Agilent Sample Preparation
The Pesticide Analysis Workflow

Insights into Preparing your Cannabis Sample and Triple Quad Mass Spec Analysis

Joan Stevens, Ph.D., and Tina Chambers

Sample Preparation Application Specialists

Outline

- Introduction to Plant Material and Edibles
- Existing Sample Prep Techniques
  - Extraction, QuEChERS
  - SPE, dispersive SPE
- Evaluation of Extraction
  - Data from Initial Studies
  - Insights and observations
- Optimization Sample Preparation Techniques
  - Custom dispersive SPE
  - EMR-Lipid dispersive SPE
- Future Investigations, Aspects and Considerations

Cannabis and Cannabis-Based Products: Pesticide Analysis

No tolerances have been established for marijuana, because of its illegal federal status and because the pesticide companies have yet to embark on the lengthy and expensive process of testing their products on cannabis……*Cannabis Now Issue 19*

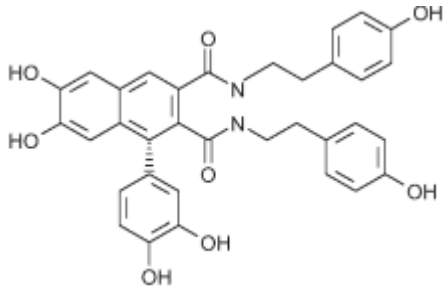
Pesticide Analytes and their action levels in Oregon

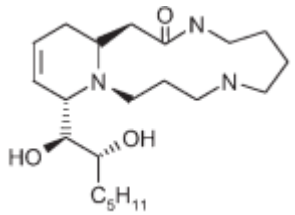
Analyte	Chemical Abstract Services (CAS) Registry Number	Action Level ppm
Abamectin	71751-41-2	0.5
Acephate	30560-19-1	0.4
Acequinocyl	57960-19-7	2
Acetamiprid	135410-20-7	0.2
Aldicarb	116-06-3	0.4
Azoxystrobin	131860-33-8	0.2
Bifenazate	149877-41-8	0.2
Bifenthrin	82657-04-3	0.2
Boscalid	188425-85-6	0.4
Carbaryl	63-25-2	0.2
Carbofuran	1563-66-2	0.2
Chlorantraniliprole	500008-45-7	0.2
Chlorfenapyr	122453-73-0	1
Chlorpyrifos	2921-88-2	0.2
Clofentezine	74115-24-5	0.2
Cyfluthrin	68359-37-5	1
Cypermethrin	52315-07-8	1
Daminozide	1596-84-5	1
DDVP (Dichlorvos)	62-73-7	0.1
Diazinon	333-41-5	0.2
Dimethoate	60-51-5	0.2
Ethoprophos	13194-48-4	0.2
Etofenprox	80844-07-1	0.4
Etoxazole	153233-91-1	0.2
Fenoxycarb	72490-01-8	0.2
Fenpyroximate	134098-61-6	0.4
Fipronil	120068-37-3	0.4
Flonicamid	158062-67-0	1
Fludioxonil	131341-86-1	0.4
Hexythiazox	78587-05-0	1
Imazalil	35554-44-0	0.2
Imidacloprid	138261-41-3	0.4
Kresoxim-methyl	143390-89-0	0.4
Malathion	121-75-5	0.2
Metalaxyl	57837-19-1	0.2
Methiocarb	2032-65-7	0.2
Methomyl	16752-77-5	0.4
Methyl parathion	298-00-0	0.2

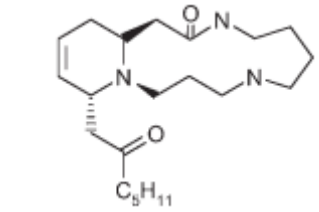
Analyte	Chemical Abstract Services (CAS) Registry Number	Action Level ppm
MGK-264	113-48-4	0.2
Myclobutanil	88671-89-0	0.2
Naled	300-76-5	0.5
Oxamyl	23135-22-0	1
Paclobutrazol	76738-62-0	0.4
Permethrins[16]	52645-53-1	0.2
Phosmet	732-11-6	0.2
Piperonyl_butoxide	51-03-6	2
Prallethrin	23031-36-9	0.2
Propiconazole	60207-90-1	0.4
Propoxur	114-26-1	0.2
Pyrethrins[17]	8003-34-7	1
Pyridaben	96489-71-3	0.2
Spinosad	168316-95-8	0.2
Spiromesifen	283594-90-1	0.2
Spirotetramat	203313-25-1	0.2
Spiroxamine	118134-30-8	0.4
Tebuconazole	80443-41-0	0.4
Thiacloprid	111988-49-9	0.2
Thiamethoxam	153719-23-4	0.2
Trifloxystrobin	141517-21-7	0.2

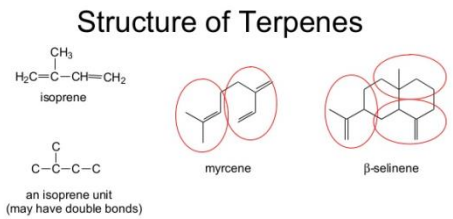
Constituents of Cannabis Plants: Complex

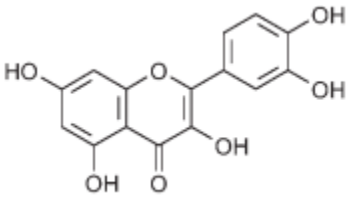
- **Nitrogenous compounds (27 known)**
- Amino acids (18),
- Proteins (3)
- Glycoproteins (6)
- Enzymes (2)
- **Sugars and related compounds (34)**
- Hydrocarbons (50)
- Simple alcohols (7)
- Aldehydes (13)
- Ketones (13)
- Simple acids (21)
- **Fatty acids (22)**
- Simple esters (12)
- Lactones (1)
- Steroids (11)
- **Terpenes (120)**
- **Non-cannabinoid phenols (25)**
- **Cannabinoids (66)**
- **Flavonoids (21)**
- Vitamins (1) [Vitamin A]
- Pigments (2)
- Elements (9).

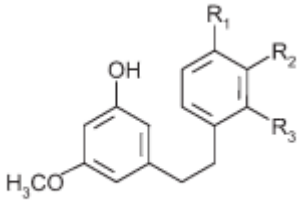




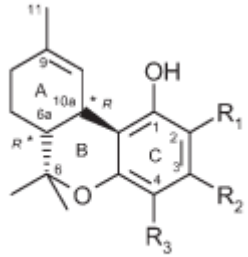








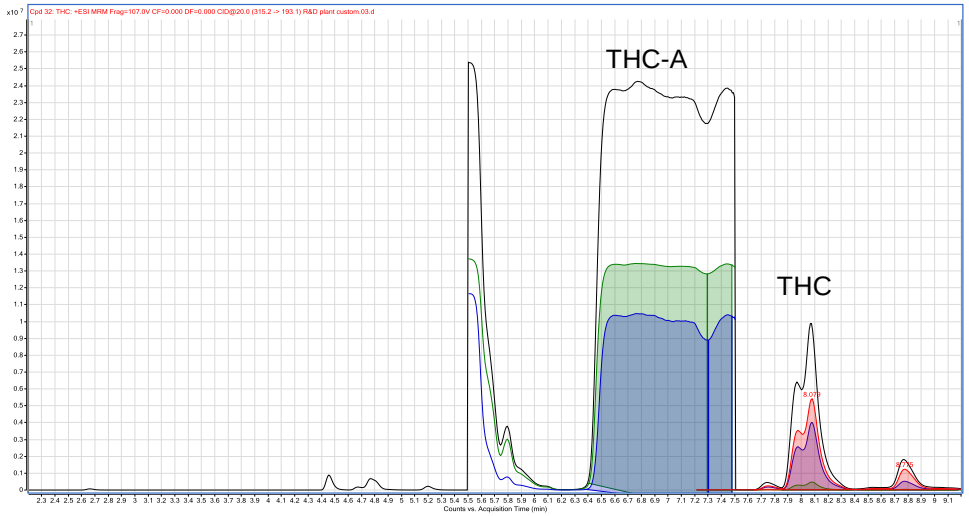
R_1 = OH, R_2 = isoprenyl, R_3 = H



R_1 = COOH, R_2 = C_5H_{11}, R_3 = H

Sample Preparation: Pesticide Analysis

- Complex matrix associated with cannabis plant material and edibles needs to be addressed
  - CBDs are in large amounts (10-20%), THCA can interfere with analysis with broad interference (100,000-200,000 ppm)
  - Terpenes and other non-cannabinoid compounds are also in large ppm quantities (10-5000 ppm)
  - Pesticides in 500 ppb amounts (0.00005%)



Sample Preparation Techniques: Pesticide Analysis

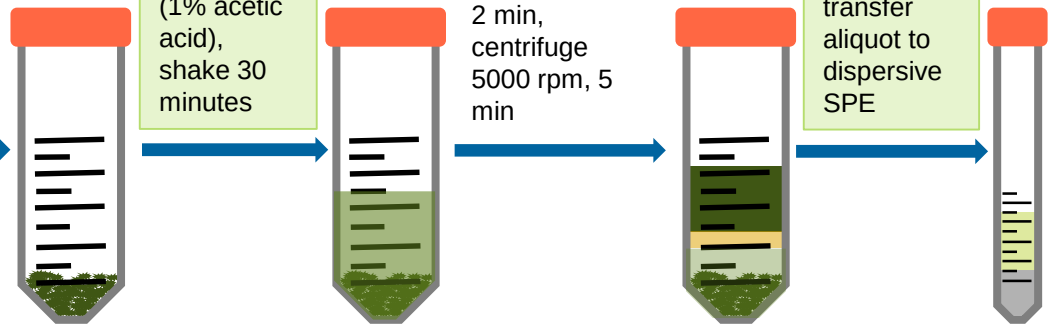
- Methanol extraction with C18 SPE
- Acetonitrile extraction with C18 SPE
- Acetonitrile extraction with both NH2 and C18 SPE
- QuEChERS extraction with SPE cleanup
- QuEChERS extraction with dispersive SPE cleanup
  - Plant material and edibles considered dry matrix; < 60% water
  - Addition of water required for QuEChERS extraction/partitioning: step 1
  - Use of Salts: Na Acetate, Citrates, NaCl with MgSO4
  - Super-saturates the water with the salts allowing separation of water from the ACN
  - Analytes of interest extract/transfer into the ACN layer
  - Clean-up of co-extractive matrix: step 2

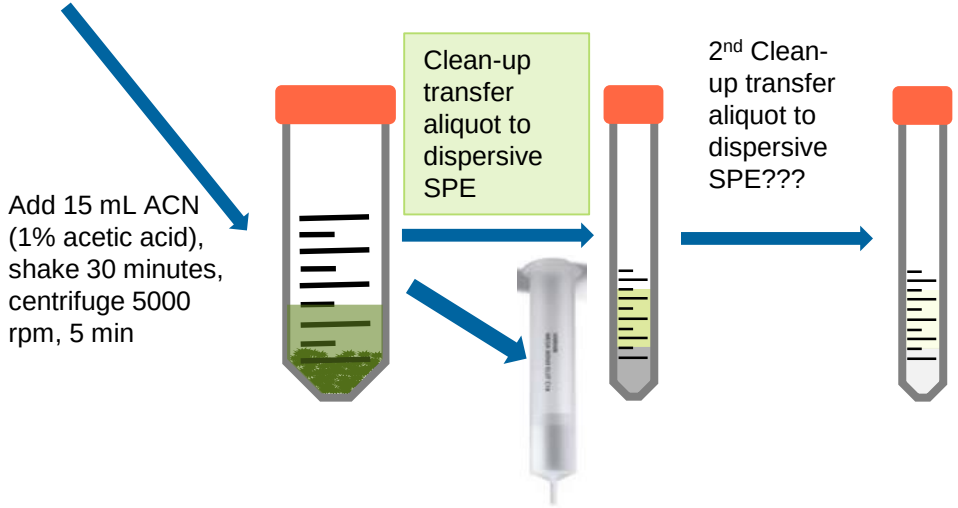
Basic Protocol: Pesticide Analysis

Grind flower or plant material (weigh1.5 g)
SPEX GenoGrinder, 2 Ceramic Homogenizers, 5 min at 1500 rpm

Individual grinding in 50 mL disposable centrifuge tubes eliminates washing, carryover and cross contamination

2010 Geno/Grinder

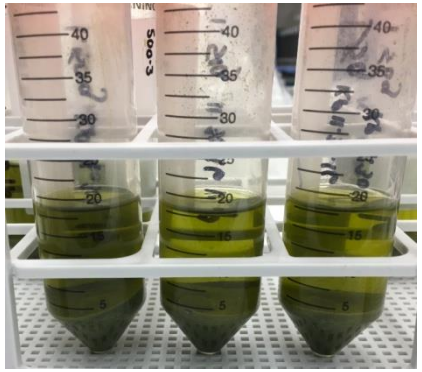


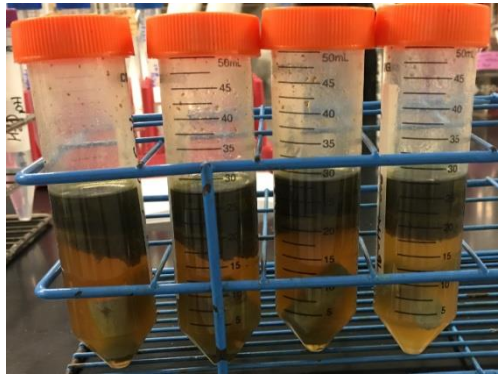


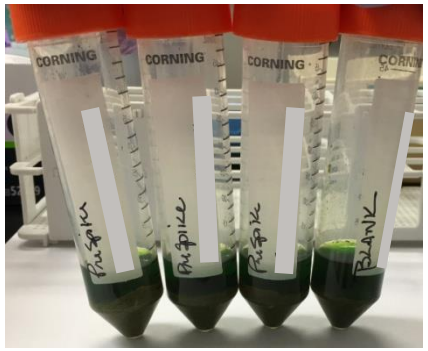
Dispersive SPE: Universal or All Food Types:
(2 mL) 50 mg PSA, 50 mg C18, 7.5 mg GCB, 150 mg MgSO4 (5982-0028)
(15 mL) 400mg PSA, 400 mg C18, 45 mg GCB, 1200 MgSO4 (5982-0029)

Optimization and Custom Dispersive SPE: Why is it Important

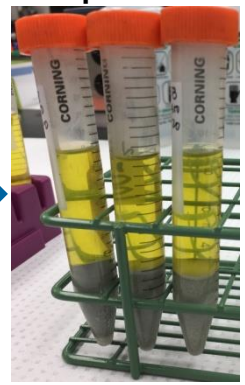
Step 1:

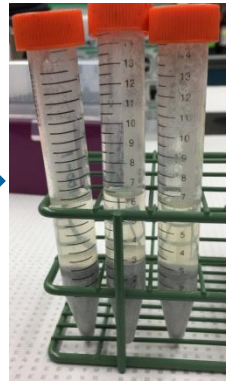






Step 2:





