FMS
Fluid Management Systems

Fast, Reliable Environmental and Food Extractions

PLE®
Pressurized Liquid Extraction

Agenda

- FMS PLE Overview
  - Pressurized Liquid Extraction
- Demonstration
- Questions

FMS, Inc.

- Fluid Management Systems
  - Founded in 1986
  - Focus
    - Automating the Sample Prep Process
  - Markets
    - Agricultural
    - Environmental
    - Clinical
    - Food and Beverage
    - Pharmaceutical
    - Petrochemical

FMS,Inc.

- Manufacture all products in Billerica, MA USA
- Applications Lab
- Sales and Service Worldwide
  - Manufacturer reps Domestic and International
  - Applications Specialists
  - Service Engineers

Our Business

- Fastest growing segments
  - Food and Beverage
    - Rapid Screening for POPs
    - Partner with Agilent to provide the total solution
    - One Step Automated Sample Prep
  - Environmental
    - One Step Automated Sample Prep
      - Put the sample in the get it out on the other end and inject

Testing in Food

- Food Safety
  - Pesticides cause Harm to Humans/Pets
  - Identify potential risks to your Supply Chain and Product line

- Analytical
  - Pesticide Analysis
  - Fast
  - Reproducible Results

Pressurized Liquid Extraction

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

Why is the PLE so effective?

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

Extraction

- A solid or semi-solid sample is placed in the Pressurized Extraction Cell 5ml to 200ml

- The Extraction cell is capped and placed into the extraction device which can be pressurized to up 2500psi

Extraction

- The Extraction cell is placed under pressure, then heated and then cooled
- For Pesticides
  - No Heat
- The Extract is flushed with Nitrogen directly to the SuperVap Concentrator

The PLE®
Pressurized Liquid Extraction

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
- Direct to Concentrator and GC vial

