Toxic Reports™

An Automated Technique for Fast, Ambient Temperature Extraction and Analysis of Multi-Residue Pesticides in Various Matrices

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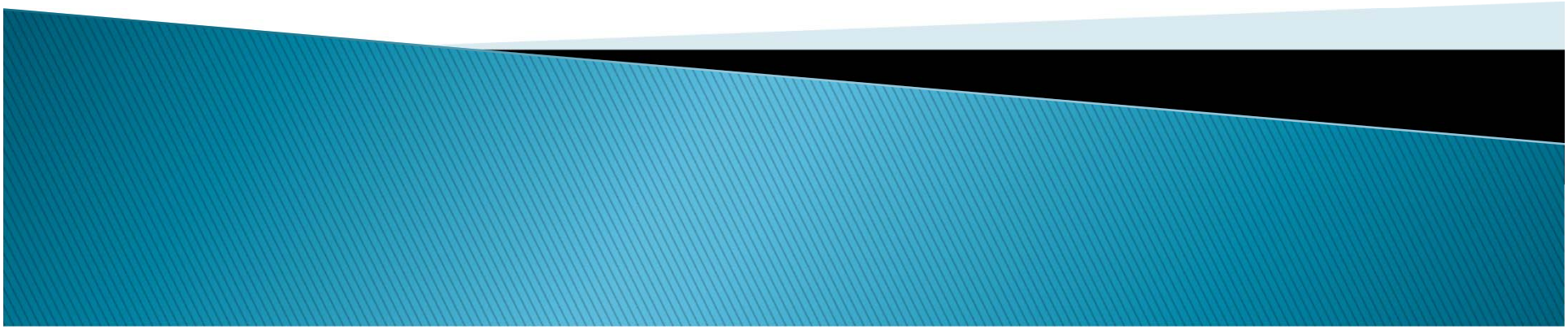

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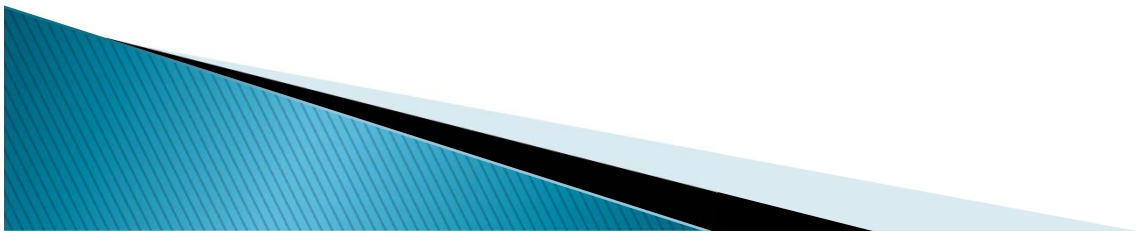
Introduction

- Food Safety
  - Pesticides cause harm to humans and pets
  - Identify potential risks in supply chain and products

- Analytical
  - Pesticide Analysis
  - Fast
  - Reproducible Results



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Pressurized Liquid Extraction

- An Extraction technique used in the Food Market
- The Technique Incorporates:
  - Solvent
  - Pressure
  - Heat
  - Time

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Effectiveness of PLE

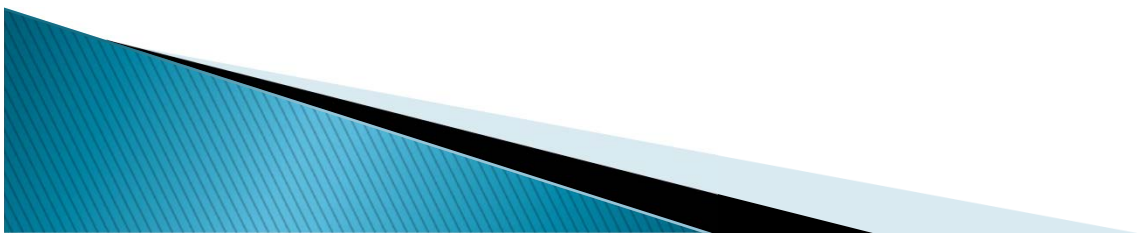
- Performed near the solvent's supercritical region
- Under Programmable Pressure
- Creates a high degree of analyte solubility releasing them from the solid matrix

Extraction (1)

- A solid or semi-solid sample is placed in the Pressurized Liquid Extraction Cell: 5 mL to 200 mL
- The Extraction cell is capped and placed into the extraction device which can be pressurized to up 2500 psi

Extraction (2)

- For pesticides, the system is pressurized for 5 min at 1000–1500 psi.
- No heat is applied – studies showed little effect of temperature on extraction efficiency.
- After depressurizing the cells are flushed with nitrogen to transfer all analytes to collection vessels.



