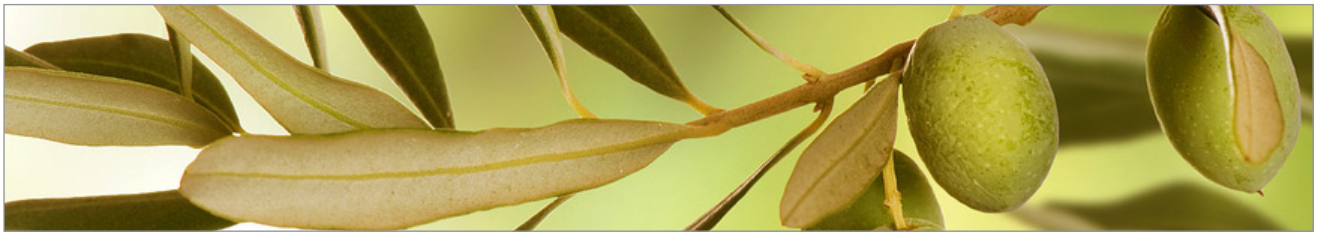

Technical Article

Prepare Olive Oil Samples for Pesticide Residue Analysis in Half the Time With a Fraction of the Solvent Using dSPE

By Michelle Misselwitz[1], Julie Kowalski[1], Mark Crawford[2], Michael Halvorson[2], and Joan M. Stevens[2]
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*Simplify and speed up sample preparation with **Resprep® dSPE tubes**! Here we show the extraction and cleanup of pesticide residues from olive oil samples—twice as fast as GPC, with only a fraction of the solvent required for conventional SPE.*

Increase sample throughput with a quick, easy sample preparation method, while protecting your column from matrix contamination.

Olive oil is considered a healthy fat source and is a staple in many recommended diets. However, concerns about potentially negative health effects associated with pesticide residues have increased consumer interest in testing. While organophosphorus pesticides are currently used in olive orchards to control pests, organochlorine pesticides are still tested, even though they are no longer in commercial use, because they are persistent organic pollutants. There are several existing methods for measuring pesticide residues in olive oil, all of which involve sample extraction and cleanup.[1] The common goal of these methods is to remove lipids that are harmful to the analytical system.[2] Efficient sample cleanup procedures are critical to maximizing sample throughput and minimizing labor and material costs. Here we demonstrate the efficiency of a dSPE cleanup procedure, as well as the capabilities of a method-specific analytical column.

Simple Procedure Uses Half the Time and Minimal Solvent

Sample extraction and cleanup can be accomplished with gel permeation chromatography (GPC), solid phase extraction (SPE), or dispersive solid phase extraction (dSPE) methods. However the dSPE method shown here is much less expensive than GPC (which requires specialized equipment) and uses substantially less solvent than comparable GPC or SPE methods (Table I).[3] The method is simple to use and allows sample extraction and cleanup to be accomplished in half the time of other techniques (Table II).

Extraction and dSPE Cleanup for Pesticide Residues in Olive Oil

Test sample: A 1.5 mL sample of commercially obtained virgin olive oil was spiked with a standard organochlorine pesticide mix. The spiked sample was processed as follows:

1. Dilute with 1.5 mL hexane.
2. Add 6 mL of acetonitrile (ACN).
3. Mix for 30 minutes on a shaker
4. Allow layers to separate (approximately 20 minutes), then collect the top (ACN) layer.
5. Repeat the liquid-liquid extraction (steps 2–4) and combine both ACN extract layers.
6. Place 1 mL of the combined ACN extract in a 1.5 mL tube containing 150 mg magnesium sulfate and 50 mg PSA.
7. Shake the tube for 2 minutes.
8. Centrifuge at 3,000 U/min for approximately 5 minutes.
9. Remove the top layer and inject directly into the gas chromatograph system.

Extracts were analyzed using the Rtx®-CLPesticides2 column (Figure 1). The Rtx®-CLPesticides2 column is a method-specific column that resolves all chlorinated pesticide residues tested. Recoveries of 70%–80% were obtained, levels comparable to conventional SPE—without the necessity of vacuum manifolds or high-pressure systems. The GPC method attained recoveries of > 95%. However this method requires large amounts of solvent and takes over twice as long as other methods.