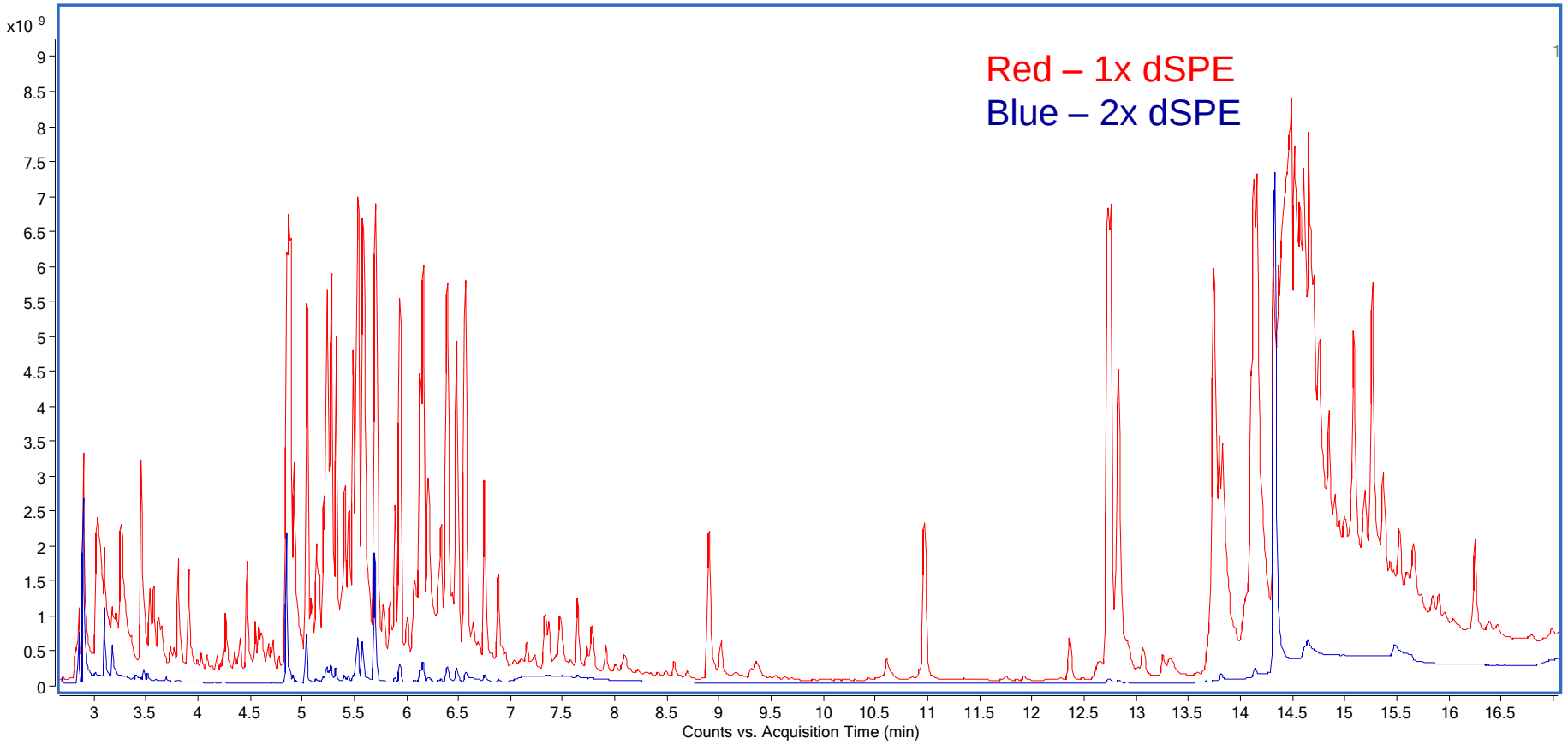
QuEChERS Extraction/Partitioning
or
ACN (1% acetic acid) extraction

- Advantages or Disadvantages
- Addition of water
- Addition of 1% acetic acid

Clean-up: Dispersive SPE

- Advantage over SPE, less steps, no manifold, vacuum
- Enhanced (custom) clean-up or additional clean-up

Cannabis mixture cleanup – QuEChERS with 1x dSPE vs. 2 x dSPE custom dispersive steps

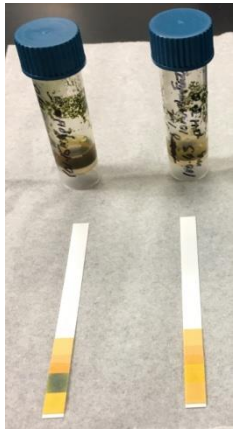


GC MS 7010 Triple Quad TIC Scan, 0.5 uL injection, Gain 10
Custom dispersive: Mixture of PSA, C18-EC, GCB, additional sorbent, and MgSO4

Sample Preparation: Insights and Observations

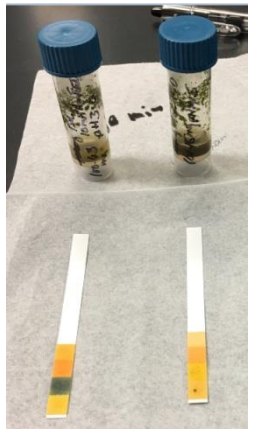
Step 1: QuEChERS or ACN Extraction

- Addition of Water, vortex 15-30 minutes



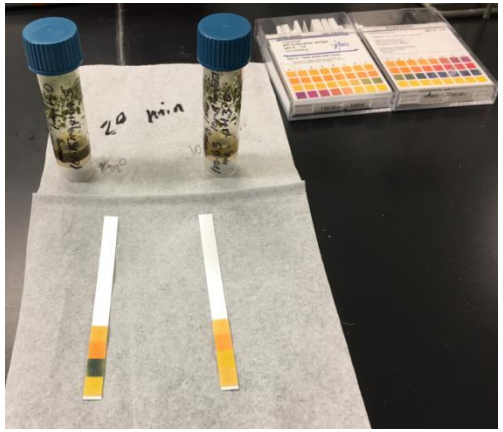
5 min

Water

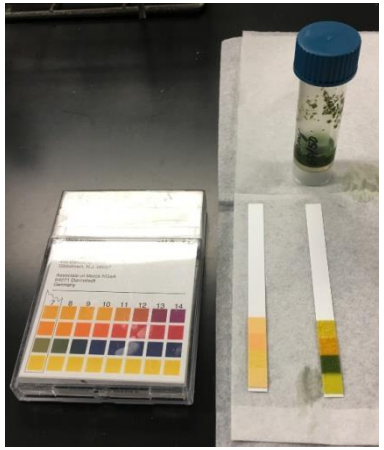


10 min

50 mM Na Acetate pH 3.5



20 min with indicator pH range

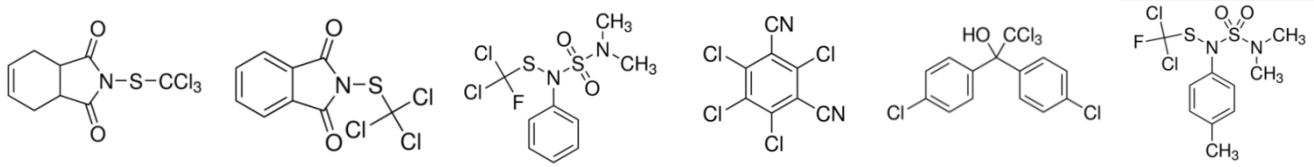


5 min with indicator pH range

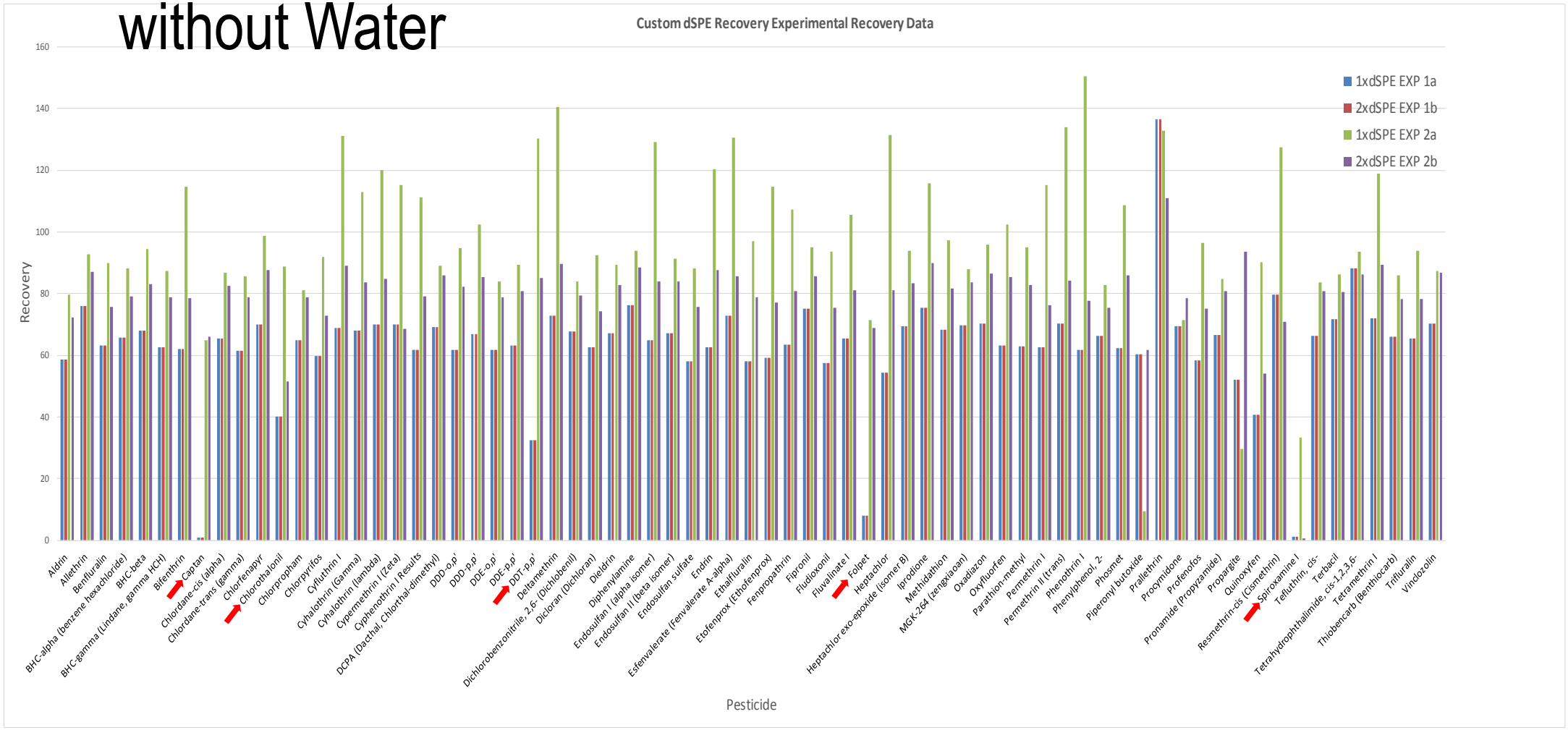
Water/ACN (1:1 v/v)

Addition of water to cannabis flower/plant turns basic almost immediately

Why is this a concern……degradation of base sensitive pesticides (fungicides)
Captan, Folpet, Dichlofluanid, Chlorothalonil, Dicofol, Tolylfluanid



Recovery Comparison using Custom dSPE with and without Water



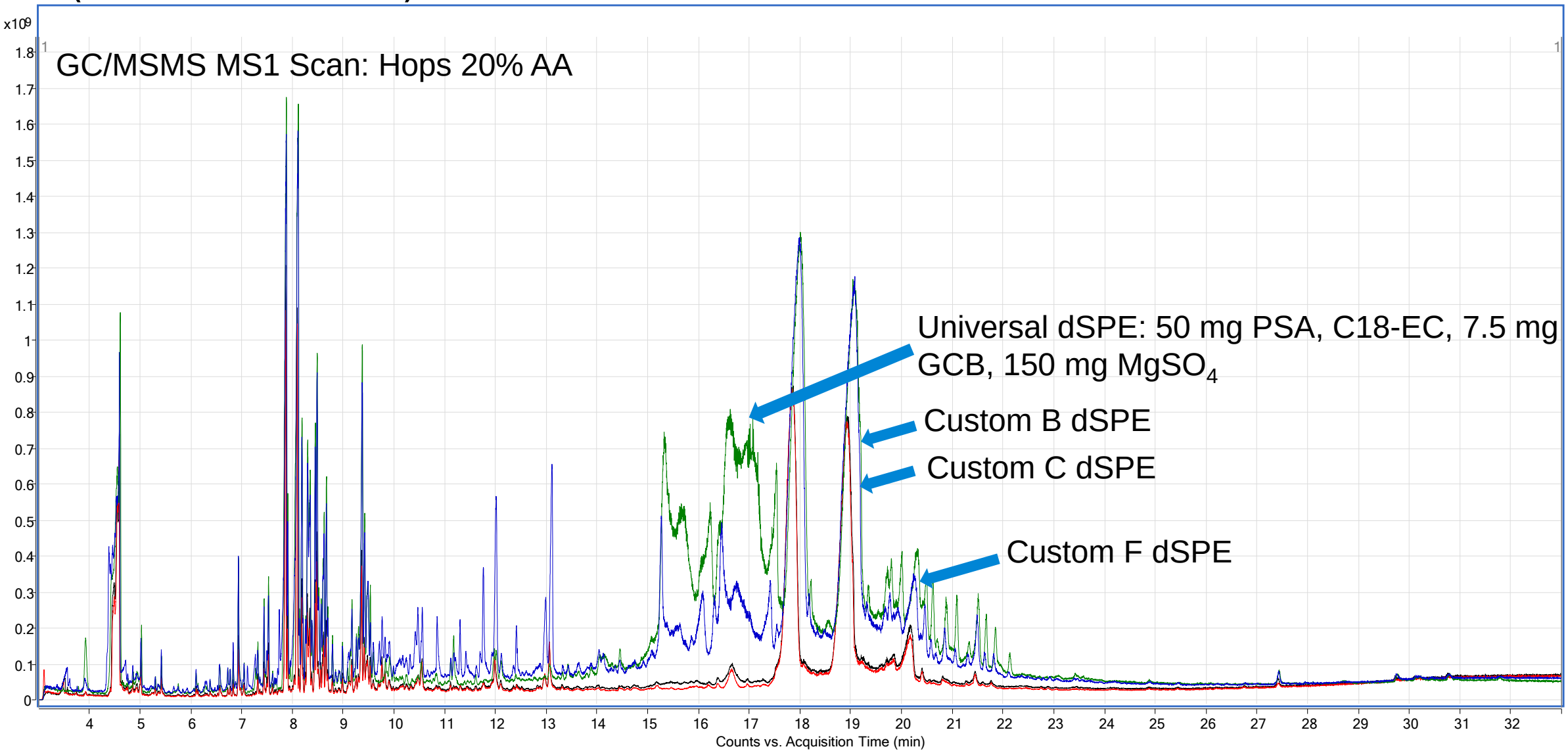
EXP 1a: AOAC QuEChERS: ACN (1%AA) 13.75 mL, dSPE custom, 1xdSPE