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PLE® (1)

PLE – Pressurized Liquid Extraction

- High Speed
- Modular and expandable from 1 to 8
- Process 1 to 8 samples in 10 to 15 min
- Extraction cell size 5 to 200 ml
- Real time plot of temperature and pressure
- Reduced Solvent Consumption
- Lower Energy Consumption
- In Cell Sample Cleanup

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PLE® (2)

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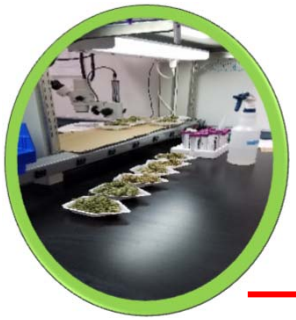
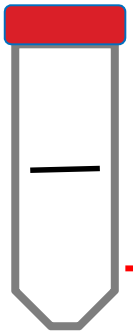
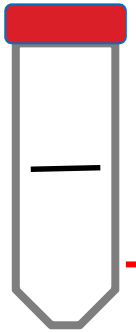
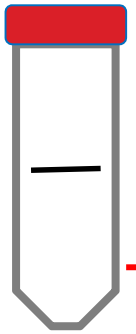
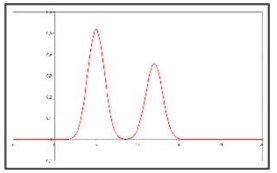
Standard Quechers Pesticide Workflow











2 minutes

Weigh the Sample

5 minutes

Load the Sample into the Vessel add H_2O and Acidified ACN

30 minutes

Shake Vessel

10 minutes

Add Quechers salt, shake and centrifuge

10 minutes

Extract Filtration

=

57 minutes

Sample Prep Total Time Ready for Injection

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Standard Quechers Pesticide Workflow

- Lots of Manual Steps and Human Interaction
  - More Error Prone due to interaction
- Labor and Solvent Intensive
  - Costs money
- Time Consuming Process
- Users Complain of Inconsistent Results

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PLE Extraction and Cleanup for Pesticides Workflow

2 minutes

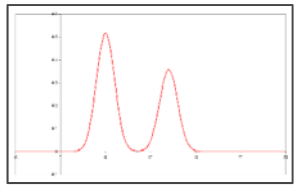
Weigh the Sample

2 minutes

Load the XtractClean™ and Sample into the Extraction Cell

8 minutes

Pesticide Extraction and In Cell Cleanup



= 12 minutes

Sample Prep Total Time Ready for Injection

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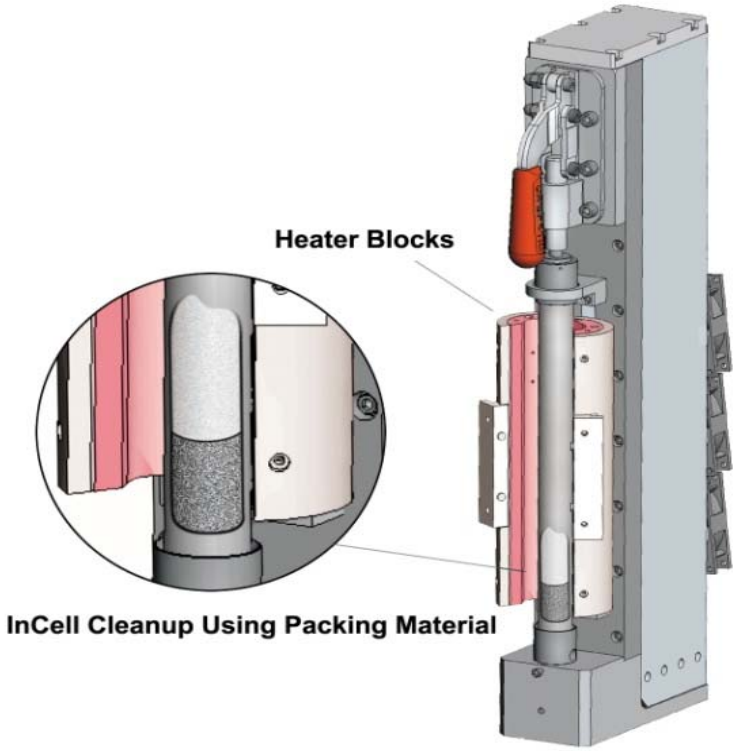
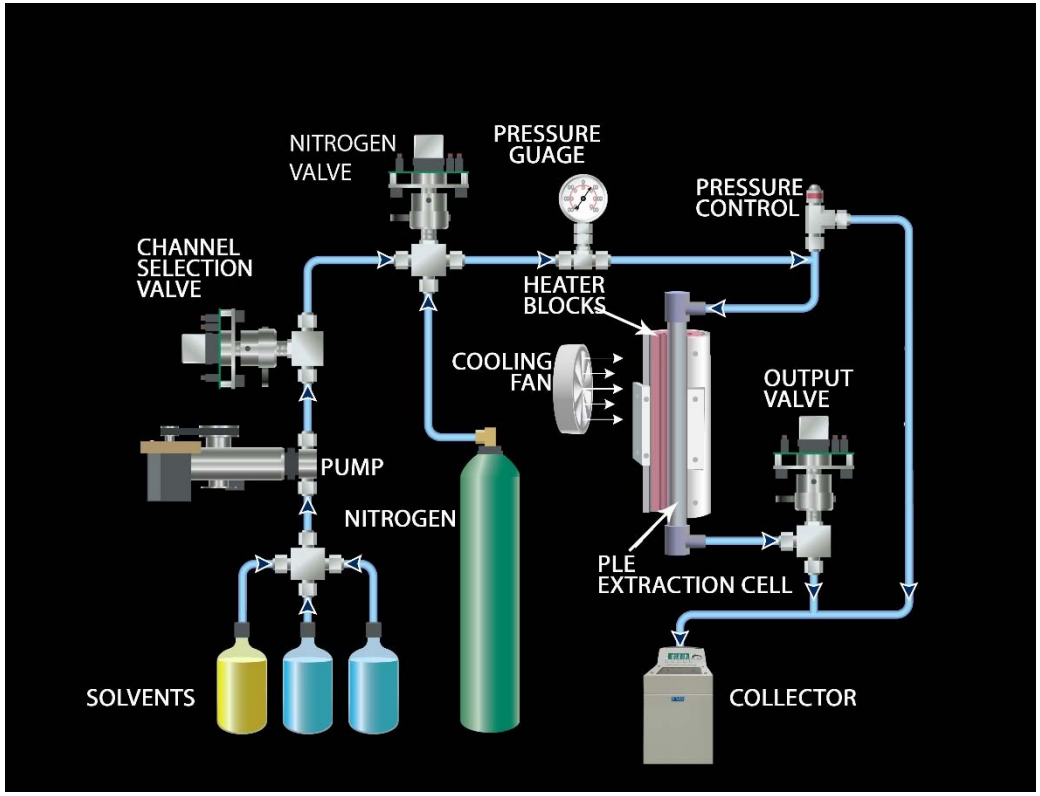
In Cell Cleanup for Pesticides