The dSPE method shown here is an efficient, cost-effective way to clean up chlorinated pesticide residues in olive oil. With good recoveries and minimal matrix interference, it is an easy way to reduce solvent usage, compared to conventional SPE, and is more cost-effective than GPC.

Table I: The Resprep® dSPE method uses 42% and 89% less solvent than SPE and GPC methods respectively.

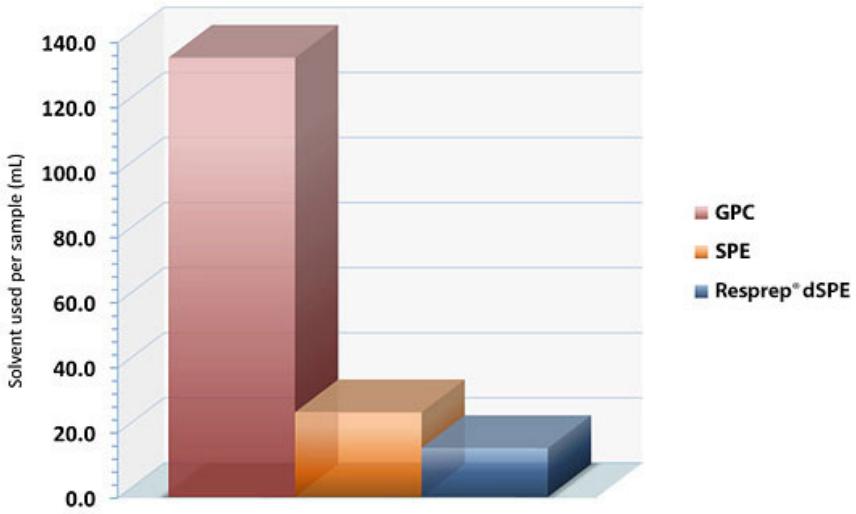


Table II: Cut extraction/cleanup time by 50% using a Resprep® dSPE method.

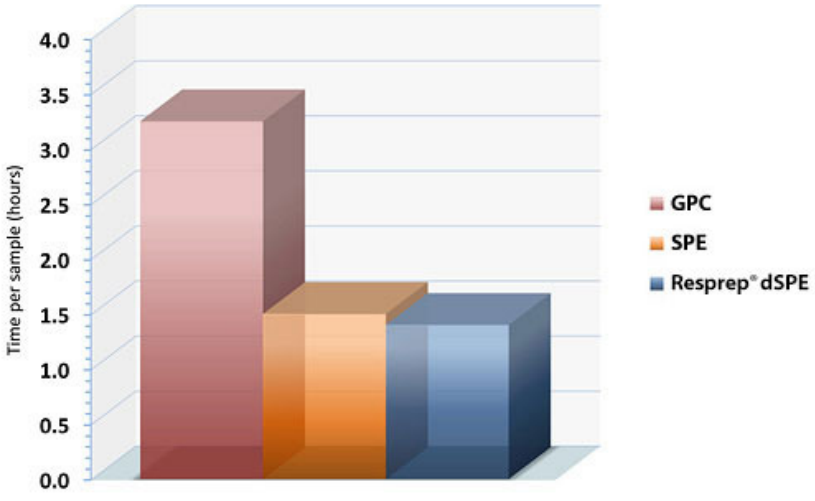
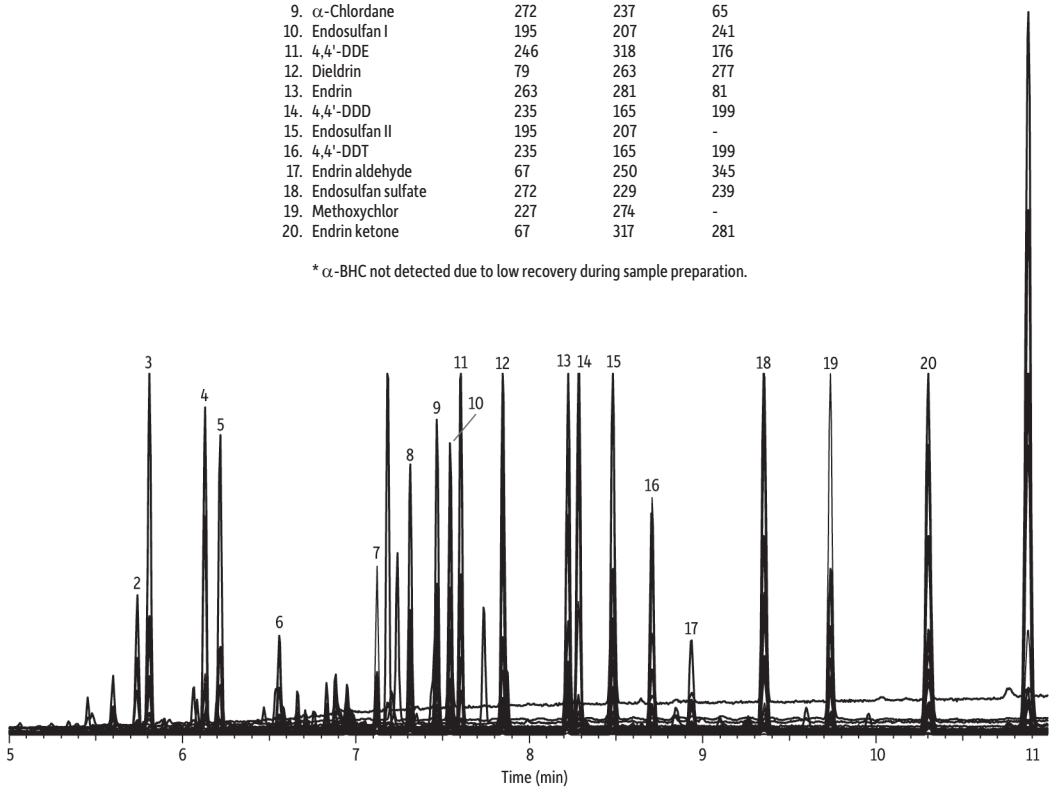