EXP 1b: AOAC QuEChERS: ACN (1%AA) 13.75 mL, dSPE custom, 2xdSPE

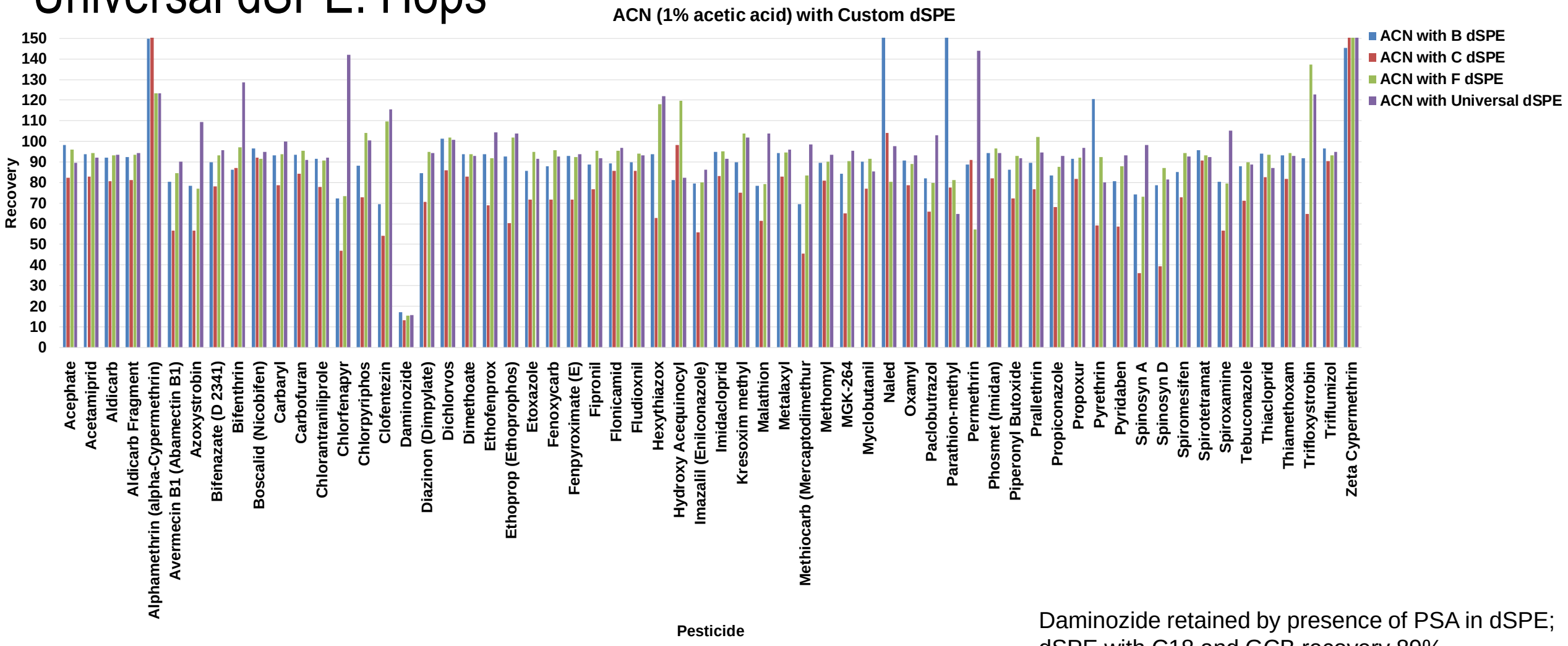
EXP 2a: ACN (1%AA) 13.75 mL only no QuEChERS salts and no H2O, dSPE custom, 1xdSPE

EXP 2b: ACN (1%AA) 13.75 mL only no QuEChERS salts and no H2O, dSPE custom, 2xdSPE

ACN (1% Acetic Acid) Extraction with Custom dSPE versus Universal dSPE



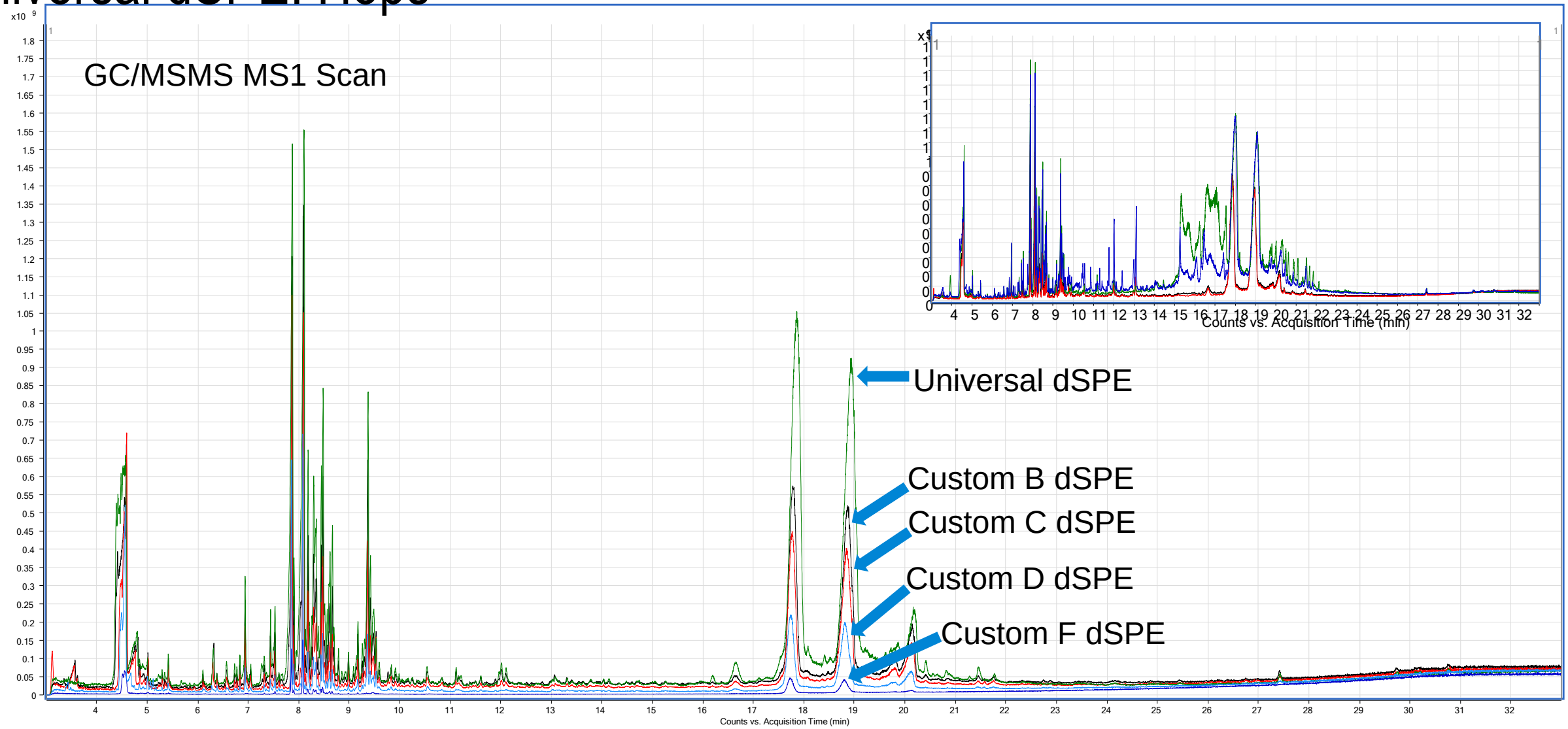
Pesticide Recovery after ACN (1% acetic acid) with Custom dSPE or Universal dSPE: Hops



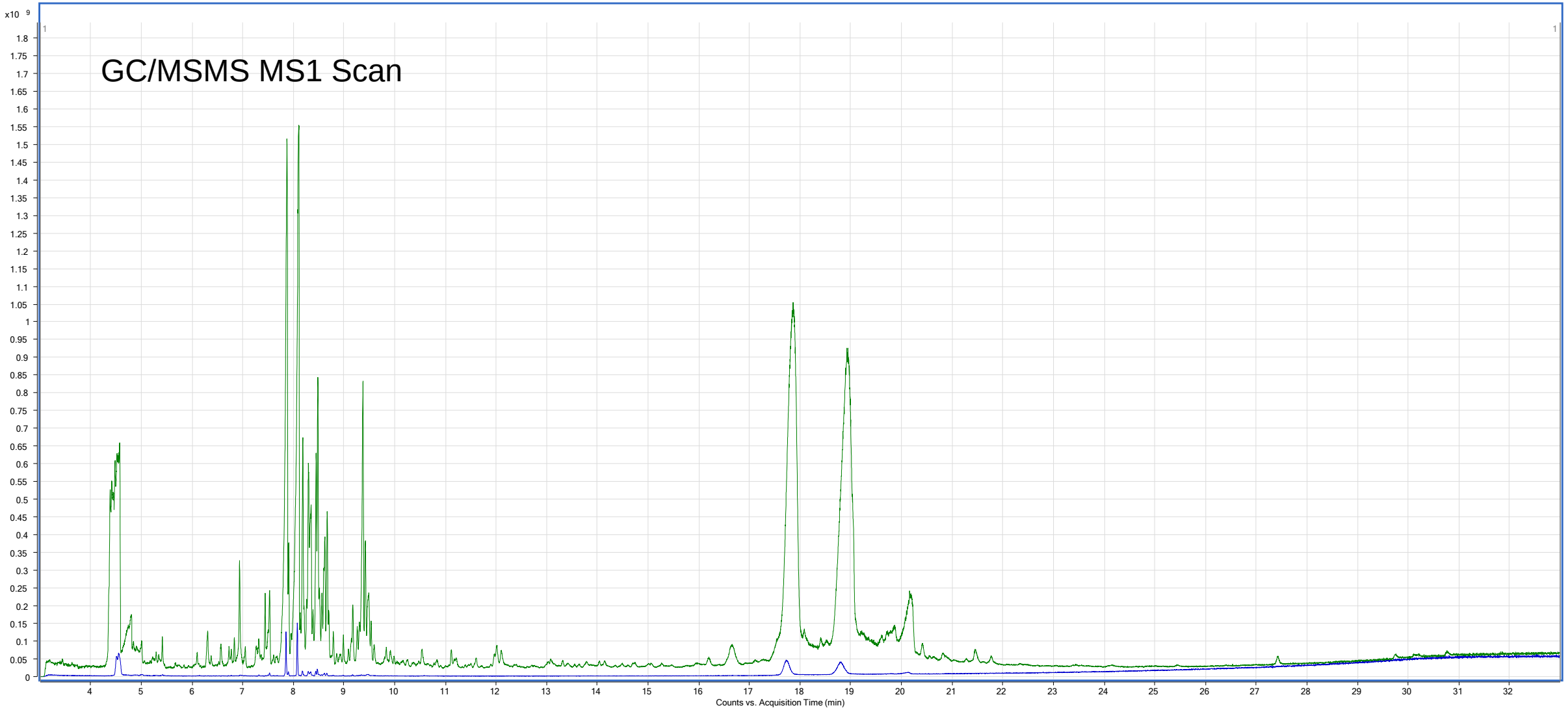
Daminozide retained by presence of PSA in dSPE; dSPE with C18 and GCB recovery 89%

Concentration of spiked pesticides: 100 ppb

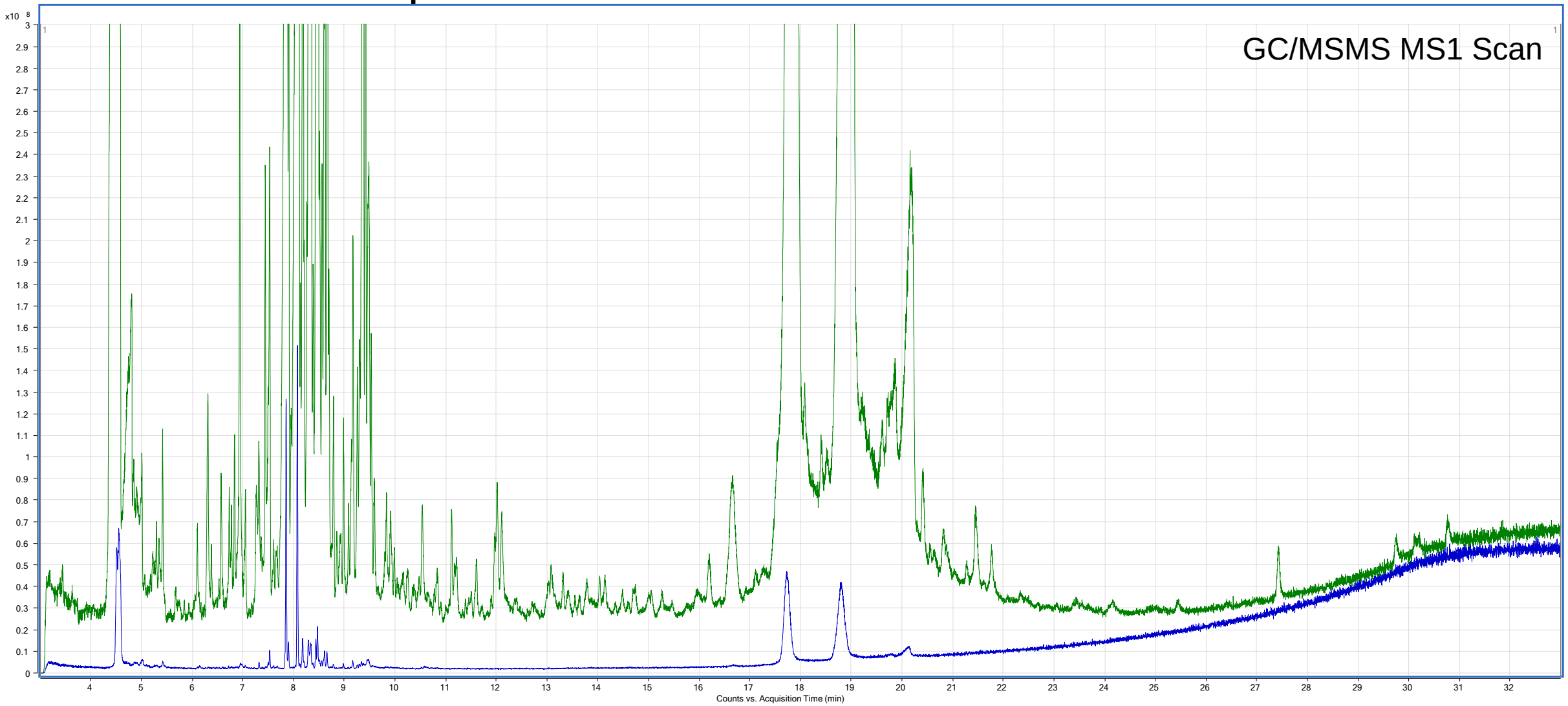
QuEChERS AOAC Extraction/Partitioning with Custom dSPE versus Universal dSPE: Hops



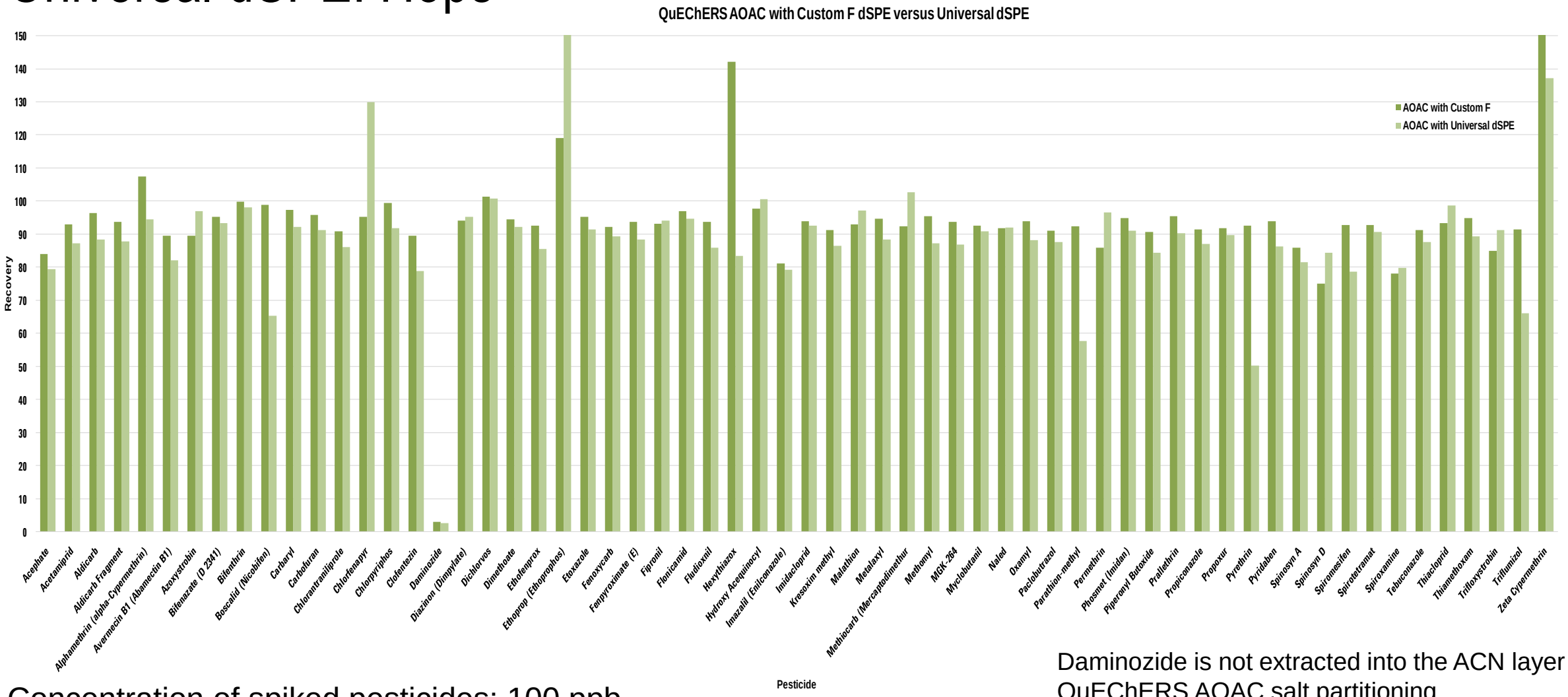
QuEChERS AOAC Extraction/Partitioning with Custom F dSPE versus Universal dSPE: Hops



