Application Note

Using Pressurized Liquid Extraction (PLE) for the Extraction and Analysis of Pesticides in Cannabis samples by GC/MS-MS.

Introduction

At present within the United States, there are 8 states with legalized recreational usage and 21 states with medical marijuana legalized. As this spreading trend continues, consumer safety is a major topic of concern. One area of considerable concern is 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 fitted to deliver a one-step extraction and extract clean-up process for rapid GC/MS-MS analysis of a wide array of pesticides.

Instrumentation

• FMS, Inc. PLE® extraction system
•Thermo Trace GC w/PTV injector port
•Thermo TSQ Quantum Ultra Mass Spectrometer

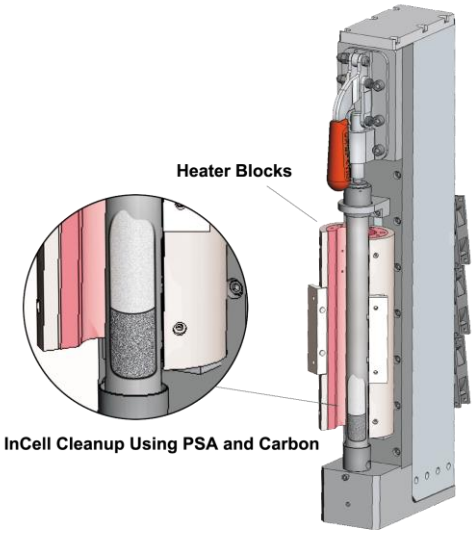
Consumables

• FMS 10 ml PLE extraction cells
• FMS Teflon PLE end caps
• Acetonitrile, LC/MS grade or equivalent
• CleanXtract™ Cleanup Material
• Ottawa Sand

Sample/Reagent Prep

1. Sample aliquots are to be weighed out, thoroughly mixed.
2. Sample aliquots are mixed with 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.

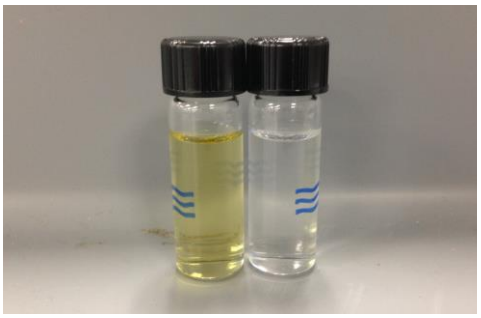
Figure #1. PLE extraction cell with sample and clean-up



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.

Figure 2. PLE Extracts of flower samples comparing in-cell clean-up with no clean-up



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Results

Table 1. Subset of 203 pesticide list spiked into flower samples (N=5) and analyzed by GC/MS-MS