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Touch Screen Control

SD Card

Concentration/Evaporation

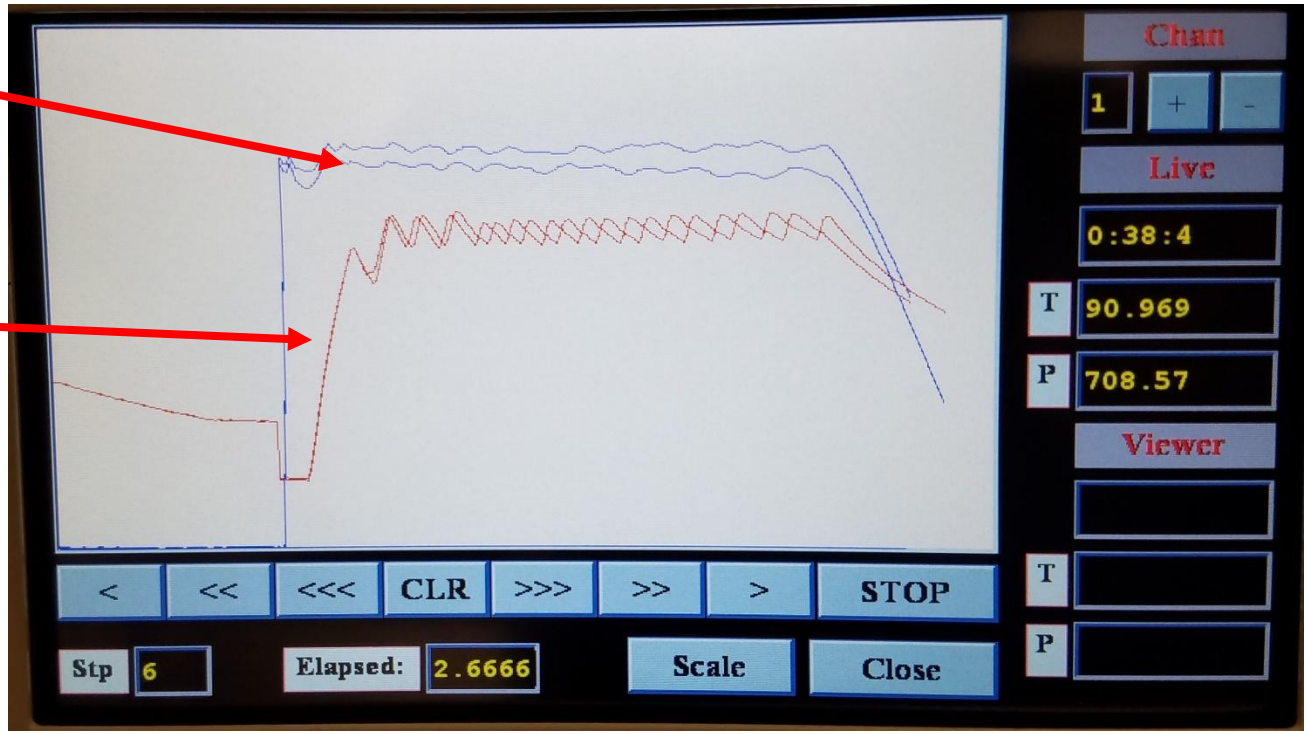
High Pressure Pump

Up to 6 Solvents

Method Documentation

Pressure

Temperature



Modular and Expandable

Expandable from 1 to 8 Modules

Parallel Extraction

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Economical Extraction Cells