QuEChERS AOAC Extraction/Partitioning with Custom F dSPE versus Universal dSPE: Hops



Pesticide Recovery after QuEChERS AOAC with Custom F dSPE or Universal dSPE: Hops

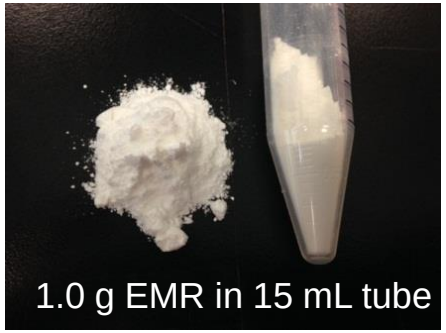


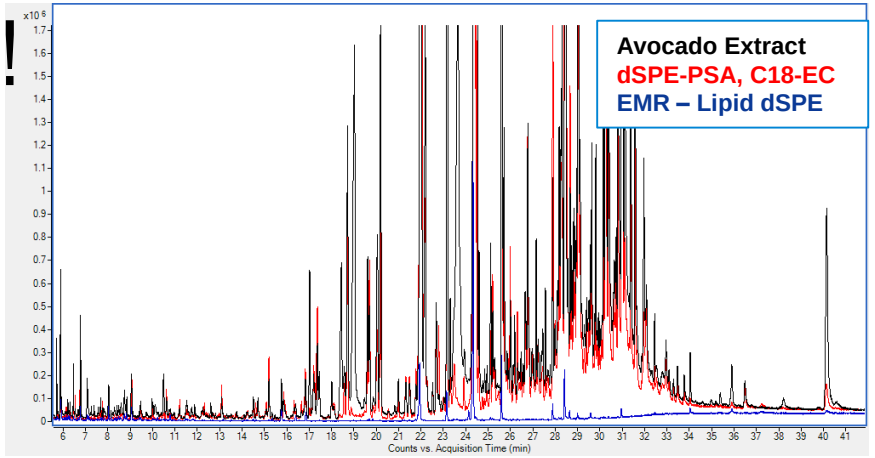
Concentration of spiked pesticides: 100 ppb

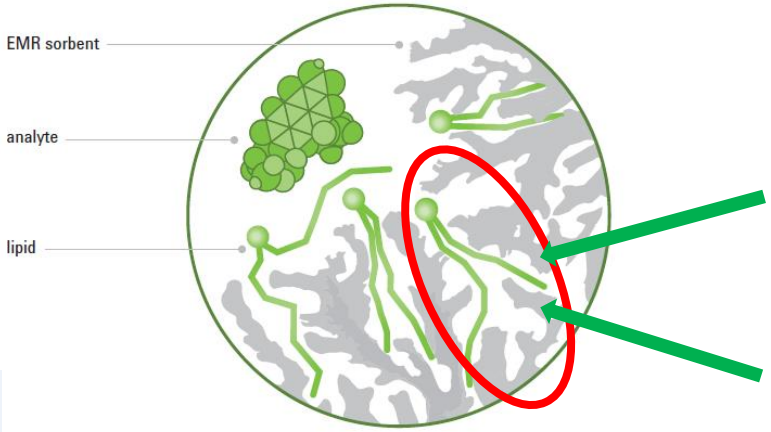
Daminozide is not extracted into the ACN layer after QuEChERS AOAC salt partitioning

Cannabis-Based Products: Unique Dispersive SPE

EMR-Lipid Innovative Sorbent!







When "activated" by water EMR-Lipid Sorbent Selectively traps lipids.

Size Exclusion: Unbranched hydrocarbon chains (lipids) enter the sorbent; bulky analytes do not.

Sorbent Chemistry: Lipid chains that enter the sorbent are trapped by hydrophobic interactions.

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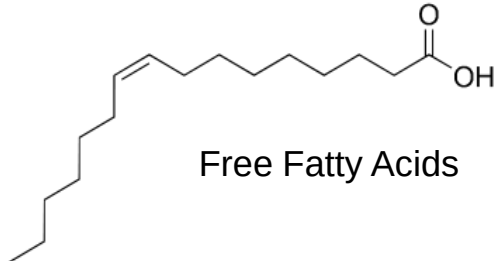
EMR-Lipid Mechanism – Size exclusion and hydrophobic interaction

… and what does it do?

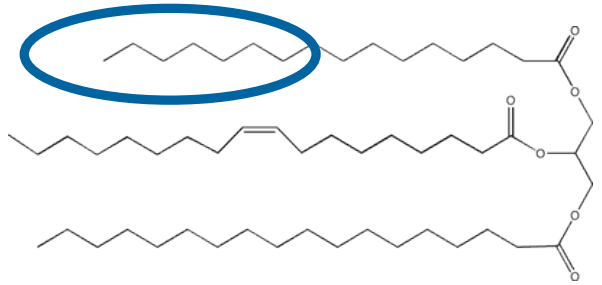
EMR sorbent removes LIPIDS it interacts with the long aliphatic chain NOT the functional groups

What are Lipids?

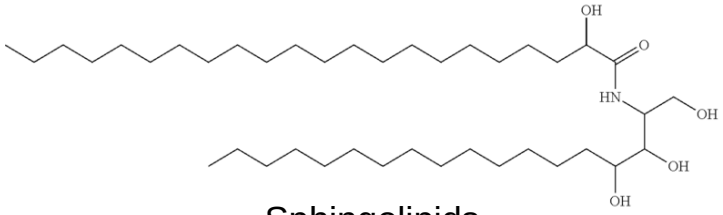
A class of naturally occurring hydrocarbon containing compounds commonly known as fats and oils



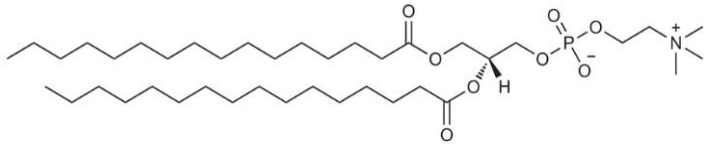
Free Fatty Acids



Triglycerides



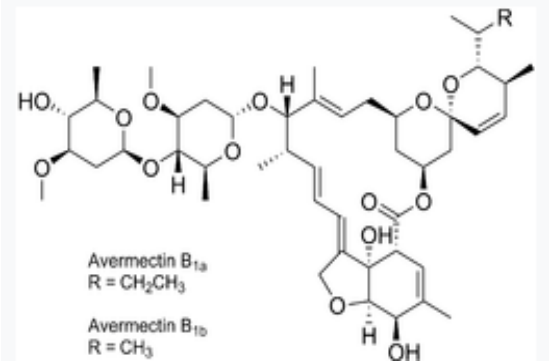
Sphingolipids



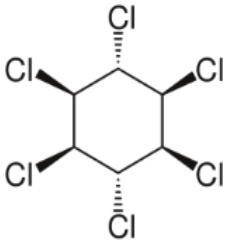
Phospholipids

What Does EMR *NOT* Interact With?

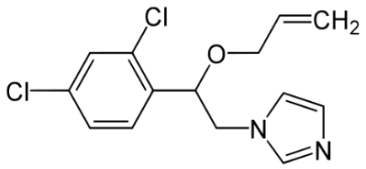
EMR does NOT remove analytes of interest



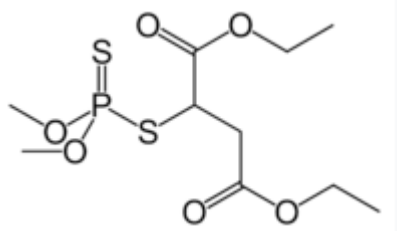
Insecticides and anthelmintics



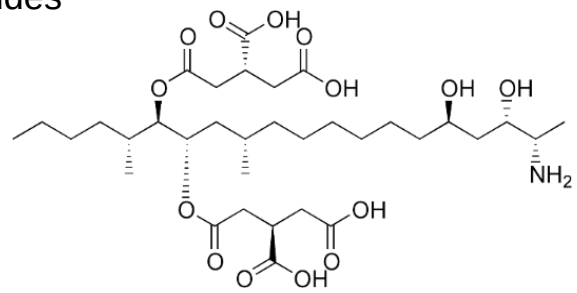
Organochlorine Pesticides



Imidazole pesticides



Organophosphate Pesticides

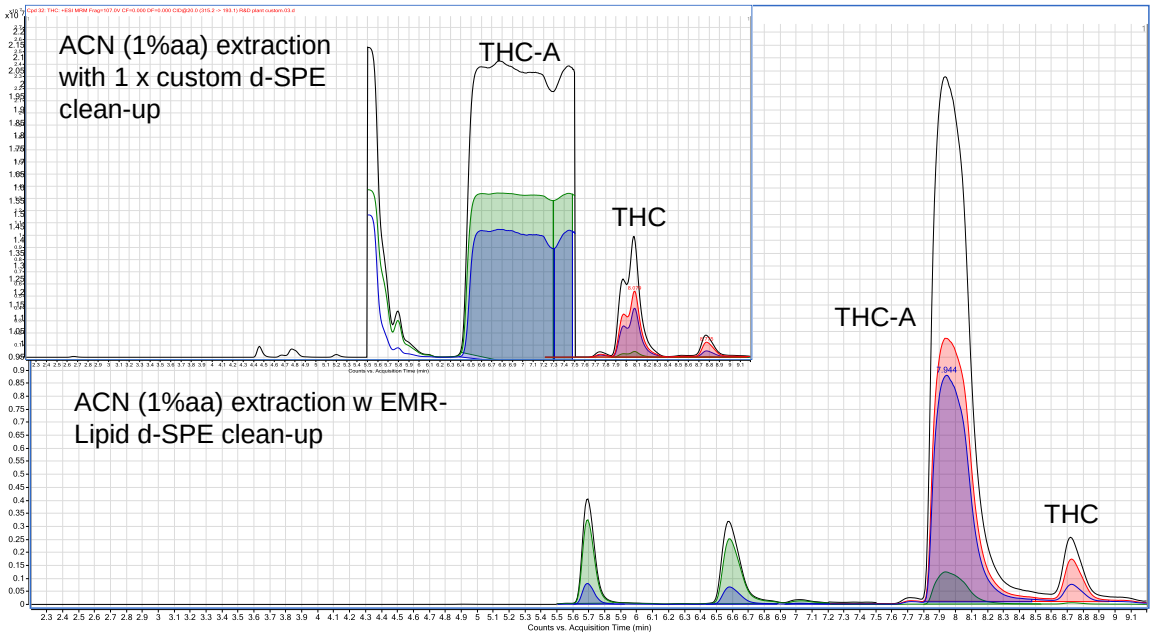


Fumonisin B2

Aflatoxins and Mycotoxins

Cannabis-Based Product: THC-Butter

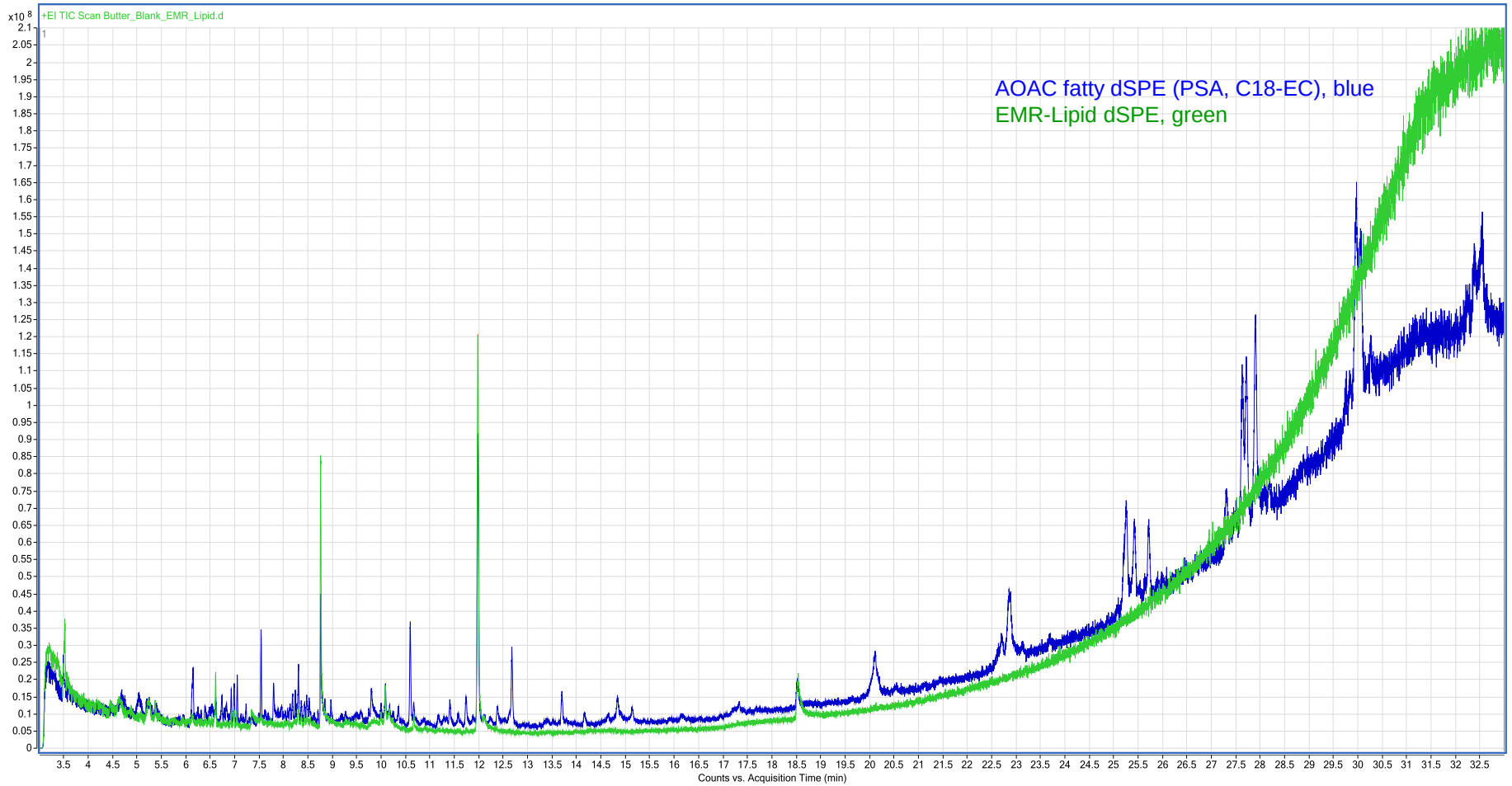
- 1.5 g of THC-butter was added to a 50 mL centrifuge tube
- Add 15 mL of ACN (1%acetic acid)
- Vortex 30 minutes, centrifuge 5000 rpm, 5 min
- Add 5 mL water to EMR-Lipid, vortex immediately 1 min
- Add 5 mL of ACN (1% acetic acid) extract, vortex immediately 2 min
- Centrifuge 5000 rpm, 5 min
- Analyze by LCMSMS



Pesticide Screened:

Abamectin B1-a
Abamectin B1-b
Azoxystrobin
Bifenazate
Etoxazole
Imazalil
Imidacloprid
Malathion
Myclobutanil
Permethrin-1
Permethrin-2
Spinosyn A
Spinosyn D
Spiromesifen
Spirotetramat
Tebuconazole

QuEChERS extraction of Butter: MS1 Scan



Blue Chromatogram: 1.5 g melted butter, AOAC salts, AOAC fatty dSPE (PSA and C18EC)
Green Chromatogram: 1.5 g melted butter, AOAC salts, EMR-Lipid dispersive SPE

Insights and Observations :

- If QuEChERS AOAC method is being used you should add the ACN (1% acetic acid immediately after the addition of water, before vortexing for 20-30 minutes
- Do not use QuEChERS EN method, water and ACN mix will be too basic for basic labile compounds
- Promising results with ACN (1% acetic acid) extraction, no QuEChERS salts
- Dispersive SPE or SPE is required to remove some of the matrix co-extractives
- The active ingredients can cause interference issue: cannabinoids
- Is the existing clean-up techniques enough for long term analysis, issues with analysis and instrument maintenance

Future Investigations and Considerations

- Continue to investigate the extraction approach: QuEChERS versus Acetonitrile extraction
- Method Development with custom d-SPE
  - Focus on improving matrix removal
  - One approach for both LC and GC MSMS
  - Simplicity and ease of use
  - Advance work with EMR-Lipid with cannabinoid products with high lipid content: food, butter and oils
- Reducing sample size
  - Miniaturization of extraction
    - Experience with approach, several application notes and presentations
    - Availability of material, cannabis and cannabis-based products
    - Substantial cost saving ~45% per sample

Agilent GC-MS/MS Cannabis Testing

Pesticide Analysis in Cannabis by GC - QQQ

Ronald Honnold and Melissa Churley

GC-MS Application Scientists

Pesticide Analysis by GC/MS/MS

Summary:

"The Oregon limits in Table 1 (next slide) are not thresholds;
they are a best guess at the analytical LOQ for that analyte.