Figure 1: Chlorinated pesticide residues in olive oil are easily separated on the Rtx®-CLPesticides2 column.

	Peaks	Quant. ion	Qual. ion 1	Qual. ion 2
1.	α-BHC*	219	181	109
2.	γ-BHC	219	181	109
3.	β-BHC	219	181	109
4.	δ-BHC	219	181	109
5.	Heptachlor	272	237	100
6.	Aldrin	263	293	220
7.	Heptachlor epoxide	263	237	81
8.	δ-Chlordane	272	237	65
9.	α-Chlordane	272	237	65
10.	Endosulfan I	195	207	241
11.	4,4'-DDE	246	318	176
12.	Dieldrin	79	263	277
13.	Endrin	263	281	81
14.	4,4'-DDD	235	165	199
15.	Endosulfan II	195	207	-
16.	4,4'-DDT	235	165	199
17.	Endrin aldehyde	67	250	345
18.	Endosulfan sulfate	272	229	239
19.	Methoxychlor	227	274	-
20.	Endrin ketone	67	317	281

* α-BHC not detected due to low recovery during sample preparation.



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Column	Rtx®-CLPesticides2 30 m, 0.25 mm ID, 0.20 µm (cat.# 11323)
Sample	Olive oil spiked with Organochlorine Pesticide Mix AB # 3 (cat.# 32415)
Conc.:	10 µg/mL
Injection	
Inj. Vol.:	1 µL splitless (hold 0.5 min)
Liner:	Single Taper w/Wool (cat.# 22286-200.1)
Inj. Temp.:	225 °C
Oven	
Oven Temp.:	140 °C (hold 0.5 min) to 268 °C at 20 °C/min to 290 °C at 3 °C/min to 330 °C at 20 °C/min (hold 5 min)
Carrier Gas	He, constant flow
Flow Rate:	1 mL/min
Detector	MS
Mode:	SIM
Transfer Line Temp.:	320 °C
Ionization Mode:	EI
Notes	Extraction and dSPE Cleanup for Pesticide Residues in Olive Oil

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References

[1] C. Lentza-Rizos, E.J. Avramides, Rev. Environ. Contam. Toxicol. 141 (1995) 111.
[2] S. Cunha, S. Lehotay, K. Mastovska, J. Sep. Sci. 30 (2007) 620.
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