Eliminates Manual cleanup

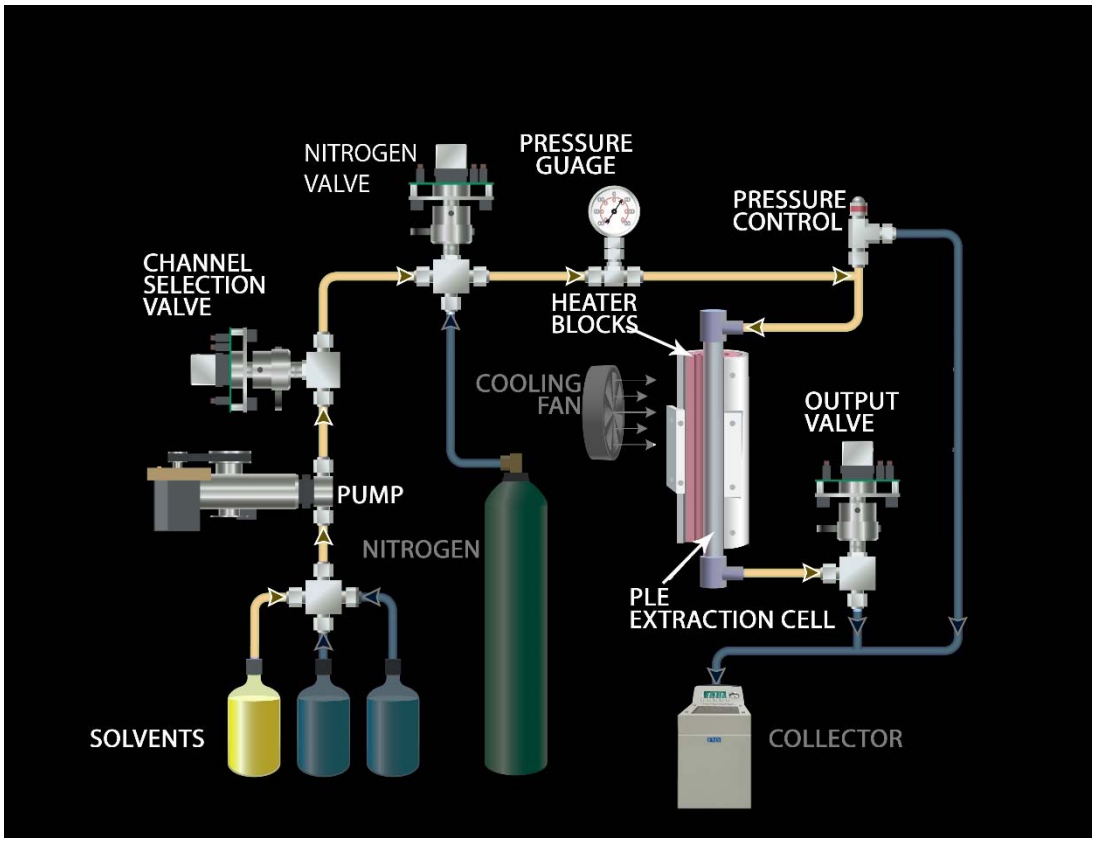
Uses In Cell Cleanup



Filling Cell with Acetonitrile



Pressurize the Cell



