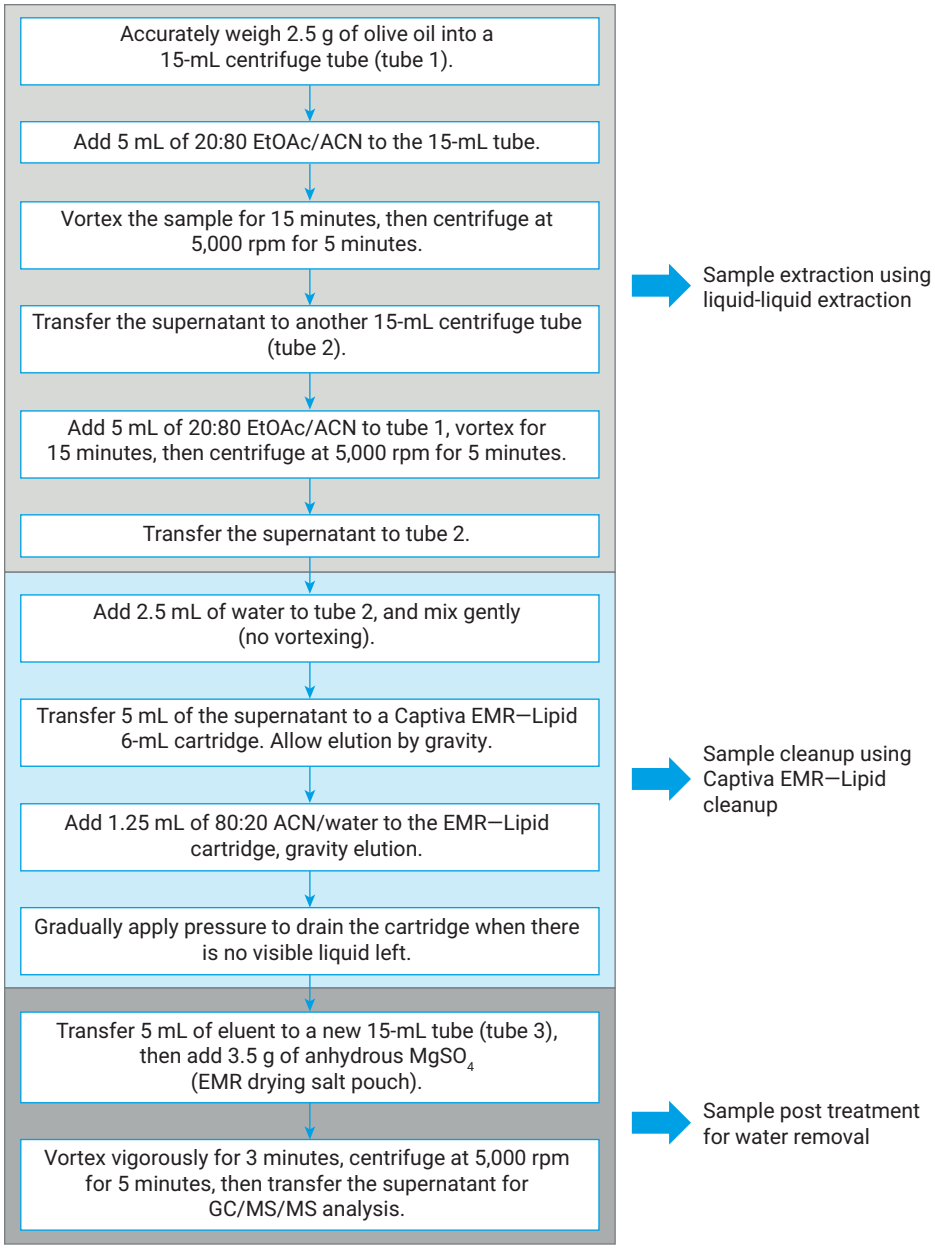

Figure 2. Olive oil preparation procedure using liquid extraction followed by EMR—Lipid cleanup.