These limits are analytical in nature only and will probably be revised when Oregon has enough data to be sure the labs can achieve lower limits".

Pesticides included in the analysis are highlighted.

Guidance for State Medical Cannabis Testing Programs, Association of Public Health Laboratories (APHL), Silver Spring, MS; www.aphl.org, May 2016

Pesticide Analysis by GC/MS/MS

Table 1: Pesticide analytes and their action levels in OR (excerpt)

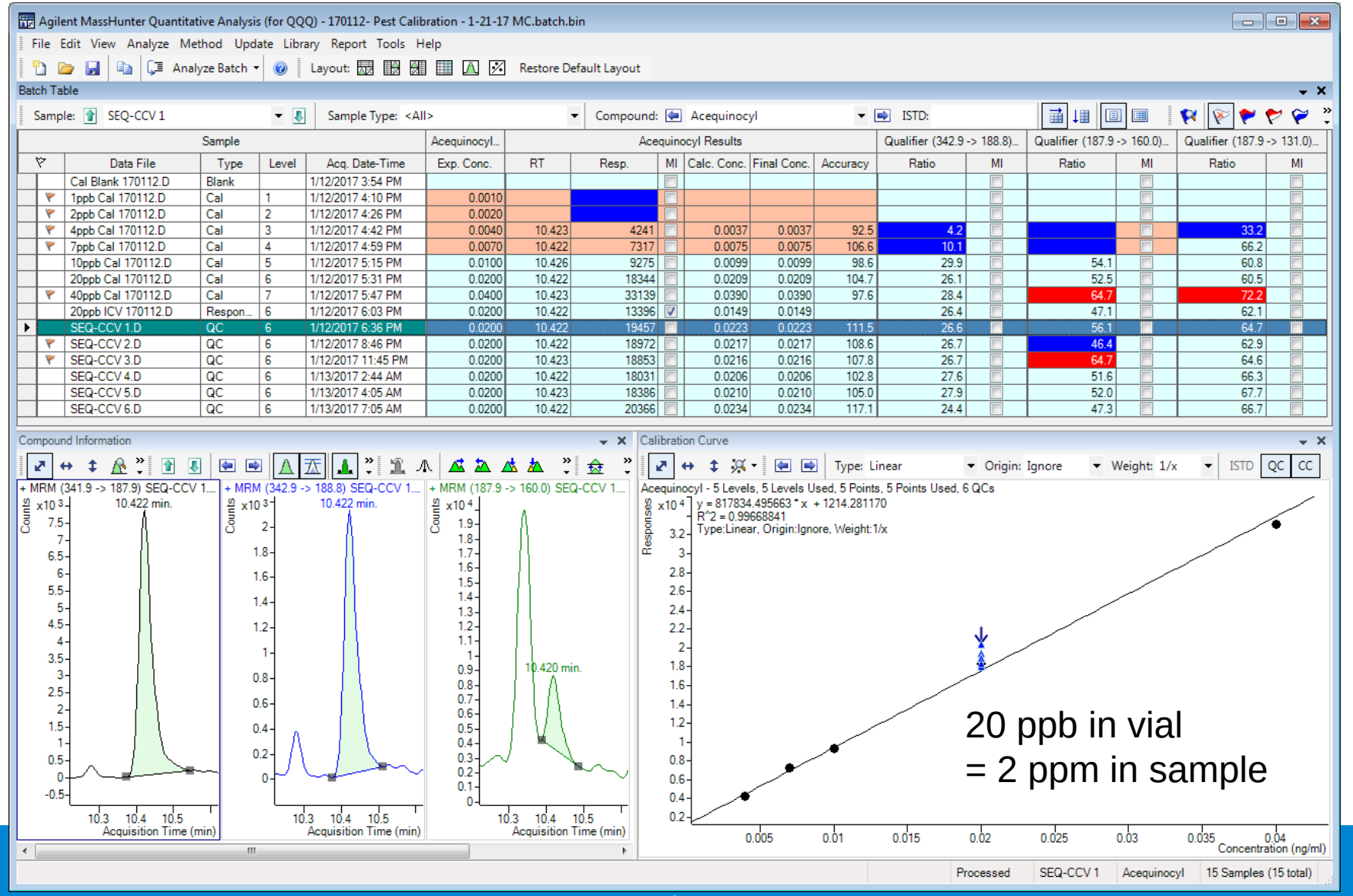
Analyte	Chemical Abstract Services (CAS) Registry Number	Action Level ppm
Abamectin	71751-41-2	0.5
Acephate	30560-19-1	0.4
Acequinocyl	57960-19-7	2
Acetamiprid	135410-20-7	0.2
Aldicarb	116-06-3	0.4
Azoxystrobin	131860-33-8	0.2
Bifenazate	149877-41-8	0.2
Bifenthrin	82657-04-3	0.2
Boscalid	188425-85-6	0.4
Carbaryl	63-25-2	0.2
Carbofuran	1563-66-2	0.2
Chlorantraniliprole	500008-45-7	0.2
Chlorfenapyr	122453-73-0	1
Chlorpyrifos	2921-88-2	0.2
Clofentezine	74115-24-5	0.2
Cyfluthrin	68359-37-5	1
Cypermethrin	52315-07-8	1
Daminozide	1596-84-5	1
DDVP (Dichlorvos)	62-73-7	0.1

Highest

Lowest

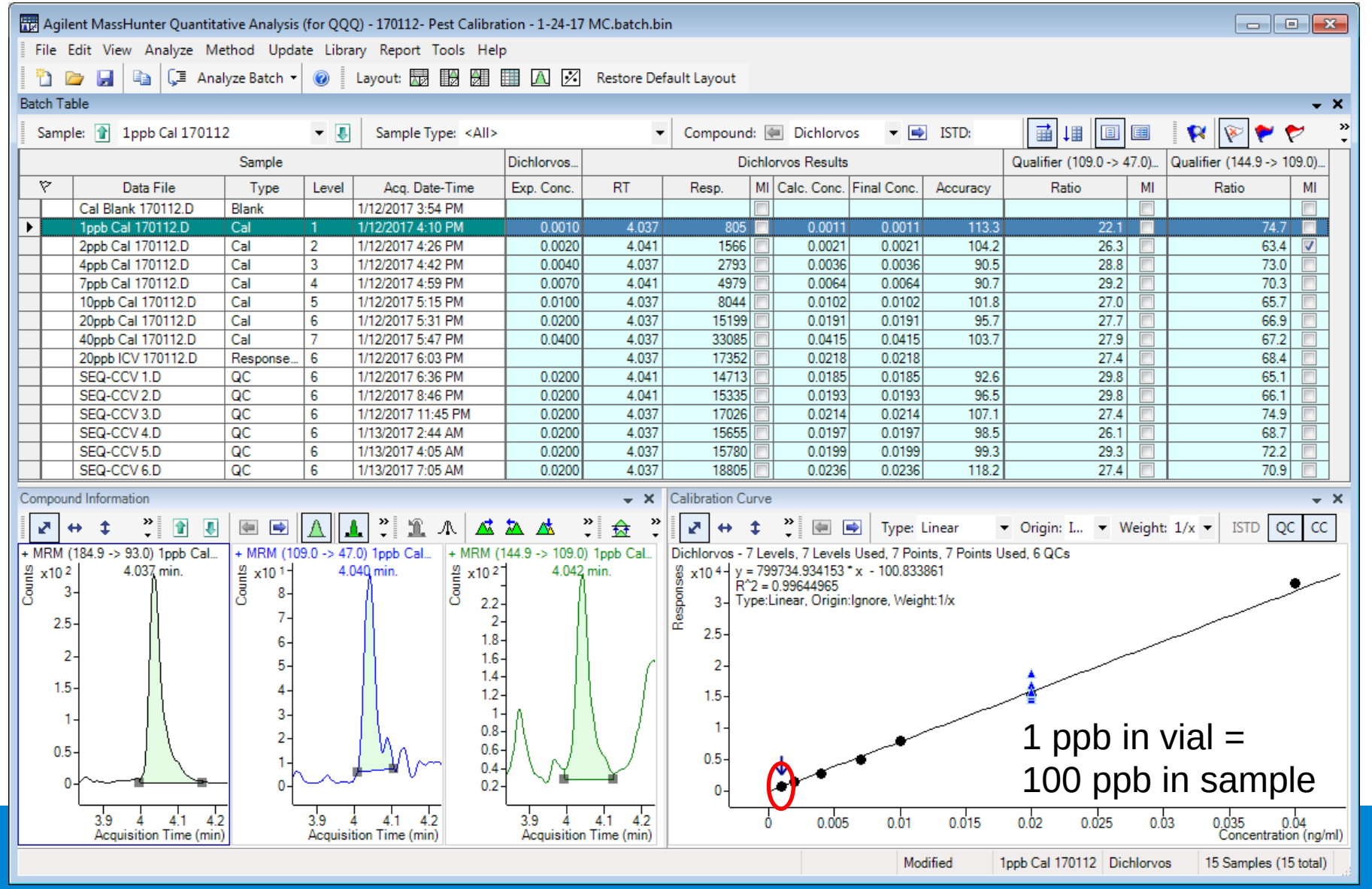
Pesticide Analysis by GC/MS/MS

Calibrators and QCs for Acequinocyl (Action Level 2 ppm in OR)



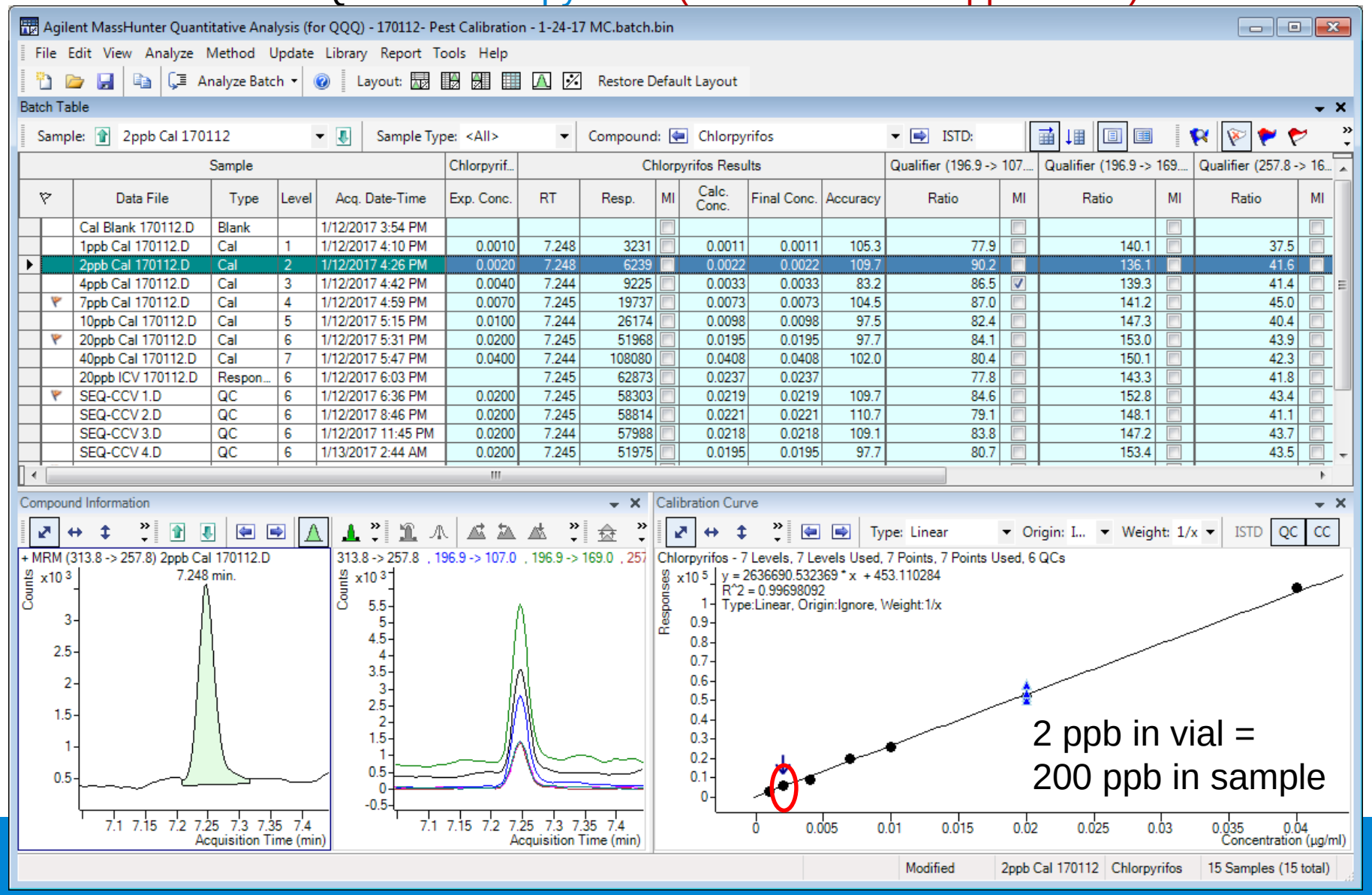
Pesticide Analysis by GC/MS/MS

Calibrators and QCs for Dichlorvos (Action Level 100 ppb in OR)



Pesticide Analysis by GC/MS/MS

Calibrators and QCs for Chlorpyrifos (Action Level 200 ppb in OR)



Pesticide Analysis by GC/MS/MS

%RSD (n=6) for 14 GCMS-amenable pesticides

Pesticide (GCMS)	%RSD (20 ppb in vial)	Action Level (ppb) Oregon
Dichlorvos	8.0	100
Naled	12.5	500
Parathion-methyl	6.2	200
Chlorpyrifos	6.7	200
MGK-264	4.9	200
Fipronil	4.9	400
Fludioxonil	6.6	400
Kresoxim-methyl	4.5	400
Chlorfenapyr	6.2	1000
Propiconazole	3.9	400
Bifenthrin	2.9	200
Acequinocyl	4.6	2000
Cyfluthrin	5.2	1000
Cypermethrin	4.0	1000