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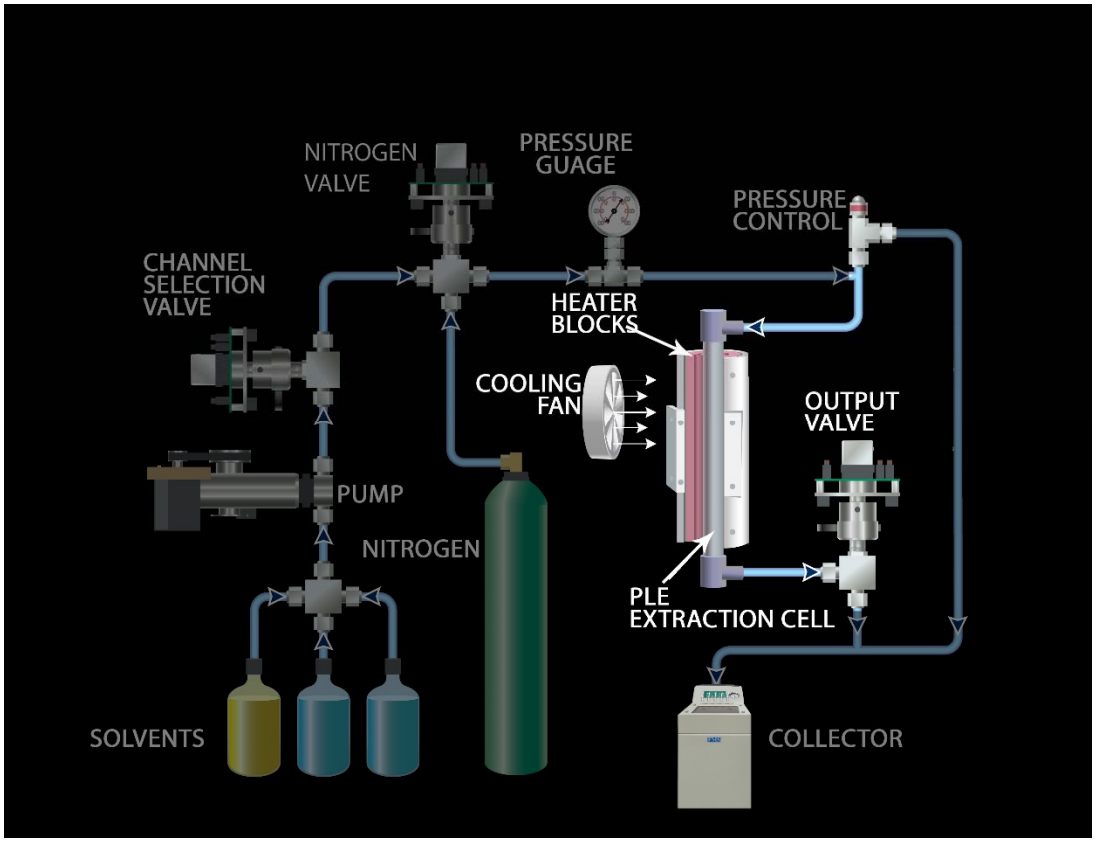
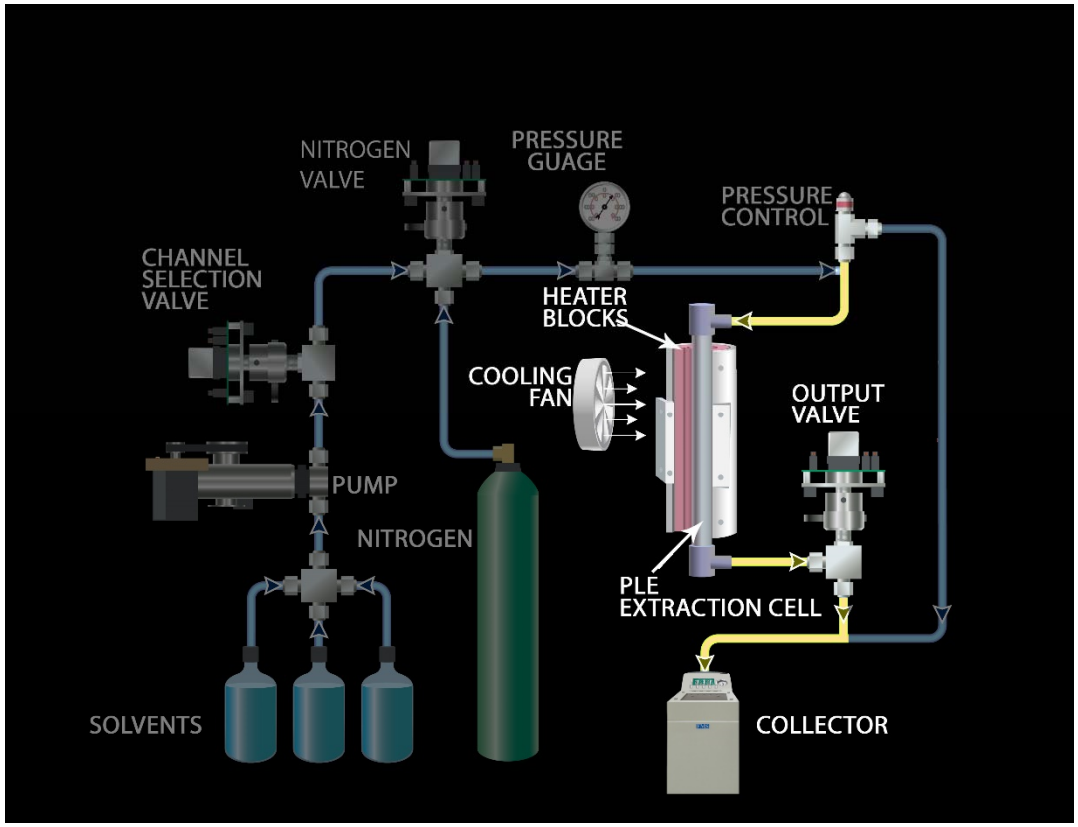
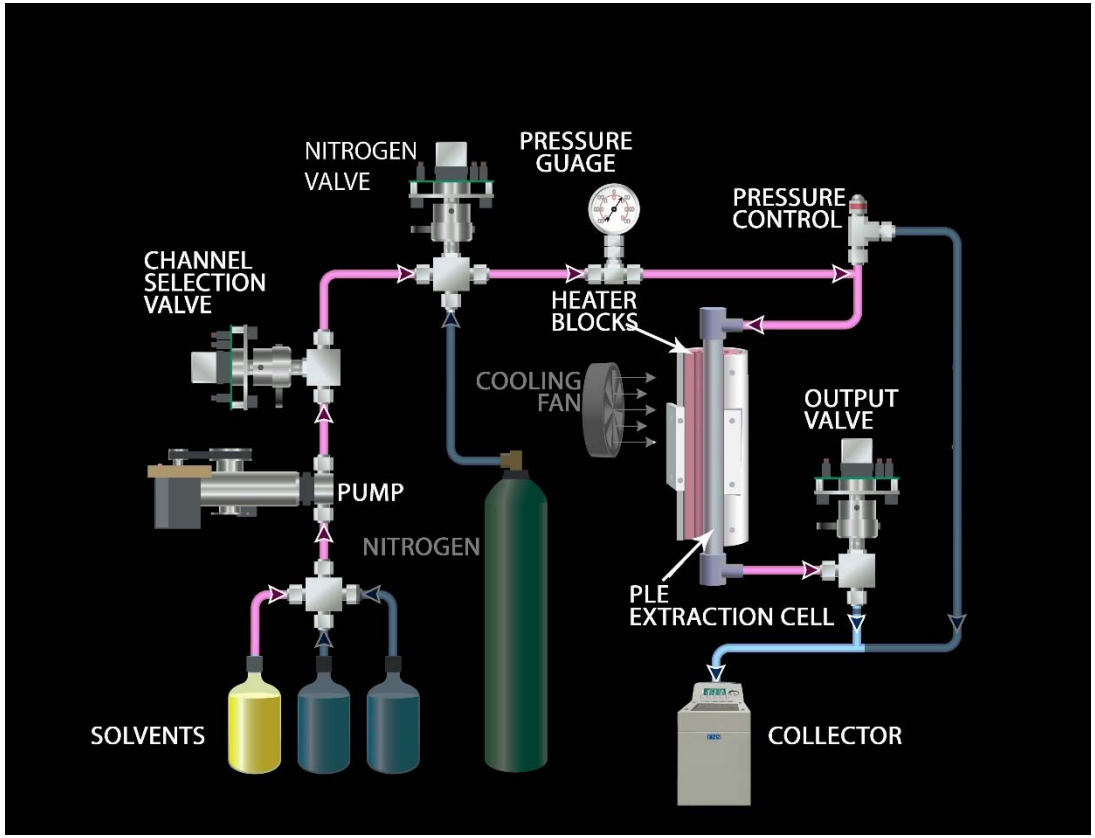
Maintain Pressure