Analyte	Class	Mean	RSD
Diazinon	Organophosphorus	92%	7%
Chlorpyrifos methyl	Organophosphorus	76%	19%
Fenitrothion	Organophosphorus	109%	10%
Pirimiphos methyl	Organophosphorus	76%	6%
Chlorpyrifos	Organophosphorus	80%	13%
Pirimiphos ethyl	Organophosphorus	75%	8%
Quinalphos	Organophosphorus	74%	21%
Phosalone	Organophosphorus	94%	27%
Chlorneb	Organochlorine	90%	22%
HCH-alpha	Organochlorine	74%	16%
Pentachloroanisole	Organochlorine	70%	26%
HCH-delta	Organochlorine	86%	17%
Heptachlor	Organochlorine	90%	9%
Heptachlor epoxide (isomer B)	Organochlorine	84%	11%
Chlorfenson (Ovex)	Organochlorine	92%	10%
Endosulfan II	Organochlorine	102%	18%
Tetrachloronitrobenzene (Tecnazene)	Organonitrogen	92%	17%
THPI (Tetrahydrophthalimide)	Organonitrogen	96%	9%
Diphenylamine	Organonitrogen	88%	19%
2,3,5,6-Tetrachloroaniline	Organonitrogen	76%	30%
Pentachlorobenzene (Quintozene)	Organonitrogen	108%	29%
Pentachlorobenzonitrile	Organonitrogen	98%	29%
Prodiamine	Organonitrogen	111%	30%
Isopropalin	Organonitrogen	98%	15%
Pendamethalin	Organonitrogen	63%	28%
Oxyfluorfen	Organonitrogen	90%	26%
Nitralin	Organonitrogen	87%	27%
Pebulate	Organonitrogen	84%	17%
N-(2,4-Dimethylphenyl)formamide	Organonitrogen	86%	7%
cis-Diallate	Organonitrogen	85%	18%
trans-Diallate	Organonitrogen	111%	14%
Clomazone (Command)	Organonitrogen	78%	8%
Propyzamide	Organonitrogen	79%	14%
Dimethachlor	Organonitrogen	108%	6%
Propanil	Organonitrogen	110%	21%
Acetochlor	Organonitrogen	108%	13%
Alachlor	Organonitrogen	105%	12%
Propisochlor	Organonitrogen	81%	11%
Linuron	Organonitrogen	77%	17%
Metolachlor	Organonitrogen	79%	5%
Diphenamid	Organonitrogen	80%	6%
Metazachlor	Organonitrogen	105%	15%
Flutolanil	Organonitrogen	97%	27%
Oxadiazon	Organonitrogen	76%	11%
Atrazine	Organonitrogen	84%	17%
Terbuthylazine	Organonitrogen	90%	78%
Vinclozolin	Organonitrogen	98%	10%
Triadimefon	Organonitrogen	76%	10%
MGK-264	Organonitrogen	98%	12%
Fipronil	Organonitrogen	116%	28%
Fludioxonil	Organonitrogen	64%	37%
Myclobutanil	Organonitrogen	111%	36%
Flusilazole	Organonitrogen	70%	27%
Chlorfenapyr	Organonitrogen	63%	10%
Hexazinone (Velpar)	Organonitrogen	96%	10%
Tetramethrin I	Synthetic Pyrethroid	98%	5%
Tetramethrin II	Synthetic Pyrethroid	133%	9%
Bifenthrin	Synthetic Pyrethroid	82%	16%
cis-Permethrin	Synthetic Pyrethroid	61%	3%
trans-Permethrin	Synthetic Pyrethroid	64%	8%
Cyfluthrin	Synthetic Pyrethroid	191%	11%
Cypermethrin	Synthetic Pyrethroid	144%	17%
Flucythrinate I	Synthetic Pyrethroid	107%	6%
Flucythrinate II	Synthetic Pyrethroid	138%	6%
Fenvalerate S	Synthetic Pyrethroid	94%	10%
Fenvalerate R	Synthetic Pyrethroid	87%	7%
Chlorpropham	Herbicide Methyl Ester	102%	17%
Methacrifos	Organophosphorus	79%	12%
Sulfotep	Organophosphorus	96%	18%
Tolclofos-methyl	Organophosphorus	76%	17%
Bromophos ethyl	Organophosphorus	94%	20%
Ethion	Organophosphorus	82%	7%
Phorate	Organophosphorus	72%	10%
Fonofos	Organophosphorus	88%	19%
Methyl parathion	Organophosphorus	108%	16%
Triazophos	Organophosphorus	84%	25%
Piperonyl butoxide	Organophosphorus	61%	5%

Conclusions

Performance of in-cell clean-up using traditional. SPE sorbents proved to be highly efficient at removing non-target interferences (Figure #2). The process of performing the clean-up in-cell required no additional sample prep steps to be employed thus enabling a true one step automated extraction. Analysis of the extracts (spiked at .05 ug/g) showed good recoveries of an extensive lists of pesticides ranging across multiple chemical classes. Reproducibility of the extract sets yielded RSDs for most analytes <30%. (Table #3).

The final conclusion is the PLE can perform faster, more reliable and truly automated extractions for Cannabis samples when compared to traditional QuEChERS extractions. The flexibility of the process enables any combination of clean-up sorbents desired to be added with no need for centrifuging or wait times. Thus the PLE is an optimal choice for a combined extraction and clean-up solution for pesticide analysis.

Figure 3. FMS Inc. Pressurized Extraction System

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