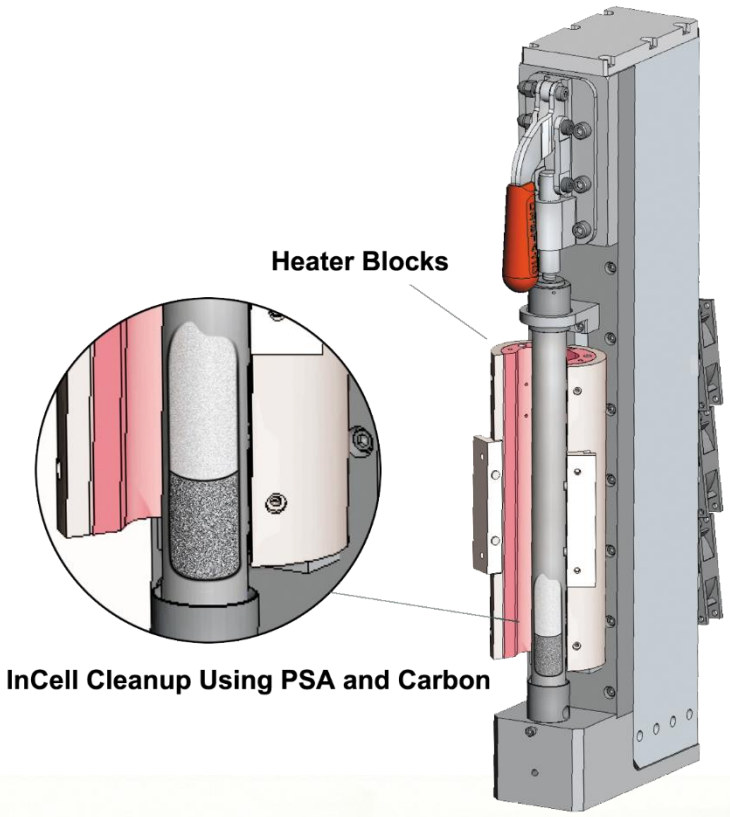
Easy to Use End Caps

In Cell Cleanup for Pesticides

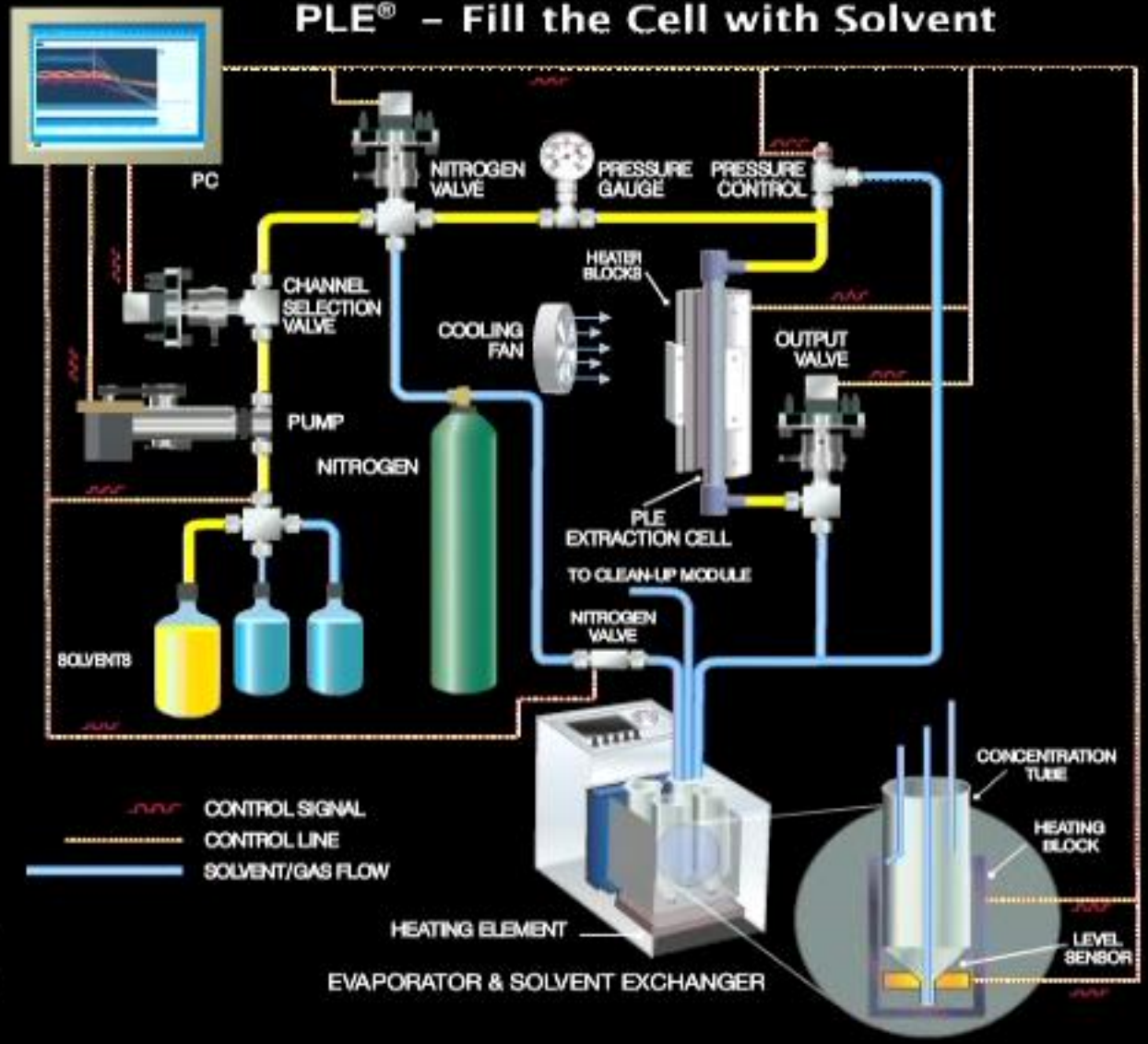
Eliminates Manual cleanup

Uses In Cell Cleanup

- Florisil
- PSA
- Carbon
- Silica



PLE® – Fill the Cell with Solvent



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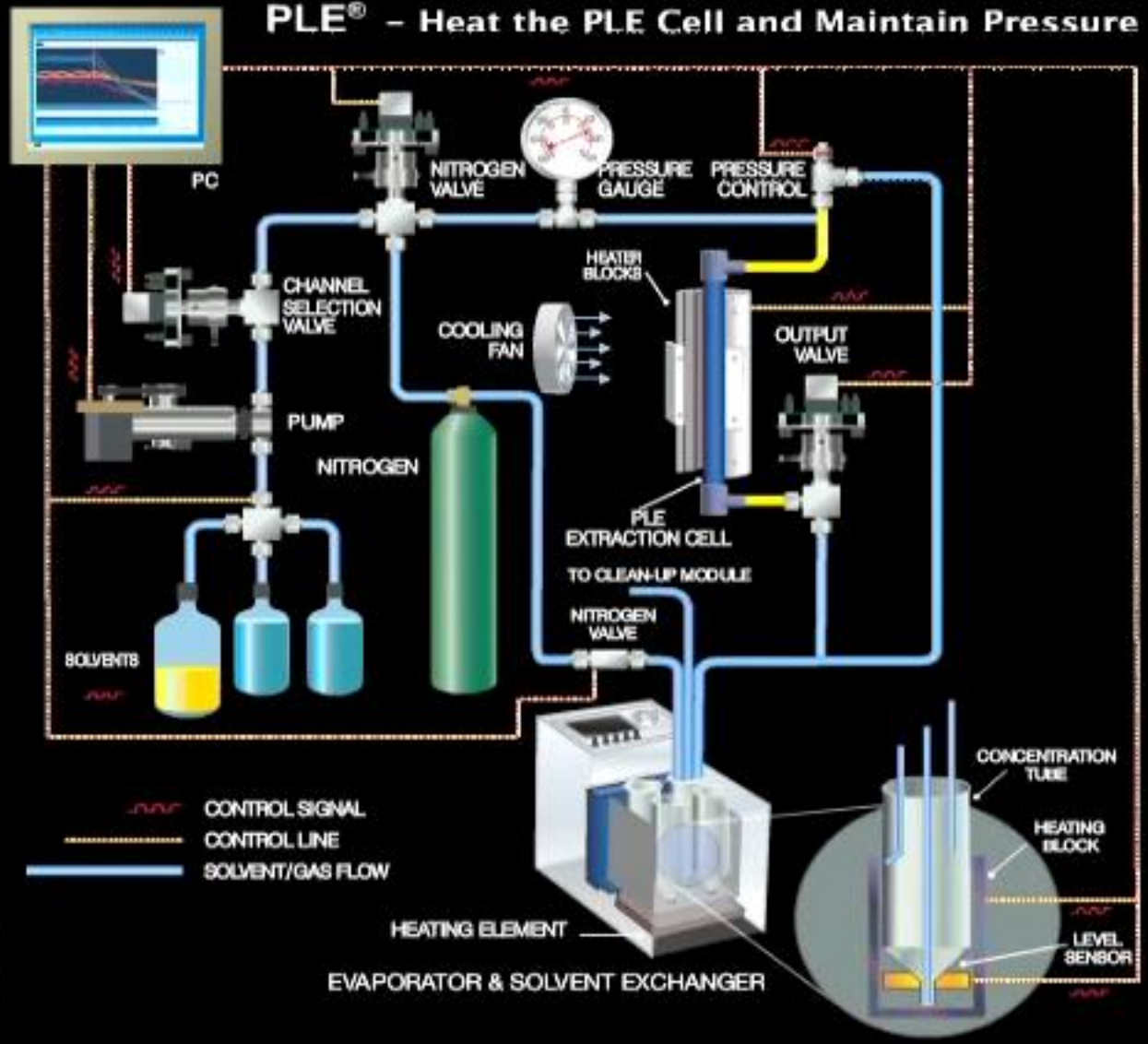
PLE® – Heat the PLE Cell and Maintain Pressure