Depressurize the Cell



Deliver the Extract to the Collection Vessel



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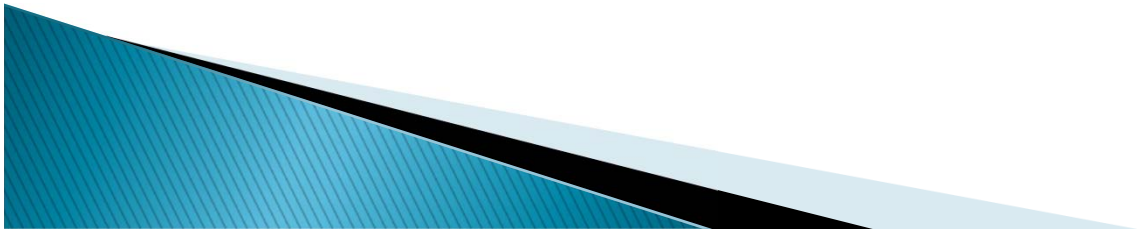
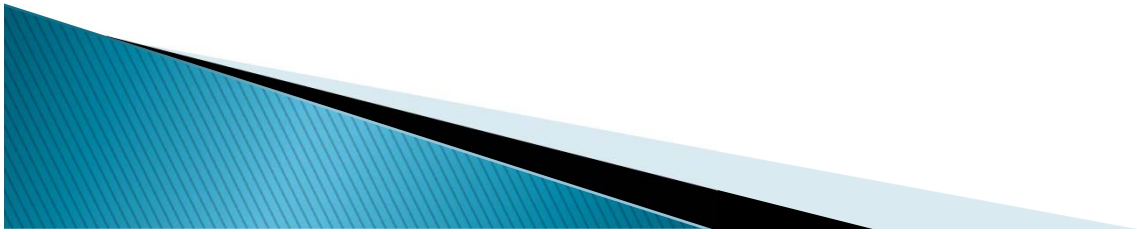
Preparing Sample

