

Using Pressurized Liquid Extraction (PLE) for the Extraction and Cleanup of Cannabis samples for Pesticide Analysis by LC/MS.

Introduction

The use of LC/MS for pesticide analysis in agricultural matrices has long been an ideal option. Their affinity for acetonitrile makes them an ideal pairing for LC runs, and has enabled a wide array of screening methods to be employed (e.g. QuEChERS). Often requiring manual interactions, solvent transfers, centrifuging and manual SPE clean-ups, the process can be time consuming and requires constant attention of laboratory staff.

Pressurized Fluid Extraction (PLE) can offer a completely automated extraction process while integrating the clean-up portion of the procedure into a single one step operation. The ability to pair up to 8 modular extraction units results in an automated extraction process that delivers multiple analysis ready extractions in fewer than 20 minutes, while operating unattended.

Instrumentation

- FMS, Inc. PLE® extraction system
- Agilent LC/MS

Consumables

- FMS 10 ml PLE extraction cells
- FMS Teflon PLE end caps
- Acetonitrile, LC/MS grade or equivalent
- Florisil® Bulk sorbent
- Graphitized Carbon Bulk sorbent
- Magnesium Sulfate
- Ottawa Sand

Sample/Reagent Prep

1. Sample aliquots are to be weighed out, thoroughly mixed.
2. Sample aliquots are mixed with MgSO₄ and loaded into extraction cells and loaded onto the PLE system.
3. Clean-up sorbents are measured and layered into the PLE extraction cells (See figure 1).
4. Cells are sealed and loaded onto PLE extraction system.

PLE Procedure Pesticides

1. Cells are filled sequentially with Acetonitrile.
2. Cells are pressurized and held at a constant temperature for 5 minutes.
3. Cells are cooled and depressurized
4. Cells are flushed with Acetonitrile nitrogen purged of remaining solvent.
5. Final extract is collected and a sample aliquot is transferred to a vial for GC/MS-MS analysis.

Table 1. PLE Extracts of flower samples with positively spiked pesticides (ppm, n=9)

Analyte	Matrix	
	Mean	RSD
Bifenazate	6.349	17
Bifenthrin	2.031	17
Cyfluthrin	1.632	27
Cypermethrin	3.483	16
Dimethomorph	7.369	17
Etoazole	10.234	13
Fenhexamid	3.535	18
Fonicamid	3.458	51
Fludioxonil	2.919	41
Imidacloprid	3.452	36
Myclobutanil	4.031	18
Piperonyl Butoxide	9.844	7
Thiamethoxam	4.089	33
Trifloxystrobin	3.449	14

Table 2. MDL study results.

Pesticide Spike	MDL	Pesticide Spike	MDL
bifenazate	0.033	piperonyl butoxide	0.024
bifenthrin	0.035	pyrethrin I	0.017
cyfluthrin	0.067	pyrethrin II	0.061
cypermethrin	0.029	spinetoram-748	0.041
dimethomorph	0.035	spinetoram-760	0.016
etoxazole	0.033	spinosyn A	0.016
fenhexamid	0.037	spinosyn D	0.017
flonicamid	0.034	spirotetramat	0.031
fludioxonil	0.029	thiamethoxam	0.031
paclobutrazol	0.033	trifloxystrobin	0.040
imidacloprid	0.029		

Results

Table 3. LCS recoveries of LC pesticides using the PLE (N=7)

Analyte	Sand Spike	
	Mean	RSD
Acequinocyl	137%	23
Bifenazate	79%	14
Bifenthrin	90%	12
Cyfluthrin	62%	34
Cypermethrin	66%	38
Dimethomorph	88%	9
Etoxazole	87%	9
Fenhexamid	87%	12
Fonicamid	104%	19
Fludioxonil	88%	9
Imidacloprid	90%	9
Myclobutanil	88%	11
Piperonyl Butoxide	89%	5
Spirotetramat	94%	6
Thiamethoxam	90%	10
Trifloxystrobin	84%	9

Conclusions

Results of LC/MS-MS data reveal the PLE can easily extract multiresidue pesticides while simultaneously performing a highly efficient in-cell clean-up. Recoveries of observed pesticides were efficiently recovered in both clean and live matrices. Additionally, results of a performed MDL study provided acceptable reporting levels.

The ability of the PLE to generate an analysis ready extract proves to be a time saver, conservatively enabling a 3X-4X decrease in sample volume as compared to traditional manual procedures. The increased efficiency and ease of operation make the PLE a true one stop solution for pesticide residue analysis of Cannabis samples.

Figure 3. FMS Inc. Pressurized Extraction System



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