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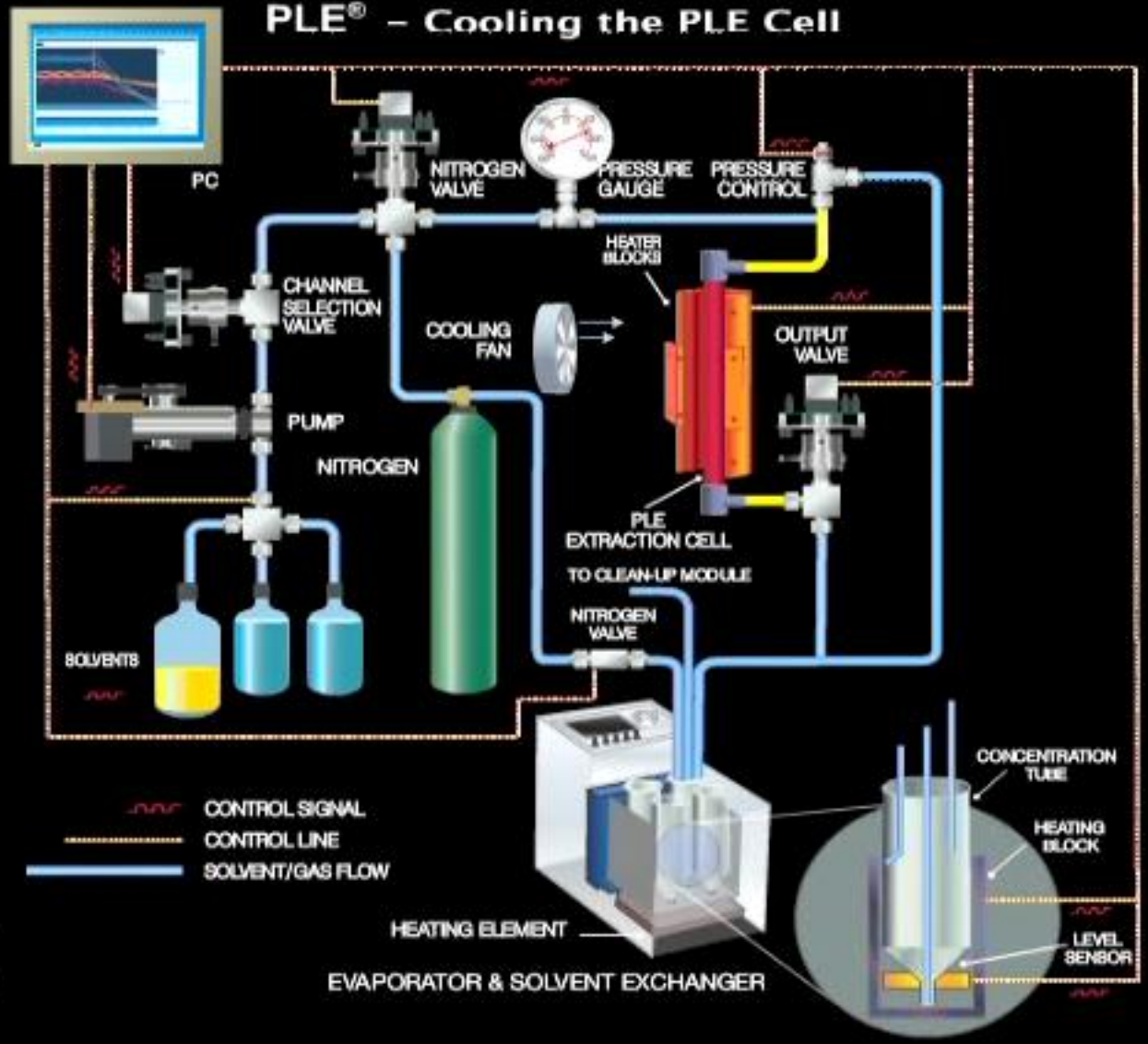

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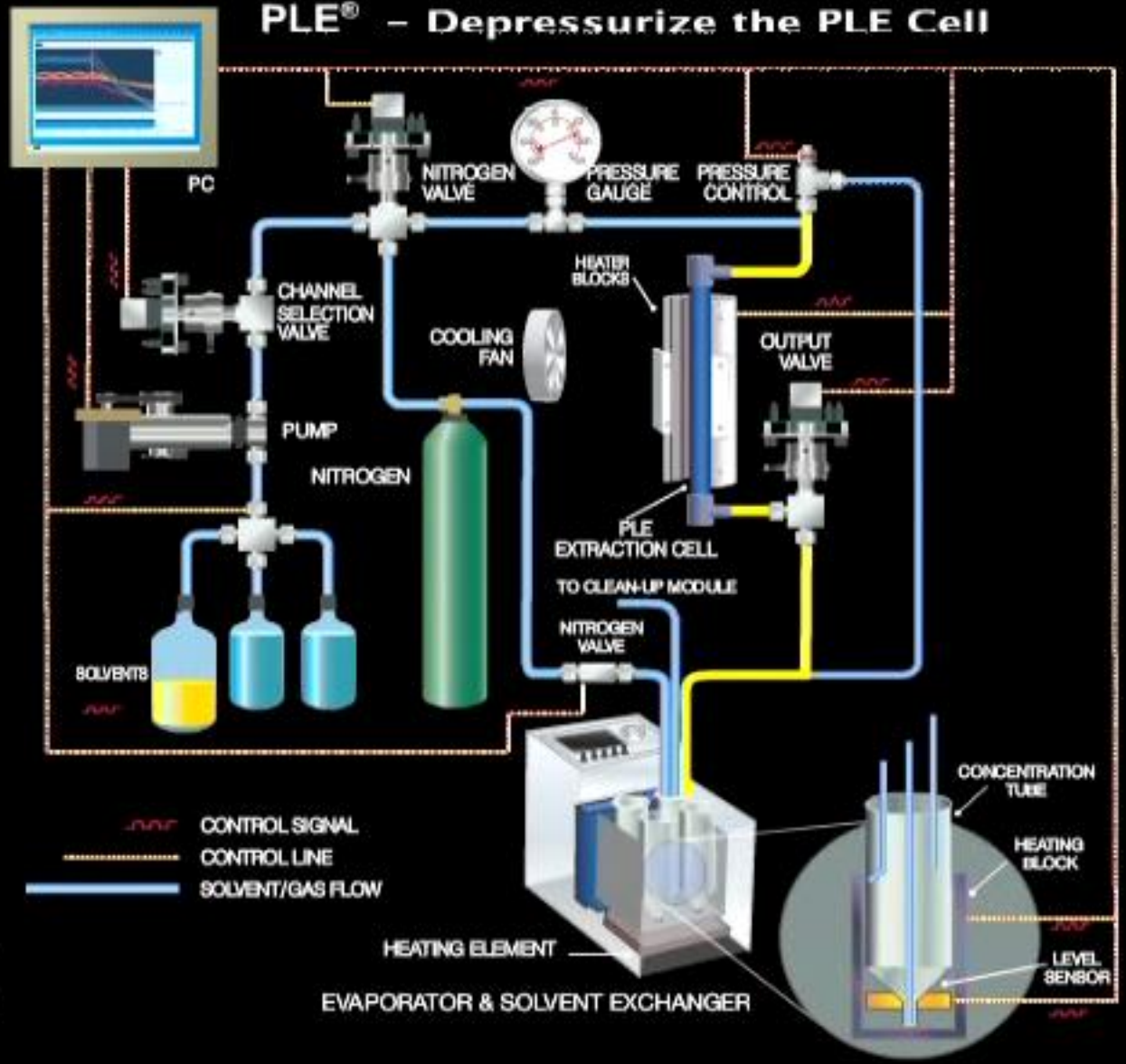