Lowest AL

Highest AL

Pesticides & Environmental Pollutants v4.0

Pesticides Monitoring in Food and Environmental

Complete Analytical System to run Application

- 7890B GC and 7000D/7010B MS
- Acquisition method, comprehensive user guide, and Instrument consumables

P&EP 4.0 Enhanced MRM database with 3-yr Subscription

- >1,100 compounds with over 7,500 matrix-optimized MRM Transitions
- Works seamlessly with dMRM method builder in MassHunter

Added Value Items

- Optional 4-Day On-site Method and Application Services (R3997A)
- Bond Elut QuEChERS: e.g., Enhanced Matrix Removal-Lipid Kit (for high lipid content)
- 20% discount for customized standard from ULTRA SCIENTIFIC

Reference Guide for GC/MS/MS Pesticide Residue Analysis

Reference Guide

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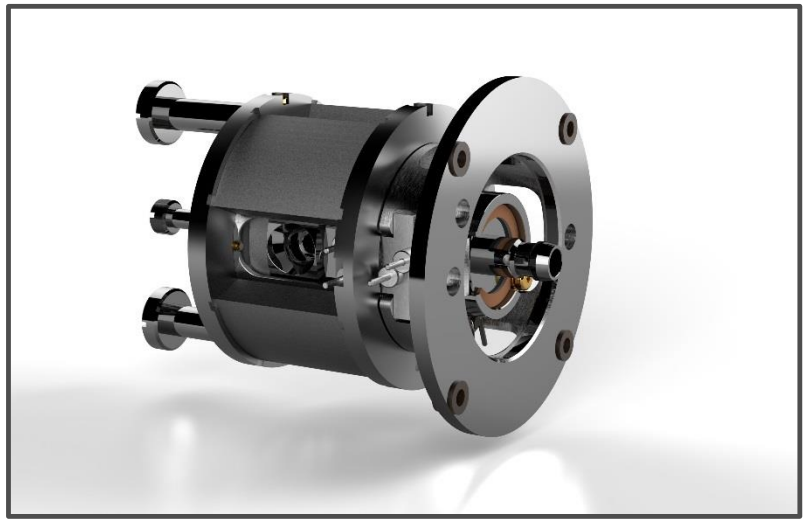
7010B Triple Quadrupole GC/MS System

- Also available: 7010B Triple Quadrupole GC/MS System
- Features the Agilent High Efficiency EI Source (HES) - the greatest advance in electron ionization in decades
- Produces up to 20X more ions so you can…
  - Reach lower detection limits, or
  - Increase your lab's efficiency by:
    - Injecting less to extend the life of your liners and columns
    - Eliminating preconcentration steps
    - Scaling down all of your sampling and sample prep procedures to save money on transportation, reagents, and waste



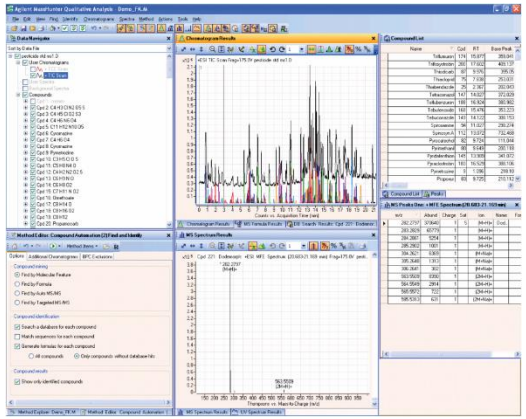
Agilent LC-MS/MS Cannabis Testing

Pesticide Analysis in Cannabis by HPLC - QQQ

Sue D’Antonio

LCMS Application Chemist

Agilent LC-QQQ for Every State, Every Detection Level



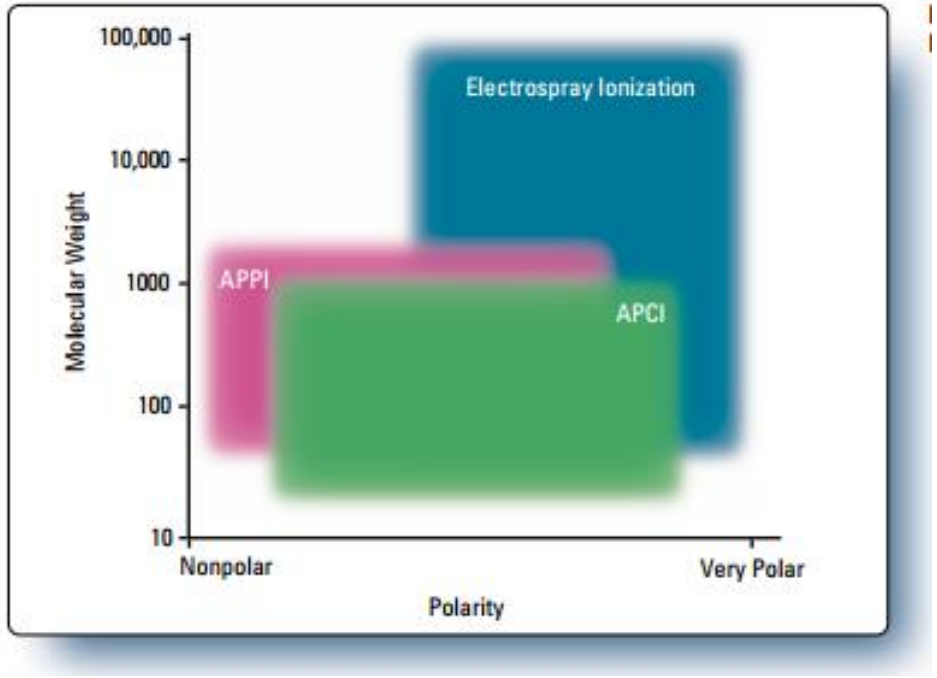
Infinity LC Series & MassHunter Software

6495B LC - QQQ

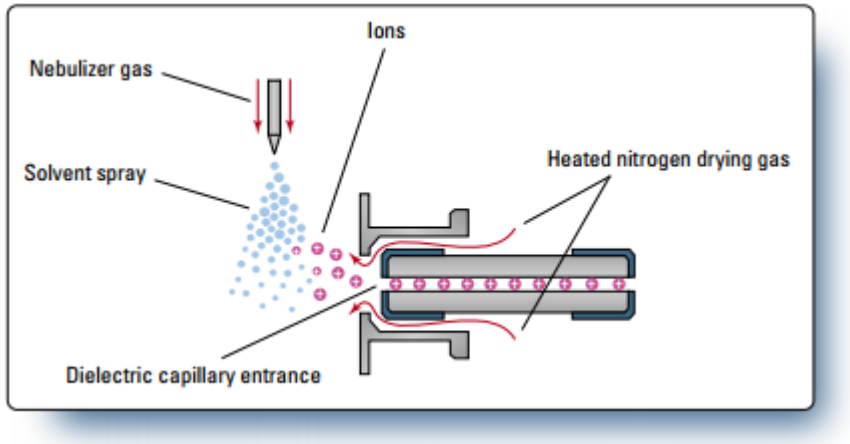
6420 LC-QQQ

6470 LC-QQQ

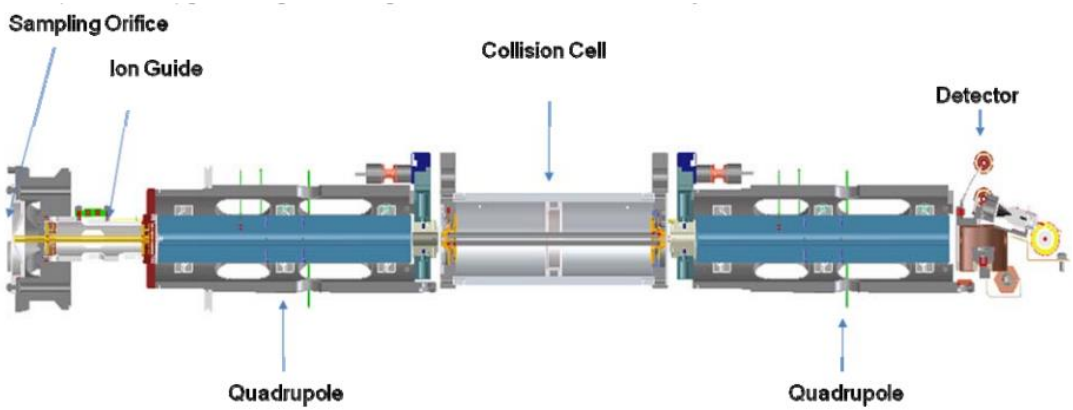
Triple Quadrupole Instruments



Ion Sources



Electrospray Ionization

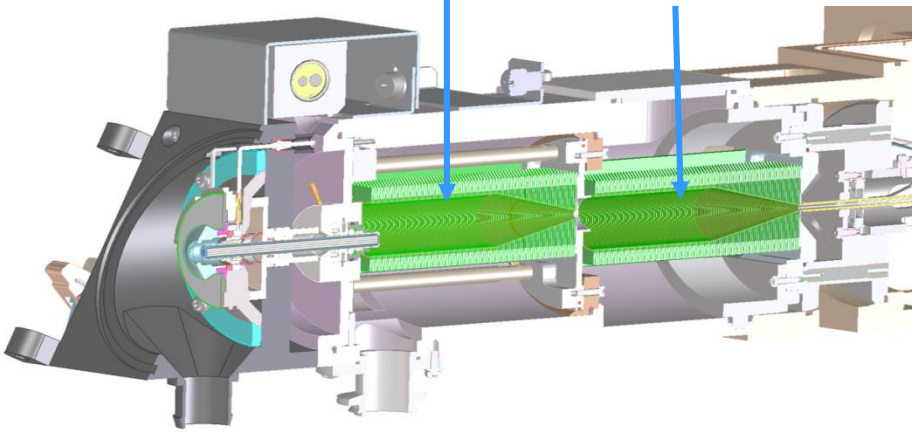


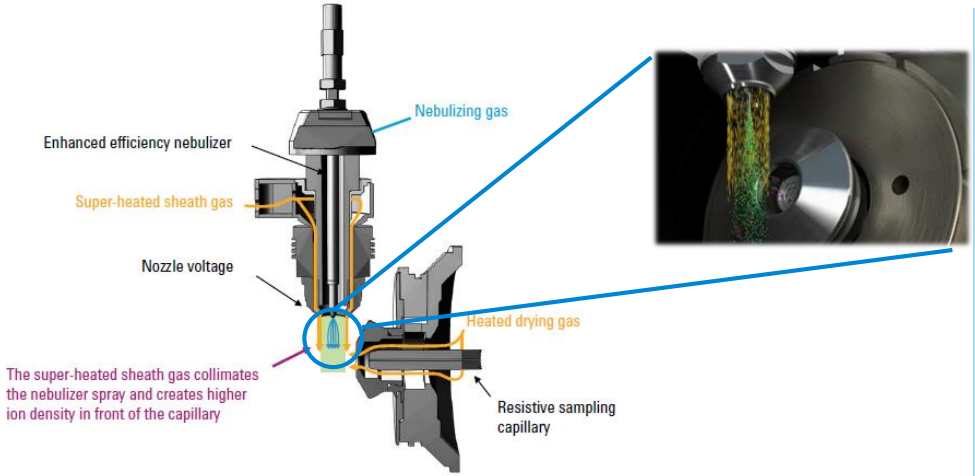
Schematic View of Triple Quadrupole

Agilent Innovations

High Pressure Stage 1

Low Pressure Stage 2



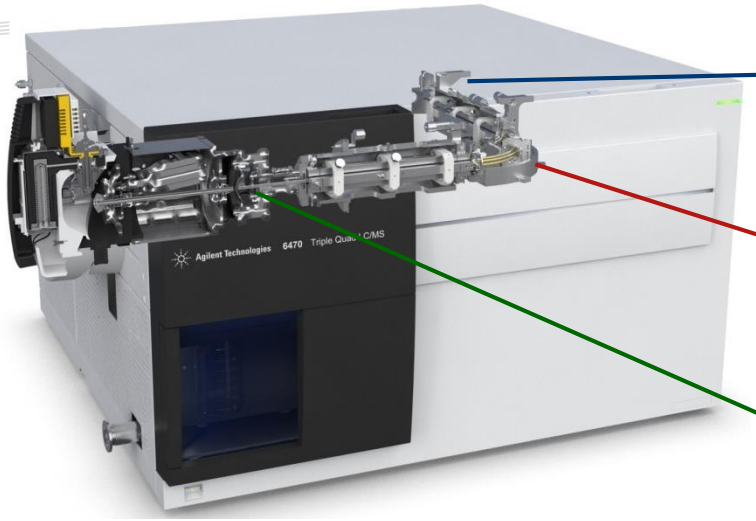


Agilent Jet Stream Technology

- Thermal gradient focusing
- Efficient desolvation
- Creates an ion rich zone
- Up to 10x gains in sensitivity

Agilent iFunnel Technology

- Hexabore Capillary
- Dual Ion Funnel
- Increased ion generation
- Enhanced ion sampling



3

- An Ion Detector with High Energy Conversion Dynode and Low Noise
- Improved ion detection

2

- A Curved and Tapered Hexapole Collision Cell
- Effective ion collection

1

- Enhanced Q1 Ion Optics
- Improved ion transmission

Triggered MRM
Quantitation with Confirmation: Fingerprinting

Full Scan Approach:
Scan the entire fingerprint

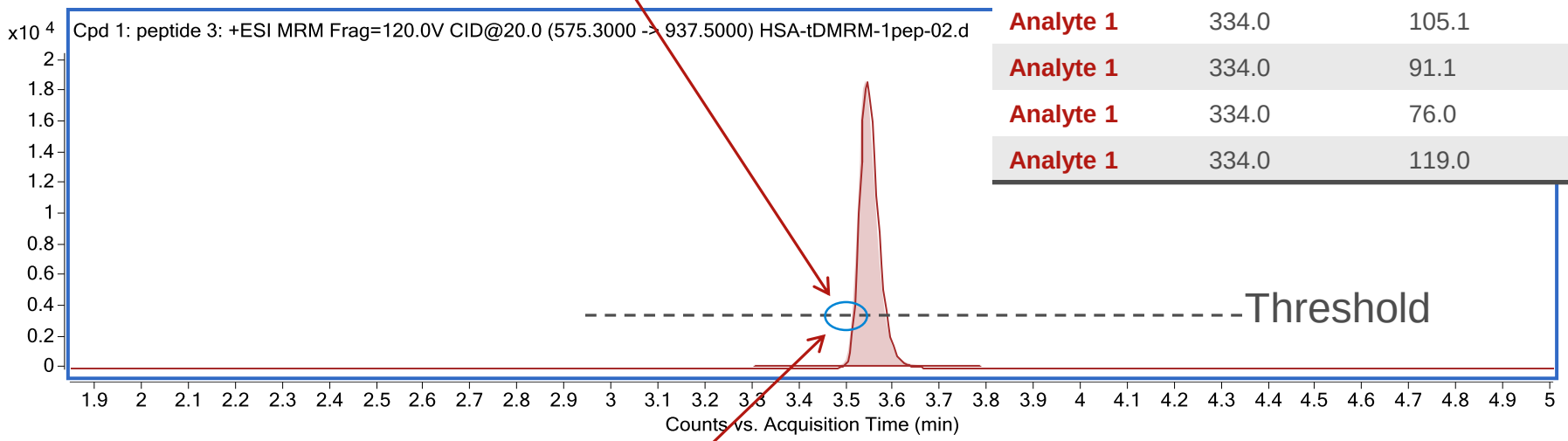
tMRM Approach:
Focus on known fingerprint features

Two possible scenarios:

- Confirmation of positive findings with additional information (spectral matching)
- Elimination of potential false detects caused by matrix interferences

Triggered MRM (tMRM) Analysis

Secondary MRM
Transitions are “Triggered”