Method validation

The optimized sample preparation method was validated in terms of selectivity, quantitation accuracy and precision, and limit of quantitation (LOQ) in olive oil. A full validation batch was run, which included matrix blanks, two sets of calibration curve standards, and prespiked QC samples. For prespiked QCs, standards and ISs were fortified into olive oil sample matrix after sample weighing before extraction. Samples were vortexed, then settled for 10 minutes to achieve equilibrium. Two sets of matrix-matched calibration standards were prepared separately by spiking standards into the matrix blanks. To ensure quantitation method consistency, matrix-matched calibration standards were run before and after the QC samples. The calibration standards included 1, 2.5, 5, 25, 50, 250, 400, and 500 ng/g in oil. Three concentrations of QC samples were quantified against calibration curves at n = 6 for low-level (1 or 5 ng/g), midlevel (10 ng/g), and high-level (100 ng/g) in oil. Analyte identification, confirmation, and quantitation were determined from retention times and MRM transitions.

Results and discussion

EMR—Lipid sorbent and product

EMR—Lipid sorbent uses a novel chemistry that combines size exclusion and hydrophobic interactions, providing high lipid removal selectivity and efficiency. Only lipid-like molecules containing unbranched hydrocarbon chains can enter the EMR—Lipid sorbent pores and be retained by hydrophobic interactions. Target analytes that do not have lipid-like structures are unable to enter the sorbent pores and remain in solution for subsequent analysis. As a result, EMR—Lipid sorbent can differentiate lipids from other target analytes, and deliver high analyte recovery and lipid removal efficiency.

Sample preparation optimization

The extraction step is challenging for achieving high recovery of nonpolar pesticides in oils due to the highly hydrophobic matrix. Among the target pesticides, over 80 % of the analytes have a log P value >3.0, which indicates high hydrophobicity.

Initially, a QuEChERS extraction was followed[11]. However, low recoveries were obtained for many hydrophobic pesticides, particularly for organochlorine pesticides such as aldrin, endrin, and DDT. The QuEChERS extraction uses acetonitrile (ACN), water, and partitioning salts. This is less effective for the extraction of pesticides from the oil, as the salt partition tends to push hydrophobic compounds back into the oil phase. Since ACN is immiscible with oil, the direct LLE was investigated, and generated better results (Figure 3A).