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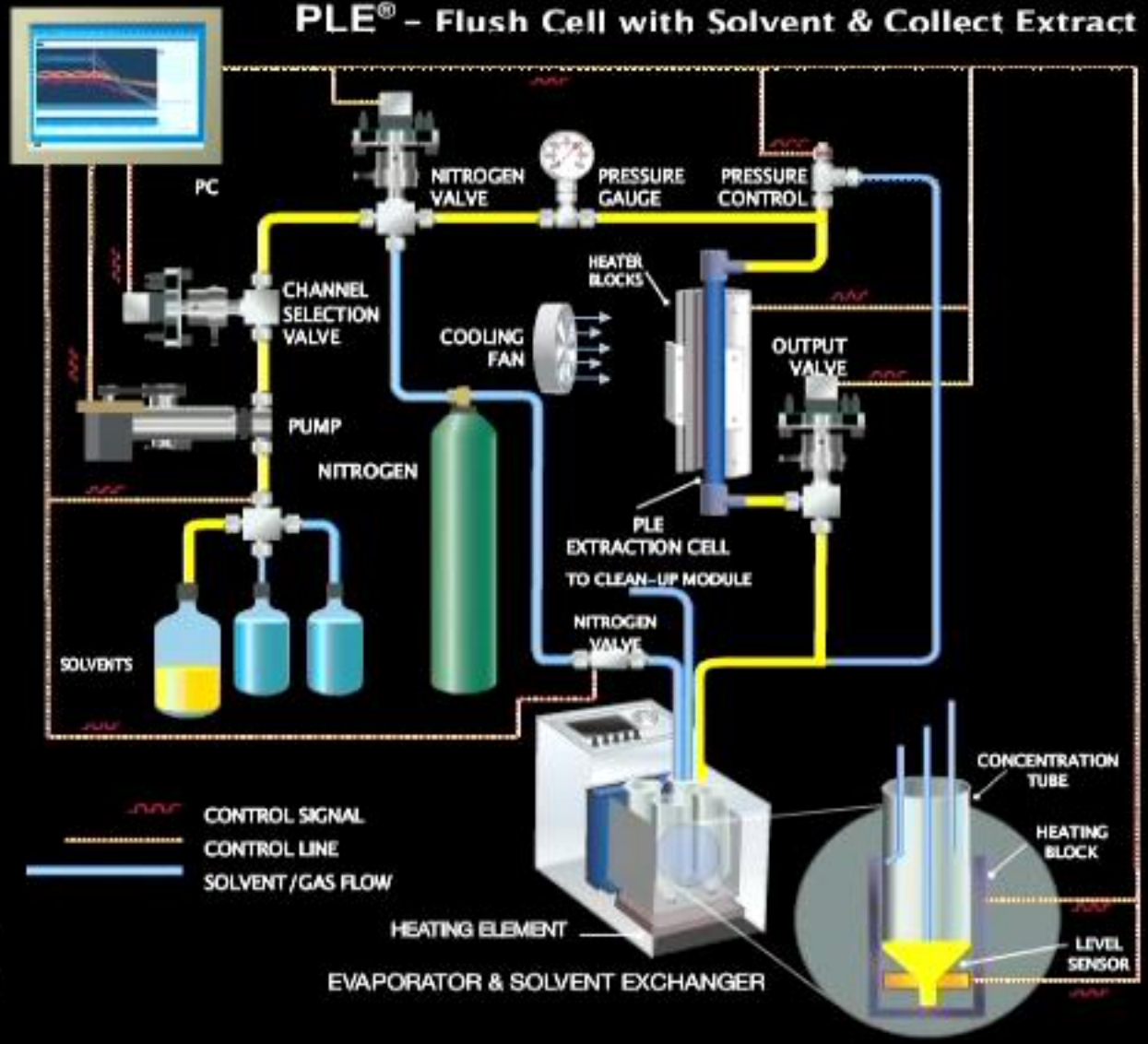

