

An MDL Study using Pressurized Liquid Extraction (PLE®) for the Extraction and Cleanup of Cannabis Samples for the Analysis of Pesticides

Introduction

At present in the United States, there are 8 states with legalized recreational usage and 29 states with legal medical marijuana use. As this trend continues, consumer safety is a major topic of concern. This is especially true for the screening for pesticides. Like most agricultural products, pesticides are widely used for crop management and can find their way into consumer goods. It is therefore important that reliable, rapid and cost-effective procedures be in place for the screening of products destined for a consumer market.

Pesticide extractions and analysis have long been in place for the food and environmental industries. Tapping into these methodologies, the usage of pressurized extraction can be implemented to deliver a one-step extraction and clean-up process for rapid LC/MS, GC/MS-MS analysis of a wide array of pesticides. In this note we focus on the results of an MDL study.

Instrumentation

- FMS, Inc. PLE® extraction system
- Thermo TSQ Quantum XLS Triple Quad GC/MS

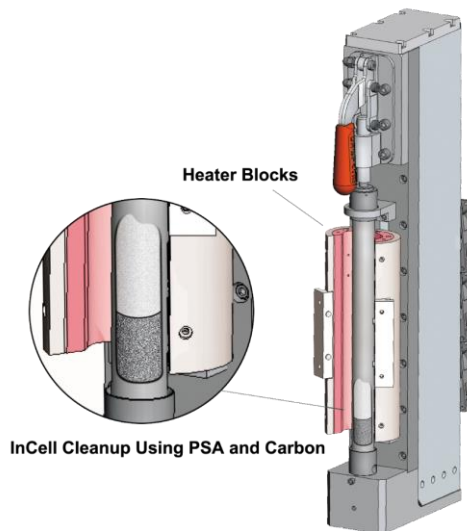
Consumables

- FMS 20 mL PLE extraction cells
- FMS Teflon PLE end caps
- Acetonitrile, LC/MS grade or equivalent
- FMS CleanXtract™
- Ottawa Sand
- Tributylphosphate and triphenylphosphate internal standards (Restek)
- Various pesticides solutions (Restek)

Sample/Reagent Prep

1. Sample aliquots are to be weighed out, thoroughly mixed.
2. Extraction cells are capped at the bottom with a Teflon end cap and filled with a layer of Ottawa Sand, layer of FMS CleanXtract, layer of cannabis, spiked standards, and layer of Ottawa Sand and capped
3. Cells are turned upside down and put onto the PLE system (See Figure 1).

Figure 1. PLE extraction cell with sample and clean-up



PLE Procedure Pesticides

1. Cells are filled with Acetonitrile.
2. Cells are pressurized to 1000 PSI at ambient temperature for 10 minutes.
3. Cells are depressurized
4. Cells are purged with nitrogen to remove remaining solvent.
5. Final extract is collected, and a 1ml sample aliquot is transferred to a vial for LC/MS and GC/MS analysis.