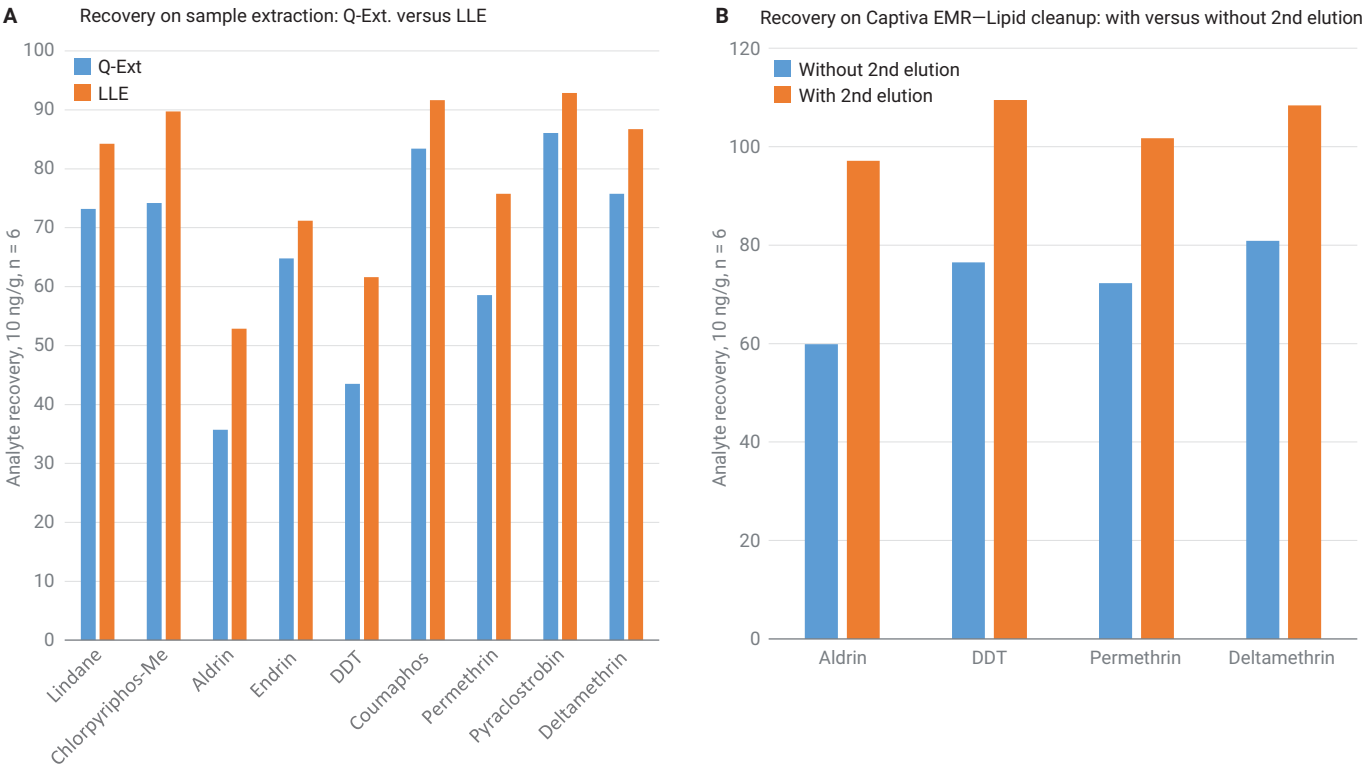

Figure 3. Sample preparation method optimization to improve analyte recoveries. A) sample extraction, QuEChERS extraction versus LLE. B) Captiva EMR—Lipid cleanup with versus without 2nd elution.

Second, the solvent extraction capability was investigated by adjusting the extraction solvent hydrophobicity in the solvent mixture. As it is important to maintain the mixture homogeneity after mixing with 20 % water, the solvent used for sample extraction has to be water miscible. Based on this requirement, a 20:80 ethyl acetate/ACN mix was found to provide higher recoveries of hydrophobic analytes, and was selected for sample extraction. Third, a two-step LLE was used instead of a single-step LLE, as it further improved the analyte extraction recoveries. The final optimized sample extraction was a two-step LLE extraction using 20:80 EtOAc/ACN. The extraction method provided >60 % recoveries for all tested pesticides.

The analyte recovery for the EMR—Lipid cartridge cleanup step was subsequently studied. Most analytes achieved high recovery during the EMR—Lipid cartridge cleanup step (>90 %) with the exceptions of aldrin, DDT, permethrin, and deltamethrin. Partial retention on the EMR—Lipid cartridge may possibly be attributed to their high lipophilicity, which results in slower movement on the cartridge due to the interaction with lipids trapped on the EMR—Lipid sorbent. As demonstrated in a previous study, a secondary elution can improve certain analyte recoveries[7]. Therefore, a secondary elution using 1.25 mL of 80:20 ACN/water was applied after the initial 5 mL of sample mixture elution. Results after this secondary elution demonstrated that recovery of the problematic pesticides was increased significantly, as shown in Figure 3B. As a result, the secondary elution was used in the final optimized protocol.

From this optimized method, the analyte recovery for the entire method was collected at a spiking level of 10 ng/g in oil in replicates of six. Each pesticide compound gave >70 % recovery, except aldrin (65 %). The average analyte recovery was 94 %, with an average RSD of 4.3 %. The results are shown in Figure 4.