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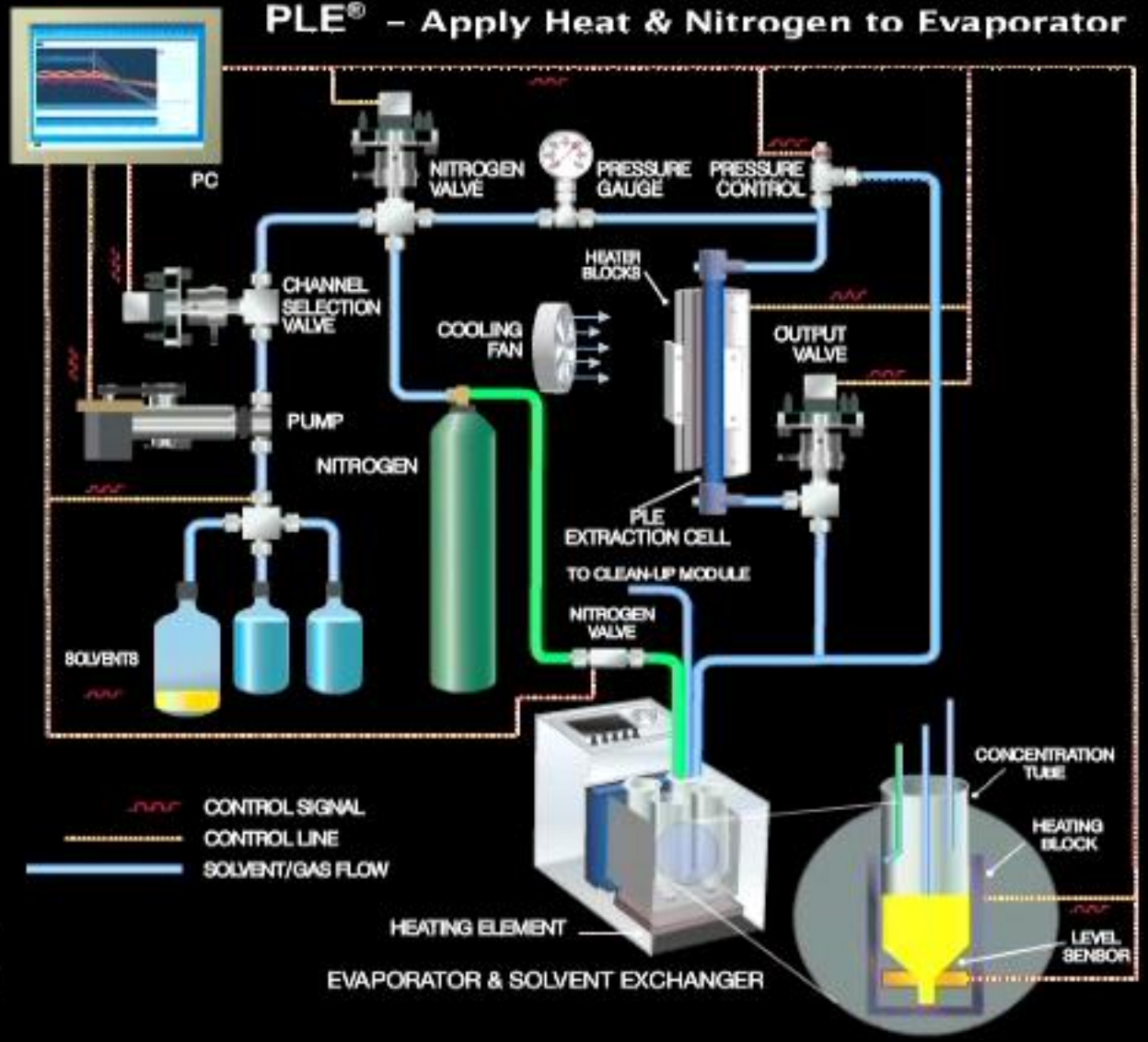
PLE® – Apply Heat & Nitrogen to Evaporator



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PCB Congeners

		Sand		Sediment	
Congener	Spiked ug/kg	Mean Recovery ug/ml	%	Mean Recovery ug/ml	%
BZ #1	100	114.2	114.2%	75.2	75.2%
BZ #5	100	125.2	125.2%	81.3	81.3%
BZ #18	100	119.3	119.3%	93.3	93.3%
BZ #31	100	120.6	120.6%	85.1	85.1%
BZ #44	100	119	119.0%	85.2	85.2%
BZ #52	100	119.2	119.2%	91	91.0%
BZ #66	100	143.6	143.6%	89	89.0%
BZ #87	100	124.7	124.7%	84.7	84.7%
BZ #101	100	122.8	122.8%	89.8	89.8%
BZ #110	100	111.7	111.7%	84.8	84.8%
BZ #138	100	109.4	109.4%	87	87.0%
BZ #141	100	106	106.0%	92.7	92.7%
BZ #151	100	113.1	113.1%	91	91.0%
BZ #153	100	113.3	113.3%	91.4	91.4%
BZ #170	100	120.8	120.8%	86.2	86.2%
BZ #180	100	100.7	100.7%	95	95.0%
BZ #183	100	100.1	100.1%	95	95.0%
BZ #187	100	103.6	103.6%	87.9	87.9%
BZ #206	100	103.2	103.2%	79.3	79.3%
TCMX (IS)	100	102.1	102.1%	96.9	96.9%

Toxaphene in Fish

	2.5 gram	Sample	
Compound	**Avg. Conc.**	**% rec.**	**RPD**
TCMX	27.8 ug/kg	70%	10%
Decachlorobiphenyl	34.8 ug/kg	87%	2%
Toxaphenne	121.2 ug/kg	76%	12%
	5 gram	**Sample**	
Compound	**Avg. Conc.**	**% rec.**	**RPD**
TCMX	12.8 ug/kg	72%	3%
Decachlorobiphenyl	18 ug/kg	74.5%	1%
Toxaphenne	57.62 ug/kg	98.5%	7%