- **Sample/Reagent Prep**
- Sample aliquots are to be weighed out, thoroughly homogenized.
- Extraction cells are filled from bottom to top.
- Cover bottom frit with sufficient Ottawa Sand.
- Add sample aliquot on top of Sand.
- Add relevant standard mixes and internal standards.
- Add FMS CleanXtract™ mix in the cell on top of Sample.
- Add Ottawa Sand if needed to top off cell and place the cell in extraction system.



Extraction Procedure

- **PLE Procedure Pesticides**
- Cells are filled with Acetonitrile.
- Cells are pressurized for 5 min. Cells are not heated, ambient temperature process.
- Cells are flushed with Acetonitrile and with nitrogen to remove remaining solvent.
- Final extract is collected and a sample aliquot (1 mL) is transferred to a vial for GC/MS analysis. No volume reduction is needed.



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Analysis

- **GC/MS-MS Conditions**
  - **Thermo Trace GC w/PTV**
  - **TSQ Quantum Ultra**
  - **30 meter, .25mm, .25µm Column w/5 meter Guard column**
  - **203 Pesticides scanned (414 transitions)**

Results

- Sample Preparation for Extractions
  - Samples weighed and prepared.
  - Analyzed un-spiked and spiked to ensure no native pesticides of interest present
  - Samples spiked at .1 ug/g
  - Samples directly loaded onto GC with no evaporation

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Results: Organophosphosus and Pyrethroid Pesticides

Analyte	LCS		Green Tea		Black Tea		Fennel Seed		Astragulus Root		Harthorn		Gota Kola Berry		Green Coffee	
	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC
Diazinon	0.063	63%	0.11	110%	0.105	105%	0.08	80%	0.119	119%	0.113	113%	0.112	112%	0.113	113%
Pirimiphos methyl	0.099	99%	0.106	106%	0.096	96%	0.108	108%	0.081	81%	0.122	122%	0.12	120%	0.132	132%
Chlorpyrifos	0.075	75%	0.07	70%	0.079	79%	0.083	83%	0.073	73%	0.066	66%	0.079	79%	0.085	85%
Phosmet	0.08	80%	0.072	72%	0.076	76%	0.061	61%	0.072	72%	0.061	61%	0.101	101%	0.075	75%
Malathion	0.08	80%	0.098	98%	0.084	84%	0.107	107%	0.106	106%	0.112	112%	0.119	119%	0.111	111%
Piperonyl butoxide	0.068	68%	0.071	71%	0.076	76%	0.063	63%	0.074	74%	0.07	70%	0.095	95%	0.069	69%

Analyte	LCS		Green Tea		Black Tea		Fennel Seed		Astragulus Root		Harthorn		Gota Kola Berry		Green Coffee	
	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC
cis-Permethrin	0.085	85%	0.081	81%	0.087	87%	0.059	59%	0.077	77%	0.067	67%	0.104	104%	0.082	82%
trans-Permethrin	0.095	95%	0.093	93%	0.089	89%	0.061	61%	0.081	81%	0.085	85%	0.127	127%	0.094	94%
Cyfluthrin*	0.086	86%	0.082	82%	0.085	85%	0.051	51%	0.071	71%	0.073	73%	0.108	108%	0.085	85%
Cypermethrin*	0.070	70%	0.076	76%	0.085	85%	0.063	63%	0.076	76%	0.068	68%	0.115	115%	0.089	89%

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Results: Organonitrogen Pesticides

