



Optimization of Sample Preparation for Pesticide Analysis in Oil-Based Cannabis Products using LipiFiltr®

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UCT Part Number

LPFLTR01

LipiFiltr® push-through cartridge



Summary:

Cannabis testing laboratories face the difficult task of removing a variety of unwanted matrix components from their samples prior to LC-MS or GC-MS analysis. High-lipid samples, in particular, pose a major challenge when testing for a large number of pesticide residues that contain different physicochemical properties and need to be detected at very low concentrations. Oil-based cannabis products often use a variety of food grade oils such as medium chain triglyceride oil, coconut oil, olive oil and hemp oil, and the complexity of these cannabis products presents additional analytical challenges that do not need to be accounted for in other laboratory settings. Typical sample preparation procedures, such as QuEChERS, are easy to carry out and well-suited for the extraction of a wide range of pesticide residues. However, they also extract a large amount of matrix components, including lipids and other hydrophobic compounds, which can lead to poor analyte recovery and reproducibility, chromatographic interference, ion suppression/enhancement, and additional instrument maintenance. Current sample cleanup options may not be suitable for effectively and selectively removing these unwanted interferences. Large amounts of lipids remaining in the sample may then necessitate the use of additional cleanup steps or the use of a product that will indiscriminately retain lipids and analytes of interest. To overcome these challenges UCT recently introduced a new product for the cleanup of fatty samples – LipiFiltr®. The ability to obtain significantly cleaner extracts, ease of use, and time and cost savings make the new LipiFiltr® push-through cartridges an attractive cleanup option for laboratories conducting pesticide residue analysis in complex fatty samples.

This application note outlines the performance benefits achieved using the LipiFiltr® cleanup cartridge for the analysis of pesticides in oil-based cannabis products using LC-MS/MS analysis. The pesticides evaluated in this study include those listed in the Oregon monitoring list (~60 pesticides). CBD oil was used as the representative matrix. A comparison of pre- and post-LipiFiltr® cleanup using full scan GC-MS is also presented to demonstrate the removal of lipid coextractives. This work was originally presented as a poster at the North American Chemical Residue Workshop (NACRW) 2018 in Naples, Florida.



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Procedure:

1. Weigh 1 g of sample into a 50 mL polypropylene centrifuge tube.
2. Add internal standard(s).
3. Add 10 mL of extraction solvent (acetonitrile/water/toluene, 90/5/5, v/v).
4. Shake or vortex the sample for 1 hour.
5. Centrifuge the sample at ≥ 1500 rcf for 5 minutes.
6. Pass 1.5 mL supernatant through a LipiFiltr[®] cartridge and collect the purified extract in an autosampler vial.

Results:

Co-extractives Removal

A comparison of pre and post LipiFiltr[®] cleanup was evaluated using a generic GC-MS full scan acquisition. All samples were run on a Rxi-5Sil MS column (30m x 0.25mm x 0.25 μ m) with a Topaz Precision split inlet liner. A 1 μ L injection volume with a split ratio of 10:1 was used.

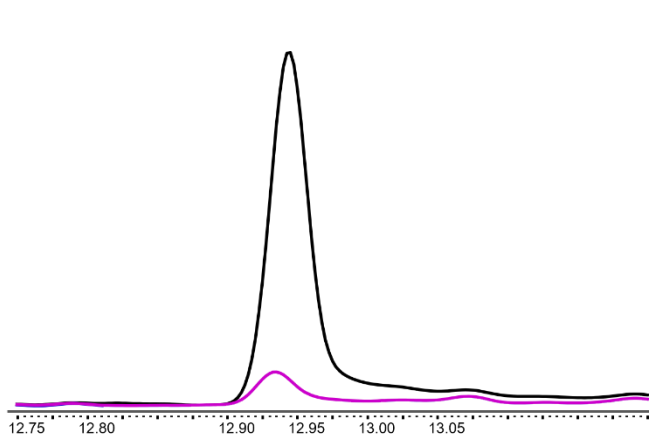


Figure 1. Comparison of hexadecanoic acid content pre and post LipiFiltr[®] cleanup

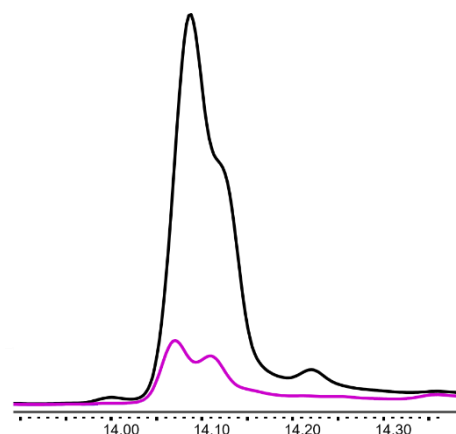


Figure 2. Comparison of octadecadienoic & octadecatrienoic acids pre and post LipiFiltr[®] cleanup