Pesticides in Soil

Compound	Recovery (ug/ml)	Recovery
TCMX	4.77	95.4%
Alpha-BHC	4.57	91.4%
Beta-BHC	4.44	88.8%
Gamma-BHC	4.44	88.8%
Delta-BHC	4.46	89.2%
Heptachlor	4.68	93.6%
Aldrin	4.26	85.2%
Heptachlor Epoxide	4.75	95.0%
Gamma-Chlordane	4.23	84.6%
Endosulfan	4.39	87.8%
Alpha-Chlordane	4.22	84.4%
Dieldrin	4.65	93.0%
4,4'-DDE	4.33	86.6%
Endrin	5.2	104.0%
Endosulfan II	5.14	102.8%
Endrin Aldehyde	5	100.0%
4,4'-DDD	4.55	91.0%
Endosulfan Sulfate	4.55	91.0%
4,4'-DDT	4.67	93.4%
Endrin Ketone	4.8	96.0%
Methoxychlor	5.11	102.2%
Deca-PCB	5.37	107.4%

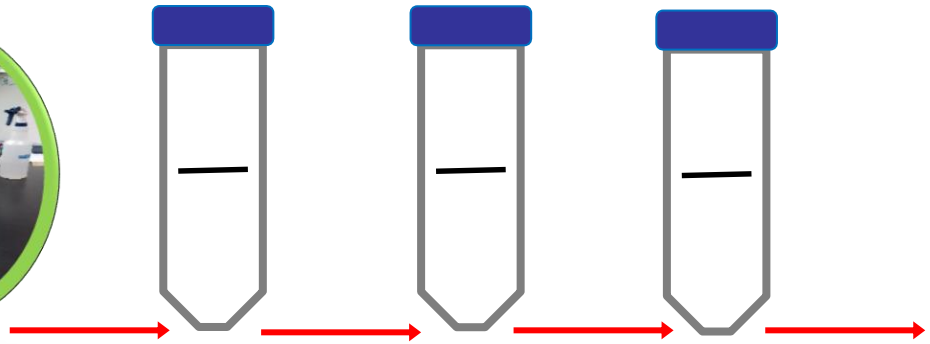
Antioxidants from High Density Polyethylene

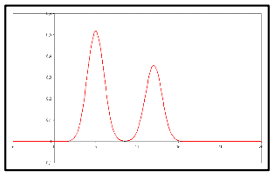
	Cell #1	Cell #2	RSD
Erucamide®	70.80%	71.60%	0.712
Irganox® 1010	81.30%	81.00%	0.8115
Irganox® 1076	81.20%	80%	0.806
Irgafos® 168	116.60%	115.60%	1.161

PAHs in Soil

Compound	Mean Rec.	STD STDEV
Naphthalene	85.1%	2.1%
2-Methylnaphthalene	91.5%	2.0%
1-Methylnaphthalene	88.9%	2.1%
Acenaphthylene	101.5%	1.9%
Acenaphthene	96.5%	2.5%
Fluorene	96.9%	3.3%
Phenanthrene	89.1%	4.6%
Anthracene	116.9%	4.5%
Fluoranthene	102.6%	5.9%
Pyrene	101.1%	5.6%
Benzo[a]anthracene	97.4%	4.6%
Chrysene	104.7%	5.1%
Benzo[b]fluoranthene	90.0%	7.1%
Benzo[k]fluoranthene	95.2%	3.7%
Benzo[a]pyrene	89.5%	3.7%
Indeno[1,2,3-cd]pyrene	82.0%	4.7%
Dibenzo[a,h]anthracene	78.7%	4.5%
Benzo[g,h,i]perylene	83.3%	4.4%

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

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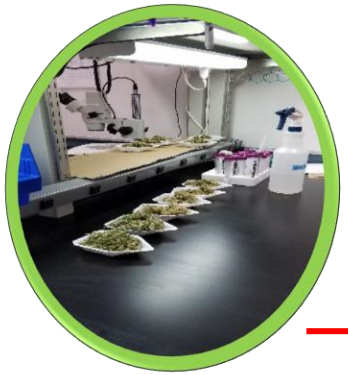
52 minutes