	LCS		Green Tea		Black Tea		Fennel Seed		Astragulus Root		Harthorn		Gota Kola Berry		Green Coffee	
Analyte	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC
Diphenylamine	0.072	72%	0.078	78%	0.075	75%	0.071	71%	0.076	76%	0.073	73%	0.091	91%	0.078	78%
2,3,5,6-Tetrachloroaniline	0.084	84%	0.091	91%	0.086	86%	0.083	83%	0.085	85%	0.09	90%	0.101	101%	0.088	88%
Propachlor	0.096	96%	0.107	107%	0.132	132%	0.1	100%	0.108	108%	0.103	103%	0.115	115%	0.106	106%
Dimethachlor	0.09	90%	0.097	97%	0.097	97%	0.089	89%	0.086	86%	0.11	110%	0.106	106%	0.101	101%
Linuron	0.082	82%	0.103	103%	0.101	101%	0.104	104%	0.079	79%	0.102	102%	0.124	124%	0.113	113%
Flutolanil	0.089	89%	0.089	89%	0.085	85%	0.228	228%	0.111	111%	0.156	156%	0.1	100%	0.109	109%
Methoxychlor	0.096	96%	0.097	97%	0.096	96%	0.07	70%	0.095	95%	0.087	87%	0.132	132%	0.111	111%
Pyridaben	0.073	73%	0.048	48%	0.08	80%	0.055	55%	0.06	60%	0.063	63%	0.062	62%	0.104	104%
Attrazine	0.103	103%	0.093	93%	0.095	95%	0.095	95%	0.066	66%	0.094	94%	0.082	82%	0.084	84%
MGK-264 I	0.064	64%	0.064	64%	0.063	63%	0.073	73%	0.057	57%	0.053	53%	0.065	65%	0.067	67%
MGK-264 II	0.059	59%	0.057	57%	0.047	47%	0.068	68%	0.039	39%	0.059	59%	0.031	31%	0.055	55%
MGK-264*	0.0615	62%	0.0605	61%	0.055	55%	0.0705	71%	0.048	48%	0.056	56%	0.048	48%	0.061	61%
Captan	0.061	61%	0.107	107%	0.035	35%	0.084	84%	0.08	80%	0.139	139%	0.044	44%	0.041	41%
Fludioxonil	0.071	71%	0.108	108%	0.072	72%	0.183	183%	0.088	88%	0.148	148%	0.1	100%	0.1	100%
Tebuconazole	0.051	51%	0.092	92%	0.084	84%	0.073	73%	0.079	79%	0.119	119%	0.103	103%	0.082	82%
Etofenprox	0.073	73%	0.078	78%	0.081	81%	0.051	51%	0.067	67%	0.066	66%	0.099	99%	0.08	80%

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Results: Organochlorine Pesticides & Methylated Herbicides

	LCS		Green Tea		Black Tea		Fennel Seed		Astragulus Root		Harthorn		Gota Kola Berry		Green Coffee	
Analyte	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC	Conc.	REC
HCH-alpha	0.085	85%	0.083	83%	0.079	79%	0.072	72%	0.075	75%	0.076	76%	0.088	88%	0.085	85%
Pentachlorothioanisole	0.071	71%	0.075	75%	0.08	80%	0.069	69%	0.064	64%	0.072	72%	0.078	78%	0.071	71%
4,4'-Dichlorobenzophenone	0.074	74%	0.075	75%	0.079	79%	0.075	75%	0.063	63%	0.076	76%	0.078	78%	0.079	79%
Chlorfenson (Ovex)	0.063	63%	0.073	73%	0.077	77%	0.1	100%	0.065	65%	0.078	78%	0.072	72%	0.074	74%
2,4'-DDT	0.085	85%	0.083	83%	0.085	85%	0.072	72%	0.075	75%	0.063	63%	0.11	110%	0.078	78%
Mirex	0.081	81%	0.079	79%	0.087	87%	0.079	79%	0.066	66%	0.06	60%	0.102	102%	0.073	73%
2-Phenylphenol	0.08	80%	0.086	86%	0.101	101%	0.083	83%	0.087	87%	0.084	84%	0.093	93%	0.088	88%

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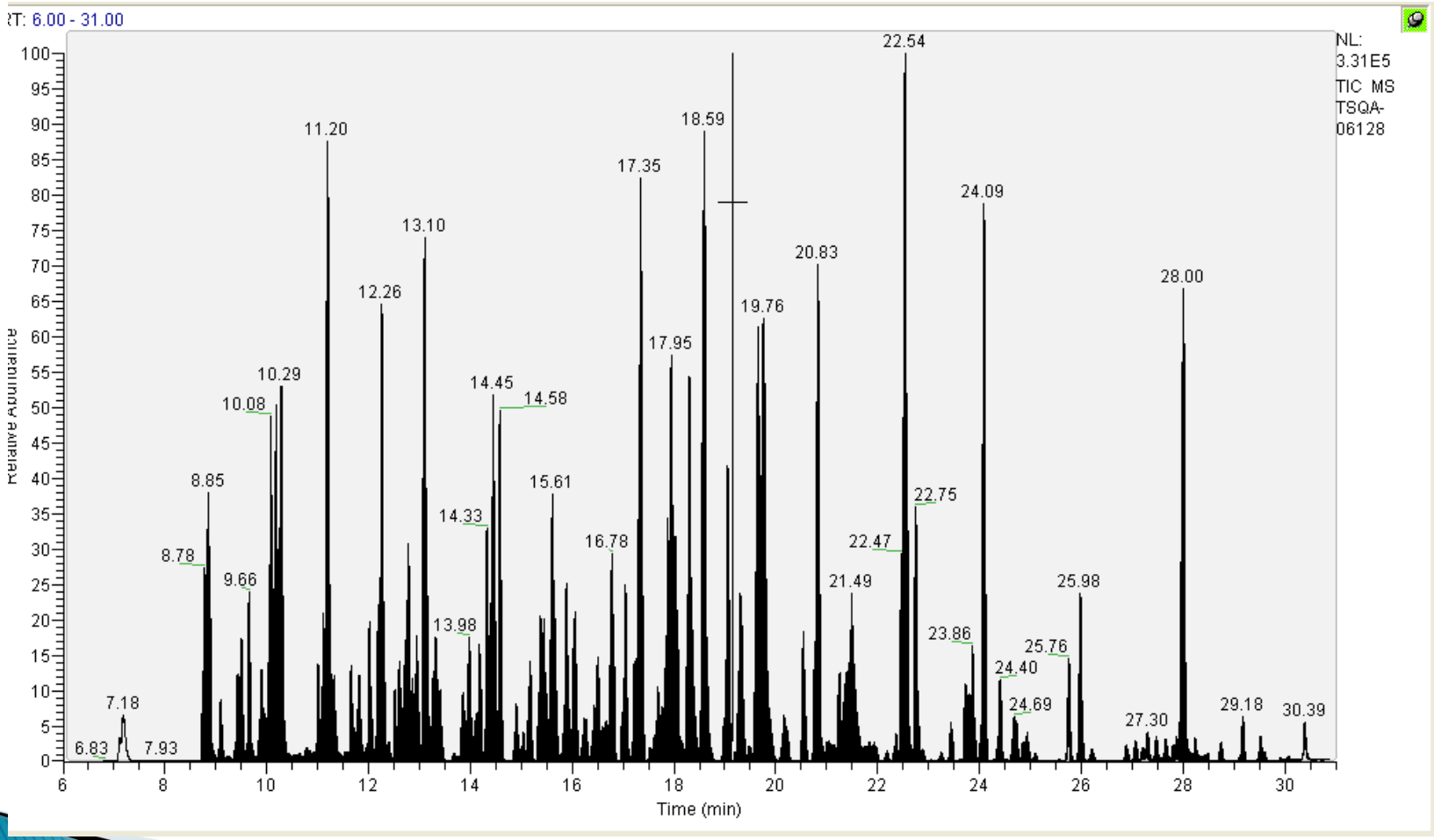
Results : TIC of Spiked Green Tea Extract from the PLE w/In-Cell Clean-up