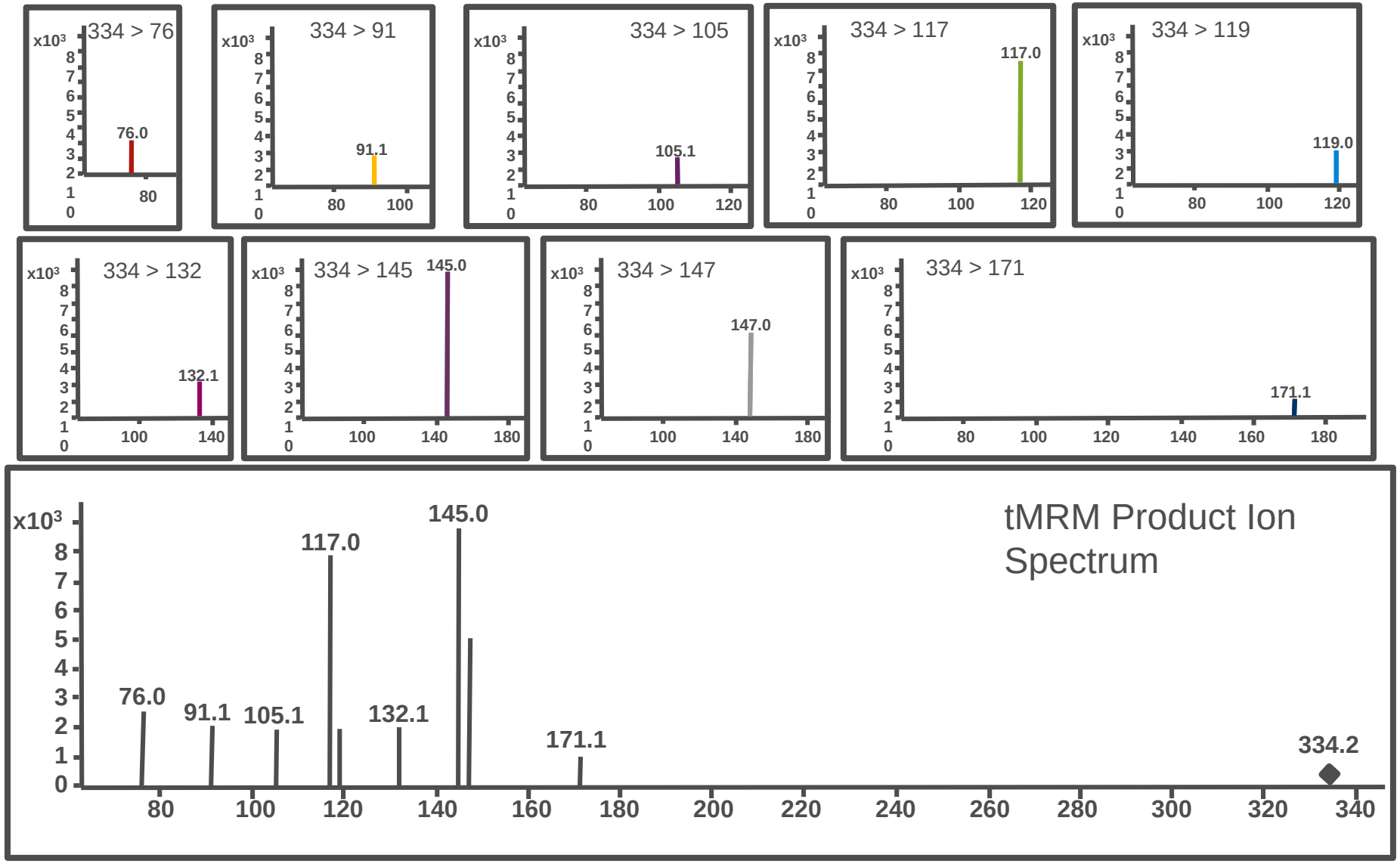
Triggered cycle (above threshold)

Compound	Precursor	Product
Analyte 1	334.0	145.0
Analyte 1	334.0	117.0
Analyte 1	334.0	132.1
Analyte 1	334.0	105.1
Analyte 1	334.0	91.1
Analyte 1	334.0	76.0
Analyte 1	334.0	119.0

Primary cycle (below threshold)

Compound	Precursor	Product
Analyte 1	334.0	145.0
Analyte 1	334.0	117.0

tMRM Product Ion Spectrum



tMRM Library Searching

Tebufenpyrad Standard 50 ppb

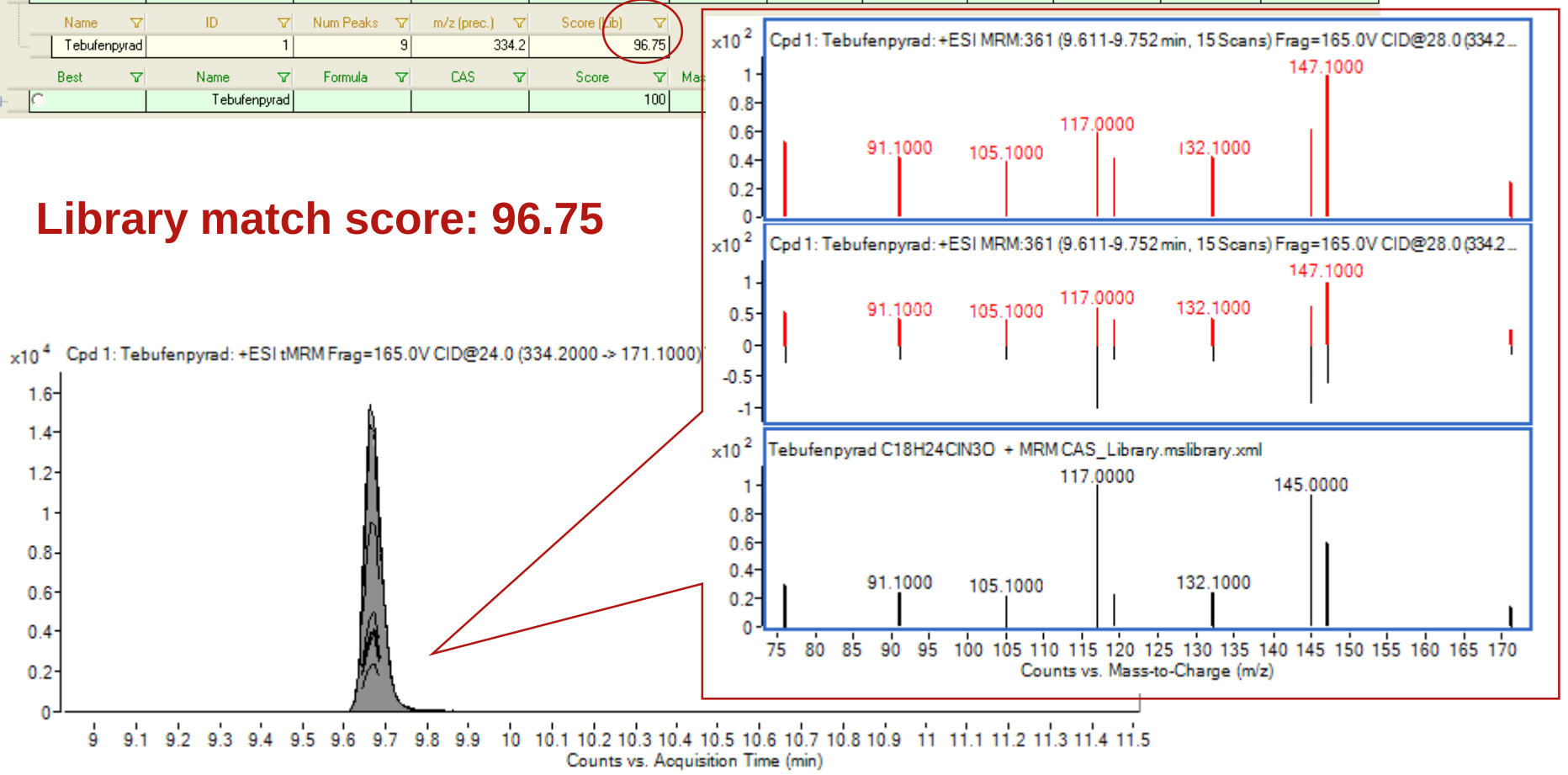
Show/Hide	Label	Polarity	Cpd	Name	Score	Start	RT	End	Width	m/z	Mass (DB)
☑	Cpd 1: Tebufenpyrad	Positive	1	Tebufenpyrad	96.75	9.602	9.664	9.761	0.046	334.2	333.8557

Best	Name	Formula	CAS	Score	Mass (DB)	RT	Score (Lib)	Precursor	Find by MRM	Score (Acq)	ID Source
◉	Tebufenpyrad	C18H24ClN3O	119168-77-3	96.75	333.8557	9.664	96.75				LibSearch

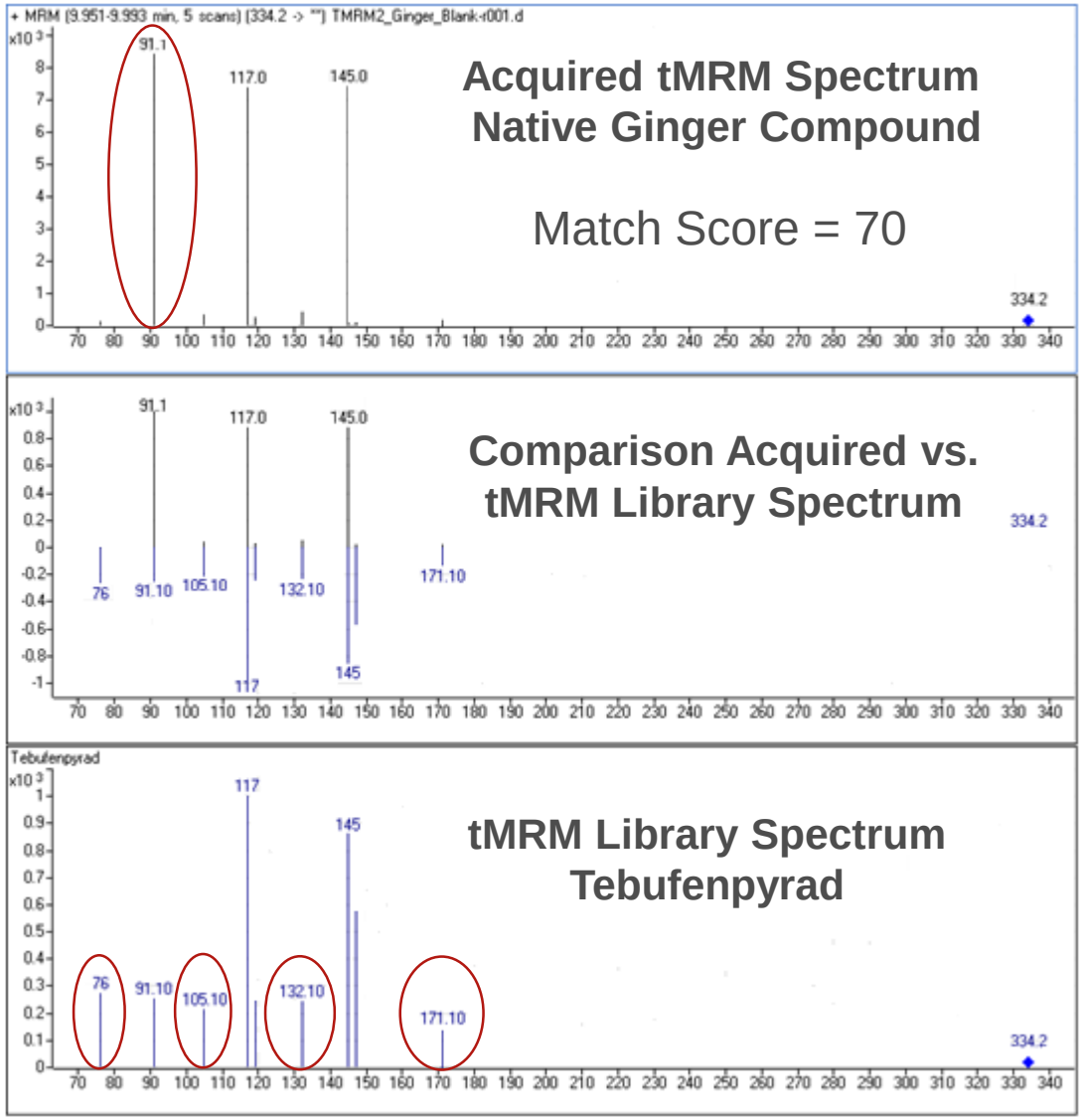
Name	ID	Num Peaks	m/z (prec.)	Score (Lib)
Tebufenpyrad	1	9	334.2	96.75

Best	Name	Formula	CAS	Score
○	Tebufenpyrad			100

Library match score: 96.75

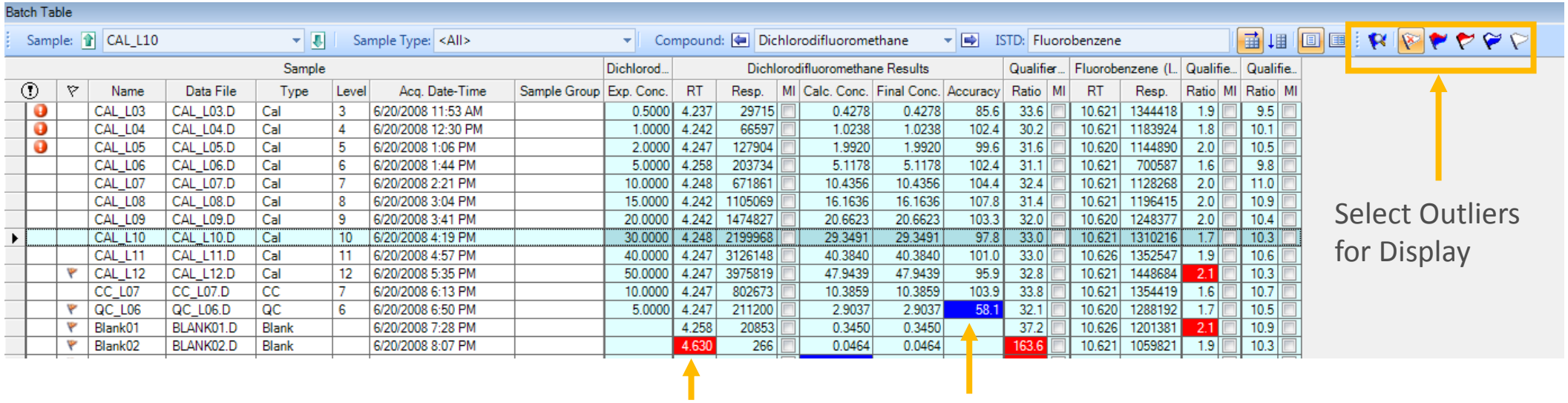


tMRM Prevents False Positive ID



Agilent Technologies

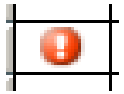
Batch Table - Messages and Outliers



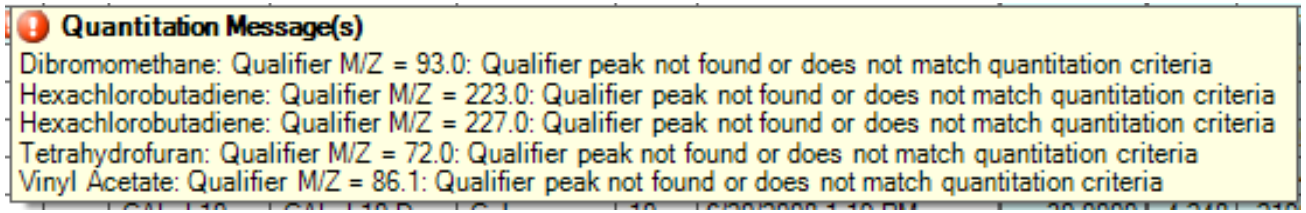
Select Outliers for Display

Red Outlier – High (above upper limit)