Sample Prep Total Time Ready for Injection

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

PLE Extraction and Cleanup for Pesticides Workflow

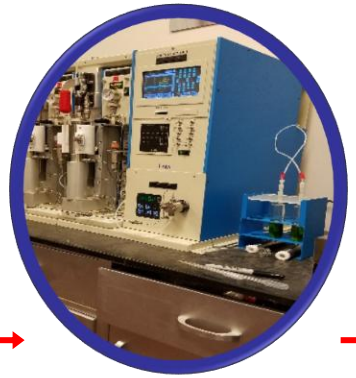


2 minutes

Weigh the Sample

2 minutes

Load the XtractClean™ and Sample into the Extraction Cell



8 minutes

Pesticide Extraction and In Cell Cleanup

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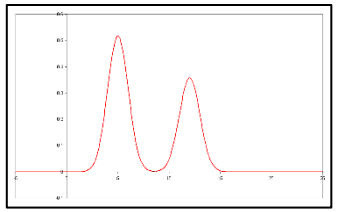
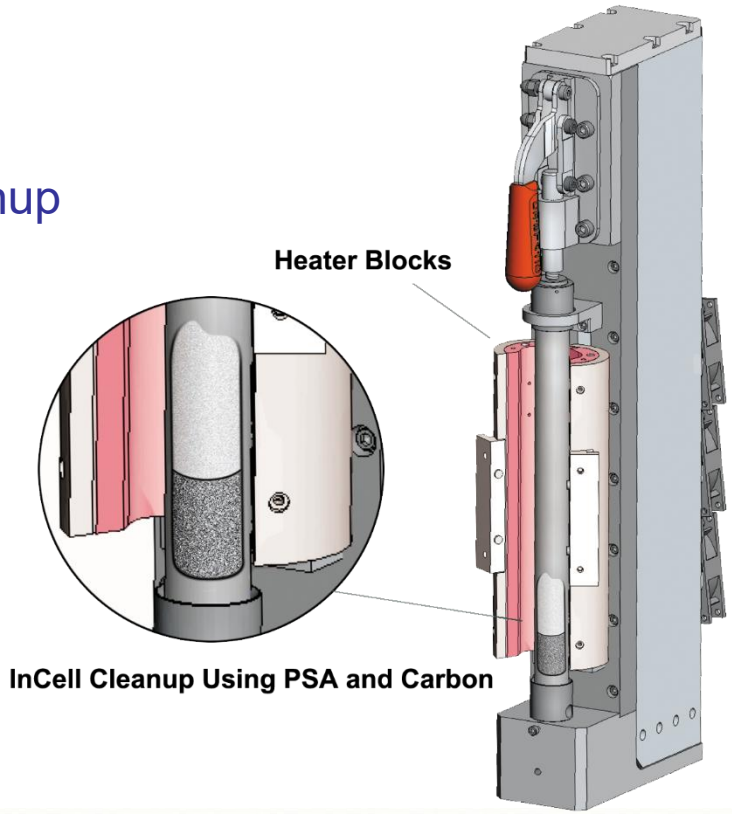


12 minutes

Sample Prep Total Time Ready for Injection

InCell Cleanup for Pesticides

Eliminates Manual cleanup



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.

Results:
Organophosphorus 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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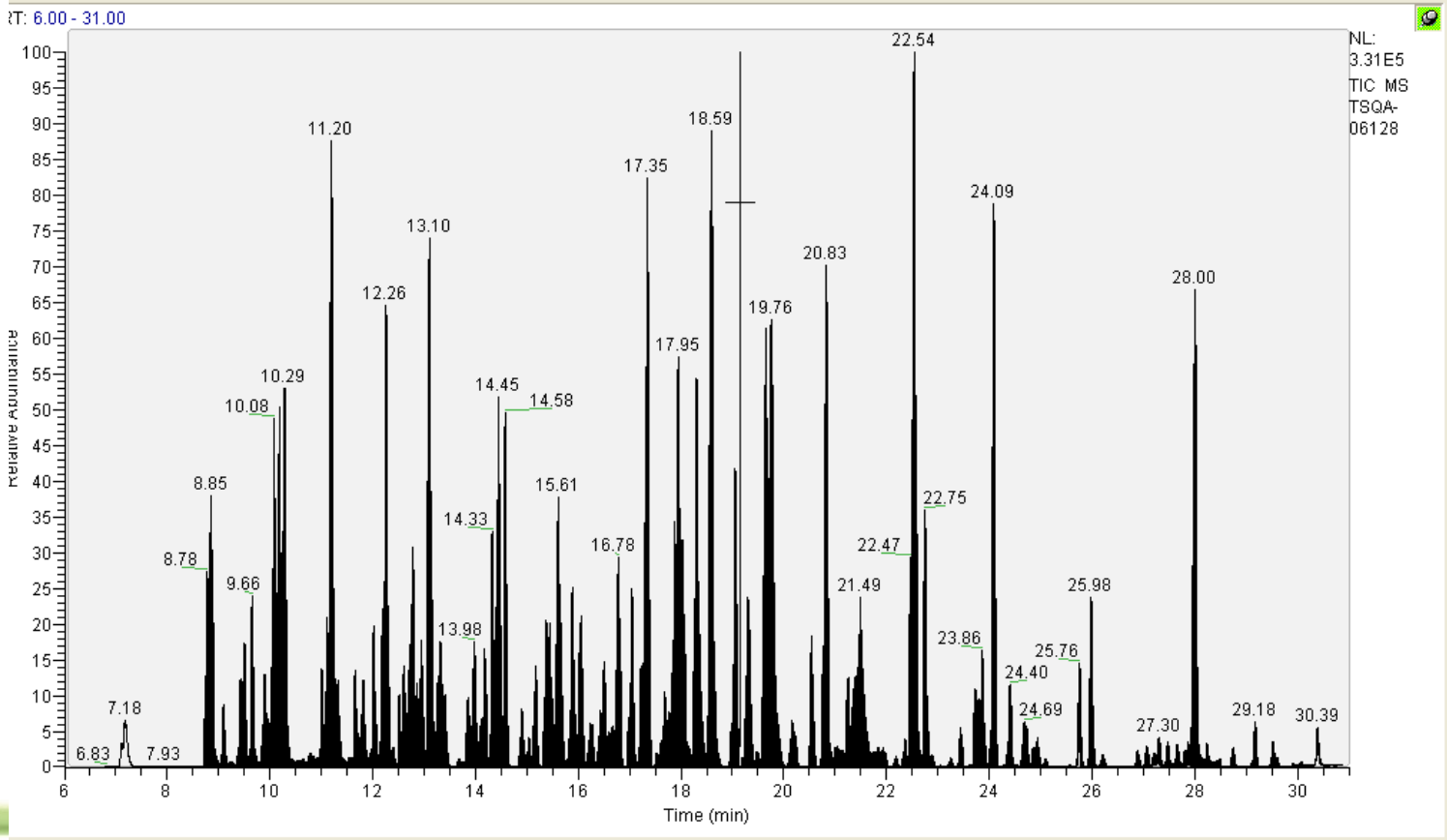
Results
Organonitrogen Pesticides

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
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%

Results
Organochlorine Pesticides & Methylated Herbicides

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
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%

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



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

Fast, Reproducible Extractions

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

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.

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Questions?