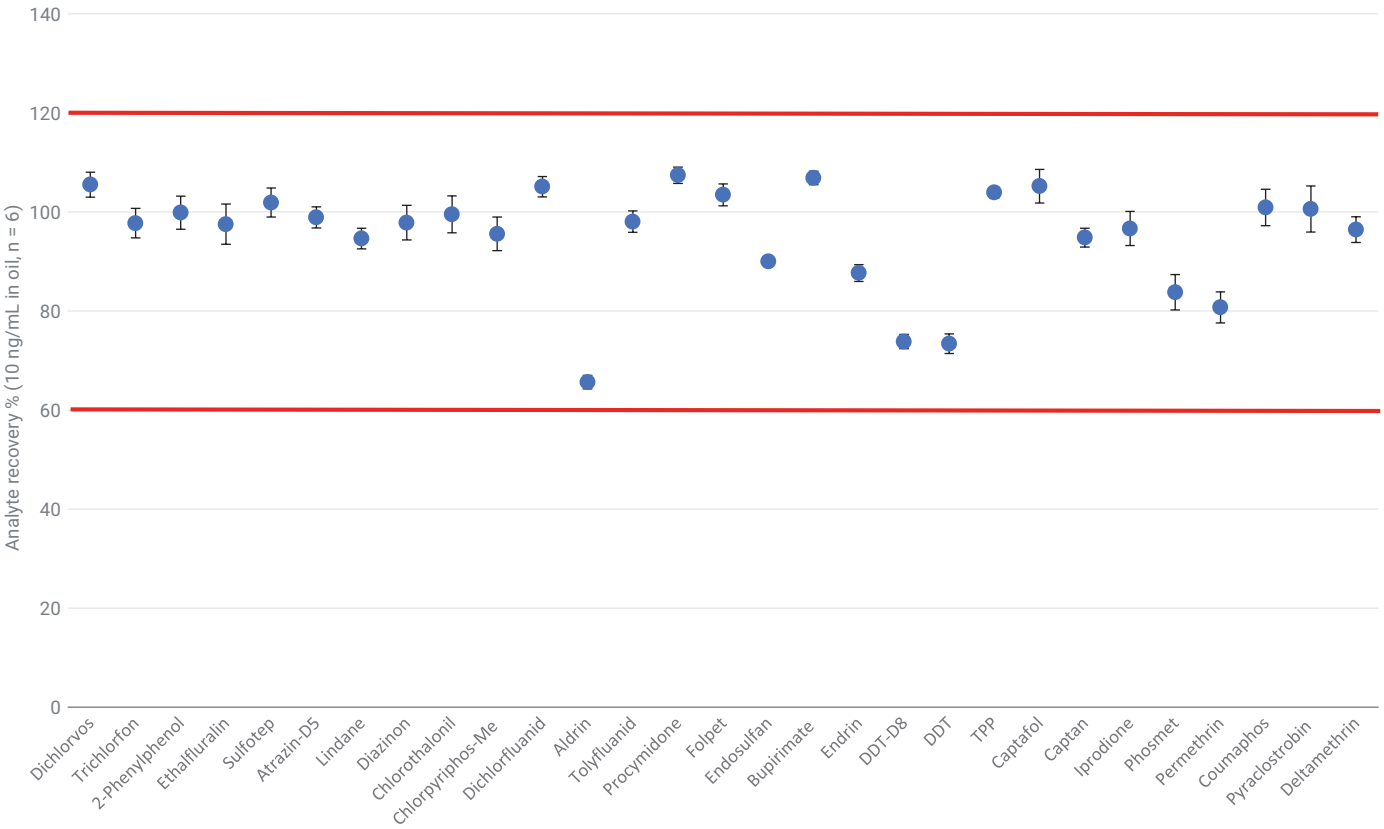

Figure 4. Pesticides recoveries using the optimized sample prep method. Olive oils samples were fortified at 10 ng/g for n = 6.

Method validation

The quantitative method validation includes limit of quantitation (LOQ), calibration curve linearity, analyte accuracy, and precision at three spiking levels. Three internal standard (IS) compounds, atrazin-D_5, DDT-D_8, and TPP, were used for analyte quantitation. Atrazin-D_5 was used for early to mideluted analytes, TPP was used for mid to later-eluted analytes, and DDT-D_8 was used for lipophilic pesticides with relatively low recoveries. Table 3 summarizes the method validation results in olive oil.

The optimized sample preparation method contributed to the improved quantitation results. The sample matrix was much cleaner, providing more consistent and reliable quantitation on the target analytes. The cleaner samples also prevented unwanted matrix accumulation in the detection system, significantly reducing the matrix impact on the detection system and flowpath.

Table 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.

Matrix cleanliness assessment

The oil matrix residue in the final extract and matrix residue removal by cleanup was investigated. In addition to EMR—Lipid cartridge cleanup, traditional C18 dSPE cleanup was tested for comparison. An olive oil sample after LLE without any cleanup generated 7.64 mg/mL of oil extract. However, a matrix sample after LLE using Captiva EMR—Lipid cleanup generated only 1.14 mg/mL of oil extract. A matrix sample after LLE using traditional PSA/C18 dSPE cleanup generated 3.47 mg/mL of oil extract. Based on the residue weight being collected, Captiva EMR—Lipid cleanup provides 85 % of matrix co-extractives removal. This is much higher than the 55 % of matrix co-extractives removal provided by traditional PSA/C18 dSPE cleanup.

Conclusion

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 method in olive oil was optimized to improve the extraction efficiency for nonpolar pesticides from hydrophobic oil matrices and complete elution on a Captiva EMR—Lipid cartridge. Captiva EMR—Lipid cartridge cleanup provided efficient lipid removal from oil extracts, without unwanted analyte loss. The quantitative analysis showed that >96 % of tested pesticides provided >70 % average recoveries, and 100 % of analytes gave excellent reproducibility, with <15 % average RSD. Results demonstrate that the optimized method provides high matrix cleanup, excellent analyte recovery, and precision results for multiclass, multiresidue analysis of pesticides in edible oil.

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