Data in ppb.												
Analyte	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7		AVERAGE	STDEV	MDL	Recoveries
Acequinocyl	11.74	11.84	11.90	11.81	11.46	11.39	12.02		11.74	0.23	0.72	117%
Bifenthrin	8.74	7.14	8.90	7.77	8.45	8.35	8.20		8.22	0.60	1.89	82%
Captan	10.32	8.91	10.76	8.07	9.31	7.72	6.75		8.88	1.43	4.50	88%
Chlorfenapyr	11.10	10.97	14.15	14.61	9.96	11.18	7.67		11.38	2.39	7.49	114%
Chlorpyrifos	5.66	6.28	8.99	6.30	6.30	10.52	11.29		7.91	2.32	7.29	79%
cis-Chlordane	8.11	8.73	8.70	5.63	7.27	7.94	7.23		7.66	1.08	3.38	77%
trans-Chlordane	10.62	6.75	5.47	9.41	9.68	6.12	4.68		7.53	2.33	7.33	75%
Coumaphos	12.23	14.69	12.09	12.21	11.20	13.20	10.62		12.32	1.33	4.18	123%
Cyfluthrin I	9.85	9.38	9.07	12.81	8.11	11.18	11.44		10.26	1.62	5.08	103%
Cyfluthrin II	9.89	7.00	5.37	8.40	11.01	8.55	9.36		8.51	1.87	5.88	85%
Cyfluthrin III	10.07	7.00	8.14	7.28	8.64	7.66	6.87		7.95	1.13	3.54	80%
Cyfluthrin IV	6.96	7.98	6.56	8.45	7.60	8.75	6.54		7.55	0.89	2.80	75%
Cyfluthrin*	9.19	7.84	7.29	9.24	8.84	9.04	8.55		8.57	0.74	2.33	86%
Cypermethrin II	9.24	9.64	6.08	6.30	7.97	7.78	6.67		7.67	1.41	4.41	77%
Cypermethrin III	10.04	5.39	7.57	7.10	6.77	7.86	6.10		7.26	1.49	4.67	73%
Cypermethrin IV	6.77	9.41	4.79	7.70	5.28	9.22	5.91		7.01	1.84	5.78	70%
Cypermethrin I	7.82	5.71	7.88	12.41	9.98	8.43	10.06		8.90	2.14	6.71	89%
Cypermethrin*	8.47	7.54	6.58	8.38	7.50	8.32	7.19		7.71	0.71	2.23	77%
Diazinon	9.96	8.30	8.50	6.47	7.23	10.67	11.30		8.92	1.79	5.62	89%
Etofenprox	7.84	8.28	8.08	6.10	7.91	7.71	7.68		7.66	0.72	2.25	77%
Fipronil	8.88	7.09	8.52	8.34	8.97	7.37	6.79		7.99	0.89	2.81	80%
Fludoxonil	6.66	12.15	6.28	6.32	6.98	8.31	5.59		7.47	2.23	7.00	75%
Malathion	10.06	4.86	9.03	9.23	7.11	7.87	8.43		8.08	1.71	5.38	81%
Metalaxyl	5.20	9.70	6.75	10.35	10.56	10.55	9.68		8.97	2.13	6.67	90%
Methyl parathion	7.20	9.41	9.13	9.99	8.43	11.74	8.91		9.26	1.40	4.40	93%
Mevinphos	13.85	10.22	11.40	9.85	10.77	12.81	11.82		11.53	1.43	4.48	115%
Myclobutanil	7.88	6.00	6.66	6.37	7.72	7.23	8.07		7.10	0.78	2.44	71%
Padobutrazol	9.76	9.27	5.52	7.92	5.97	5.26	5.82		7.07	1.88	5.91	71%
cis-Permethrin	10.49	9.32	10.59	9.87	9.60	10.34	10.23		10.06	0.48	1.50	101%
trans-Permethrin	8.88	8.76	9.26	7.64	6.67	7.64	8.70		8.19	0.90	2.83	82%
Phosmet	13.70	5.67	6.67	11.72	8.14	8.78	9.05		9.10	2.79	8.76	91%
Piperonyl butoxide	10.73	10.03	10.54	10.64	14.00	8.72	13.30		11.14	1.86	5.83	111%
Pyridaben	10.58	7.55	12.51	9.75	5.30	8.26	7.06		8.72	2.41	7.57	87%
Tebuconazole	10.80	7.48	7.26	4.10	7.05	10.63	7.96		7.90	2.30	7.21	79%

Table 1. Method Detection Level data (runs # 1 - 7 sand/FMS CleanXtract/ 10 ppb of each pesticide/ internal standards).

Data in ppb.	Cannabis A	Cannabis B	Sand
Analyte			
Acequinocyl	ND	ND	ND
Bifenthrin	6.18	3.56	ND
Captan	2.35	2.84	ND
Chlorfenapyr	ND	0.66	ND
Chlorpyrifos	0.60	0.67	ND
cis-Chlordane	ND	ND	ND
trans-Chlordane	ND	ND	ND
Coumaphos	ND	ND	ND
Cyfluthrin I	3.84	2.71	ND
Cyfluthrin II	0.32	0.11	ND
Cyfluthrin III	0.48	0.21	ND
Cyfluthrin IV	0.65	0.52	ND
Cyfluthrin*	1.63	1.01	ND
Cypermethrin II	0.21	0.07	ND
Cypermethrin III	0.09	0.06	ND
Cypermethrin IV	0.58	0.41	ND
Cypermethrin I	10.34	8.64	ND
Cypermethrin*	2.80	2.30	ND
Diazinon	ND	ND	ND
Etofenprox	0.51	0.36	ND
Fipronil	ND	4.80	ND
Fludioxonil	0.34	0.30	ND
Malathion	8.56	6.54	ND
Metalaxyl	10.81	3.03	ND
Methyl parathion	1.31	0.54	ND
Mevinphos	0.98	1.38	ND
Myclobutanil	0.08	0.30	ND
Pacllobutrazol	17.91	8.73	ND
cis-Permethrin	ND	ND	ND
trans-Permethrin	0.10	0.10	ND
Phosmet	1.71	0.72	ND
Piperonyl butoxide	12.02	12.82	ND

Table 2. Analysis of 1 g cannabis A and B and method blank (sand). Concentrations reflect residual pesticides in material analyzed.

Figure 2. FMS Inc PLE System.



Conclusions

The PLE with Incell clean-up proved to generate acceptable MDL data (Table 1) for the tested analytes of interest with both clean backgrounds and reproducibility between replicates. The end result was a calculated MDL that met expected detection requirement.

Additional runs of cannabis (Table 2, no additional pesticides added, just internals) proved the Incell clean-up produced extracts that did not require any additional manual purification steps while demonstrating consistent extractions when positive detection was present. The entire sample extraction and cleanup process for 8 samples simultaneously takes 12 minutes delivering a sample ready for analysis. The final extract is used for both LC/MS and GC/MS analysis of pesticides.

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