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Results: pesticides in cannabis

Analyte	Mean	Analyte	Mean
Diazinon	92%	Linuron	77%
Chlorpyrifos methyl	78%	Metolachlor	79%
Fenitrothion	108%	Diphenamid	80%
Pirimiphos methyl	76%	Metazachlor	105%
Chlorpyrifos	80%	Flutolanil	97%
Pirimiphos ethyl	75%	Oxadiazon	76%
Quinalphos	74%	Attrazine	84%
Phosalone	94%	Terbuthylazine	90%
Chlorneb	90%	Vinclozolin	98%
HCH-alpha	74%	Triadimefon	76%
Pentachloroanisole	70%	MGK-264*	98%
HCH-delta	86%	Fipronil	116%
Heptachlor	90%	Fludioxonil	64%
Heptachlor epoxide (isomer B)	84%	Myclobutanil	110%
Chlorfenson (Ovex)	92%	Flusilazole	70%
Endosulfan II	102%	Chlorfenapyr	63%
Tetrachloronitrobenzene (Tecnazene)	92%	Hexazinone (Velpar)	96%
THPI (Tetrahydrophthalimide)	96%	Tetramethrin I	98%
Diphenylamine	88%	Tetramethrin II	133%
2,3,5,6-Tetrachloroaniline	76%	Bifenthrin	82%
Pentachlorobenzene (Quintozene)	108%	cis-Phenothrin	61%
Pentachlorobenzonitrile	98%	trans-Phenothrin	64%
Prodiamine	111%	Cypermethrin*	144%
Isopropalin	98%	Flucythrinate I	107%
Pendamethalin	63%	Flucythrinate II	138%
Oxyfluorfen	90%	Fenvalerate S	94%
Nitralin	87%	Fenvalerate R	87%
Pebulate	84%	Chlorpropham	102%
N-(2,4-Dimethylphenyl)formamide	86%	Methacrifos	79%
cis-Diallate	85%	Sulfotepp	96%
trans-Diallate	111%	Tolclofos-methyl	76%
Clomazone (Command)	78%	Bromophos methyl	94%
Propyzamide	79%	Ethion	82%
Dimethachlor	108%	Phorate	72%
Propanil	110%	Fonofos	88%
Acetochlor	108%	Methyl parathion	108%
Alachlor	105%	Triazophos	84%
Propisochlor	81%	Piperonyl butoxide	61%

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Conclusions: PLE for the Analysis of Pesticides

- High Throughput Pesticide Analysis
  - 20 minutes per run up to 24 samples per hour
  - 192 samples per 8 hour Shift
- One Extraction Method for all Matrices
- One/Same Extraction for GC/MS and LC/MS analysis
- Eliminate Manual Steps and Human Error
  - Automated Extraction and Cleanup

Toxic Reports™

Fast, Reproducible Extractions

- Using the PLE®
  - Sample Prep processes are combined into one step
    - Extraction
    - Cleanup
  - Put the sample in get it out and directly inject it
  - Consistent, Reproducible, Results
  - Increased productivity

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Fast, Reproducible Extractions

- Faster and easier operator training
- Automatic documentation of extraction and cleanup and concentration conditions
- Reduced errors due to mistakes eliminating manual steps and conditions.
- Reduced solvent usage and disposal costs.