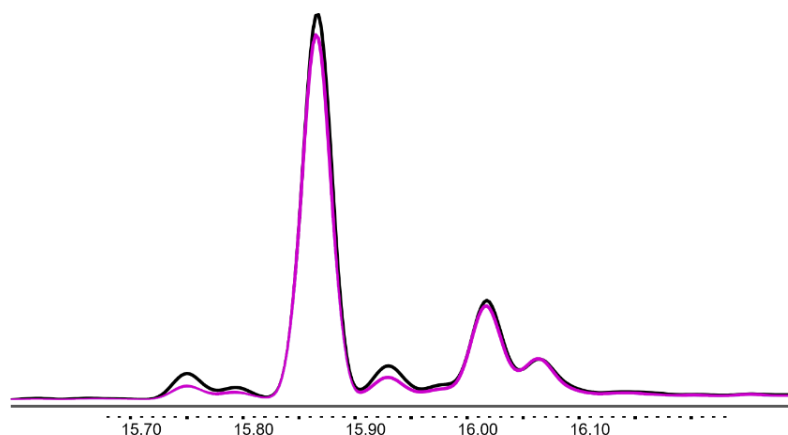


Figure 3. Comparison of cannabidiol content pre and post LipiFiltr[®] cleanup

■ CBD oil extract (before LipiFiltr[®])
■ CBD oil extract (after LipiFiltr[®])



Pesticide Recovery

A single matrix-matched calibration point (200 ppb) was used to calculate the recoveries (n=5). Linuron-d6 was utilized as the internal standard. The mass spectrometer used for analysis was a SCIEX Triple Quad 6500+ using the SCIEX vMethod™ for Pesticides in Cannabis. The ion ratio acceptance criteria was set at $\pm 30\%$ (relative).

Compound	Average Recovery (%)	RSD (n=5, %)
Acephate*	48	3
Acequinocyl**	46	14
Acetamiprid	84	6
Aldicarb	79	6
Avermectin B1a	107	18
Azoxystrobin	93	5
Bifenazate	90	5
Bifenthrin	75	2
Boscalid	84	2
Carbaryl	87	6
Carbofuran	88	5
Chlorantraniliprole	87	8
Chlorfenapyr	89	6
Chlorpyrifos	77	5
Clofentezine	80	6
Cyfluthrin*	ND	NA
Cypermethrin	94	17
Daminozide***	ND	NA
Diazinon	84	6
Dichlorvos	86	5
Dimethoate	86	4
Ethoprophos	86	7
Etofenprox*	58	6
Etoxazole	81	6
Fenoxycarb	88	5
Fenpyroximate	83	5
Fipronil	96	8
Flonicamid	83	6
Fludioxonil	96	9
Hexythiazox	76	5
Imazalil	81	4
Imidacloprid	86	5
Kresoxim-methyl	96	11
Malathion A	89	6
Metalaxyl	84	10
Methiocarb	87	4
Methomyl	84	6
MGK 264	81	11
MGK 264 Isomer	79	8
Myclobutanil	89	5
Naled	86	5
Oxamyl	82	4
Paclobutrazol	86	6
Parathion_methyl	101	23
Permethrin, cis*	72	12
Permethrin, trans*	ND	NA
Phosmet	88	3



Piperonyl butoxide	84	3
Prallethrin	86	9
Propiconazole	84	5
Propoxure	88	5
Cinerin I	85	11
Cinerin II	92	32
Jasmolin I	77	7
Jasmolin II	91	17
Pyrethrin I	81	8
Pyrethrin II	100	38
Pyridaben	73	7
Spinosad A	81	7
Spinosyn D	82	5
Spiromesifen	79	14
Spirotetramat	85	6
Spiroxamine	81	4
Tebuconazole	81	6
Thiacloprid	85	6
Thiamethoxam	80	6
Trifloxystrobin	87	7

* Selected pesticides are more amendable to GC-MS analysis.

** Acequinocyl is unstable and degrades to acequinocyl-hydroxy.

*** Daminozide is a highly polar molecule that is difficult to incorporate in a multi-residue method (sample extraction & HPLC separation).

ND = not detected

Discussion:

Several combinations of solvents were evaluated to find the best extraction solution for the wide range of pesticides included in the method, including acetonitrile/methanol (90/10, v/v), acetonitrile/IPA (80/20, v/v) and acetonitrile/water/toluene (90/5/5, v/v). It was found that the recovery of the hydrophobic pesticides improved with the addition of a small amount of toluene. Without the inclusion of toluene some of the hydrophobic pesticides remained in the lipid layer and did not partition efficiently into the more polar extraction solvent. Most compounds were found to have a recovery value in the range of 70-120%. A few polar compounds, such as acephate, exhibited low but reproducible (<10% RSD) recovery. Increasing the polarity of the extraction solvent may improve the recovery of these pesticides. Alternatively, the use of a suitable isotopically labelled internal standard can also be used to correct for any discrepancy in recovery.

GC-MS (full scan) analysis showed significant removal of lipids and other high molecular weight matrix co-extractives using the LipiFiltr® cartridge, with no significant negative effect on CBD. This was backed up by LC-DAD analysis (not detailed here). While further work should be completed on other oil-based products, including those with a high THC content, the results highlighted here demonstrate that the LipiFiltr® cartridge can be successfully used to remove matrix co-extractives from oil-based cannabis samples without compromising CBD content or pesticide recoveries.

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