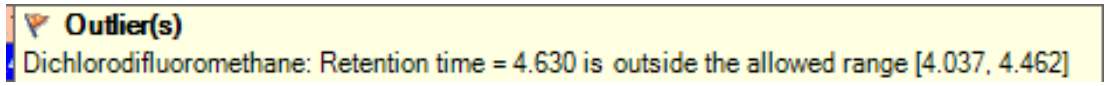
Blue Outlier – Low(below lower limit)



Messages



Outliers



Hover cursor over the outlier or message to display details

tMRM Application Kits For LC/MS

Targeted Screening & Confirmation with QQQ

Pesticides

Test Mix: 254 compounds

DB: 700+ compounds

Library: 200+ compounds

Veterinary Drugs

Test Mix: 146 compounds

DB: 500+ compounds

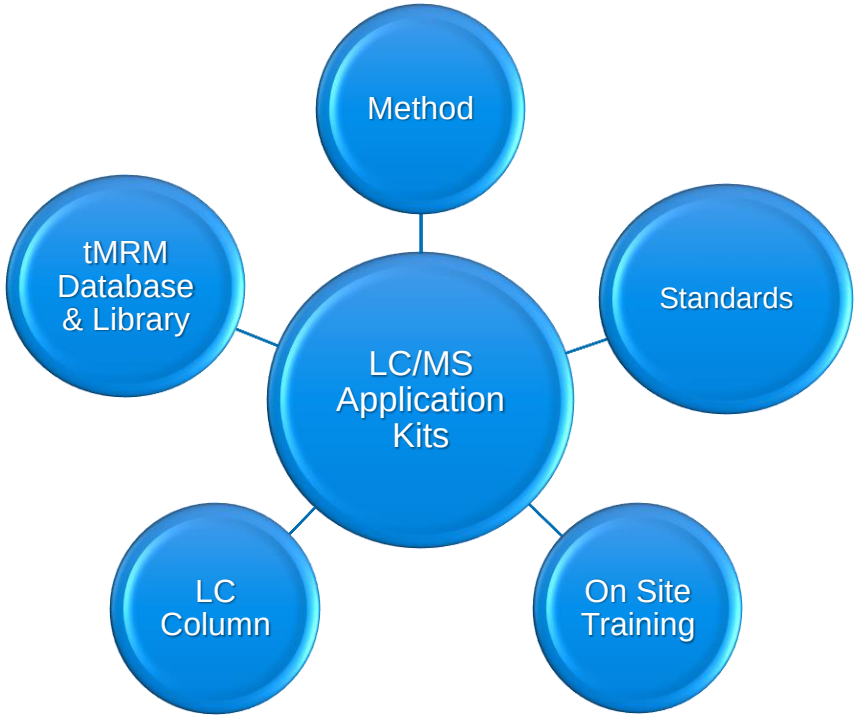
Library: 100+ compounds

Forensic Toxicology

Test Mix: 139 compounds

DB: 2500+ compounds

Library: 100+ compounds



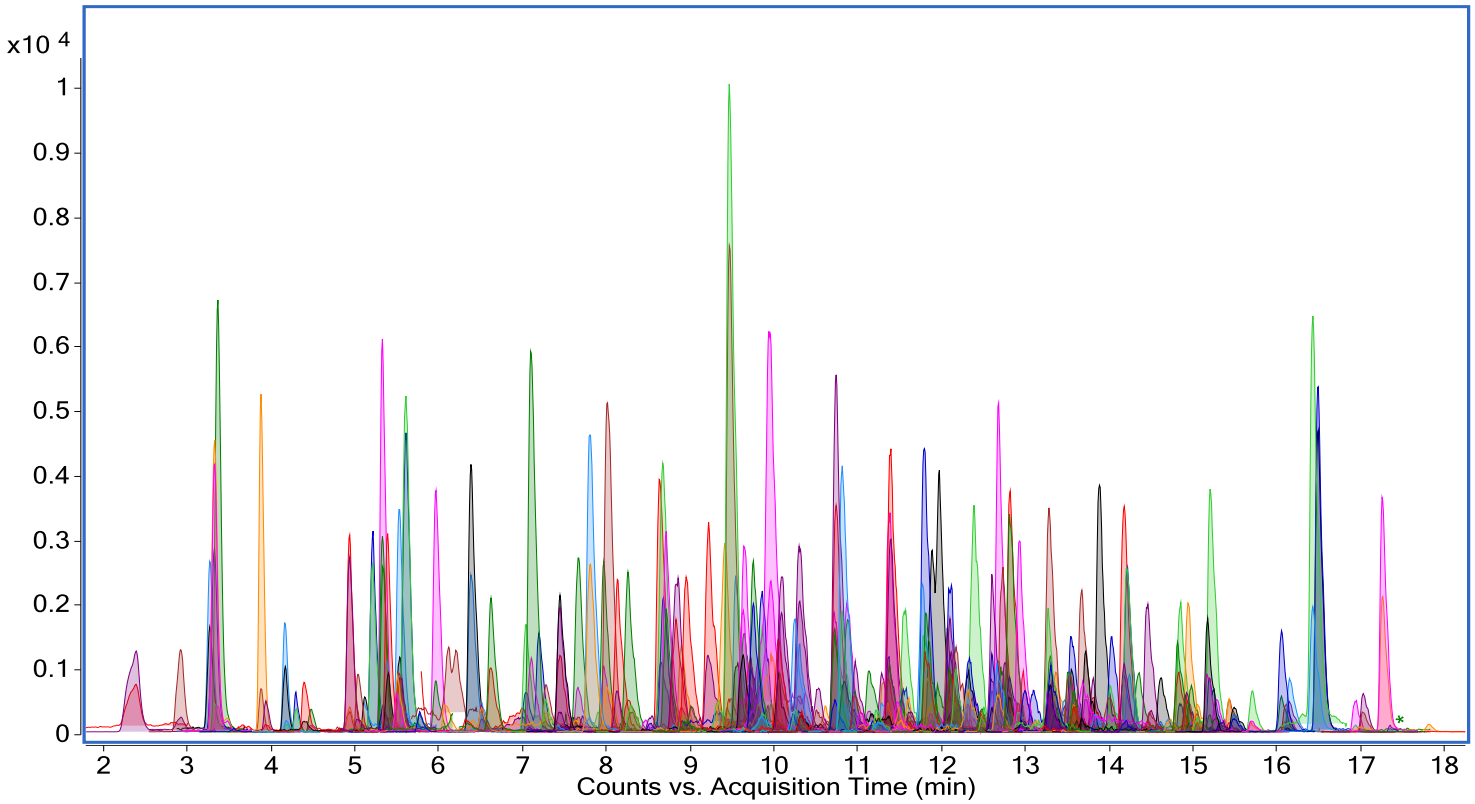
- ***Agilent unique data dependent acquisition for fast and sensitive compound screening, quantitation and confirmation.***

Multi-Residue Pesticide Analysis in Food
6470 QQQ

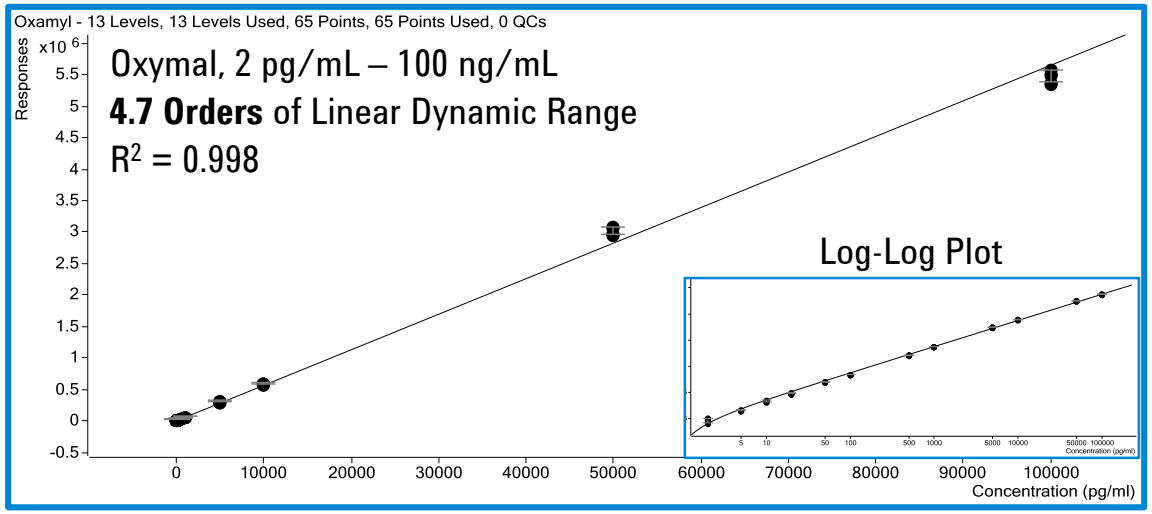
1:20 dilution of > 250 pesticides spiked into black tea at 10 ng/mL (ppb)

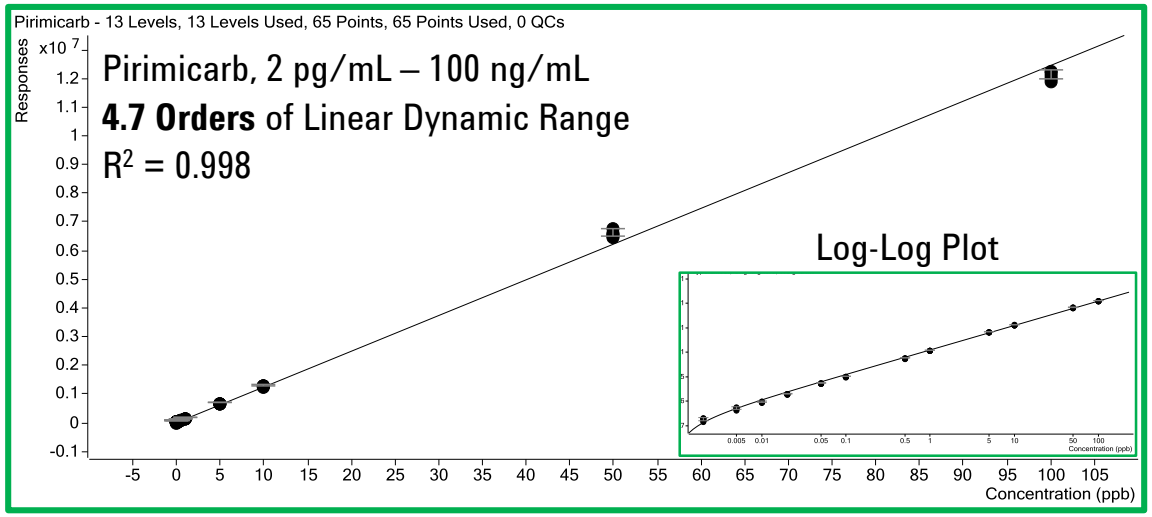
Injection volume = 2 µL

- Multi-residue pesticide analysis in food products – most demanding food safety applications
- Improved **sensitivity** and **precision** of the 6470 allows **accurate** quantitation of pesticides <Maximum Residue Limits (MRLs) imposed by EU, with higher degrees of sample dilution
- Sample dilution reduces matrix effects, improves method **robustness,** allows more efficient ionization and enables the use of solvent calibration with better **accuracy**



Confident Quantitation Over ~5 Orders of Dynamic Range





Levels	%RSD (n = 5)	%Accuracy
2 pg/mL	18.3	114.0
5 pg/mL	5.4	86.8
10 pg/mL	7.4	87.1
20 pg/mL	5.5	83.1
50 pg/mL	1.9	86.6
100 pg/mL	1.1	82.4
500 pg/mL	1.7	89.6
1 ng/mL	1.2	93.8
5 ng/mL	1.0	104.4
10 ng/mL	2.1	103.3
50 ng/mL	2.3	106.2
100 ng/mL	1.8	96.5

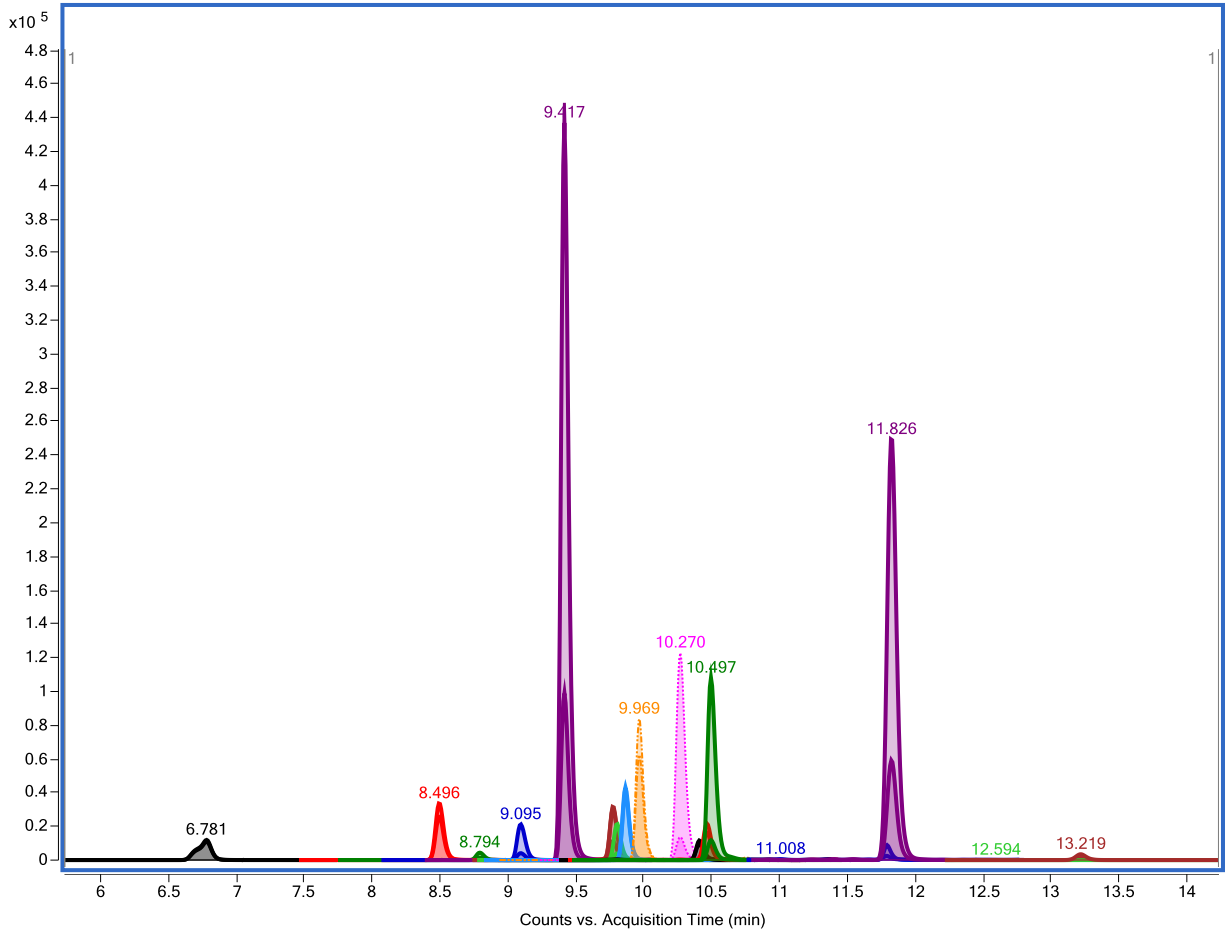
Levels	%RSD (n = 5)	%Accuracy
2 pg/mL	15.0	117.2
5 pg/mL	10.7	97.1
10 pg/mL	4.4	82.2
20 pg/mL	1.7	80.6
50 pg/mL	2.8	85.5
100 pg/mL	2.3	80.2
500 pg/mL	1.2	88.3
1 ng/mL	1.1	93.3
5 ng/mL	0.7	103.1
10 ng/mL	2.0	101.6
50 ng/mL	1.7	105.6
100 ng/mL	1.3	97.0

15 Pesticides Screened in Marijuana

Colorado Department of Agriculture Marijuana Testing Metadata

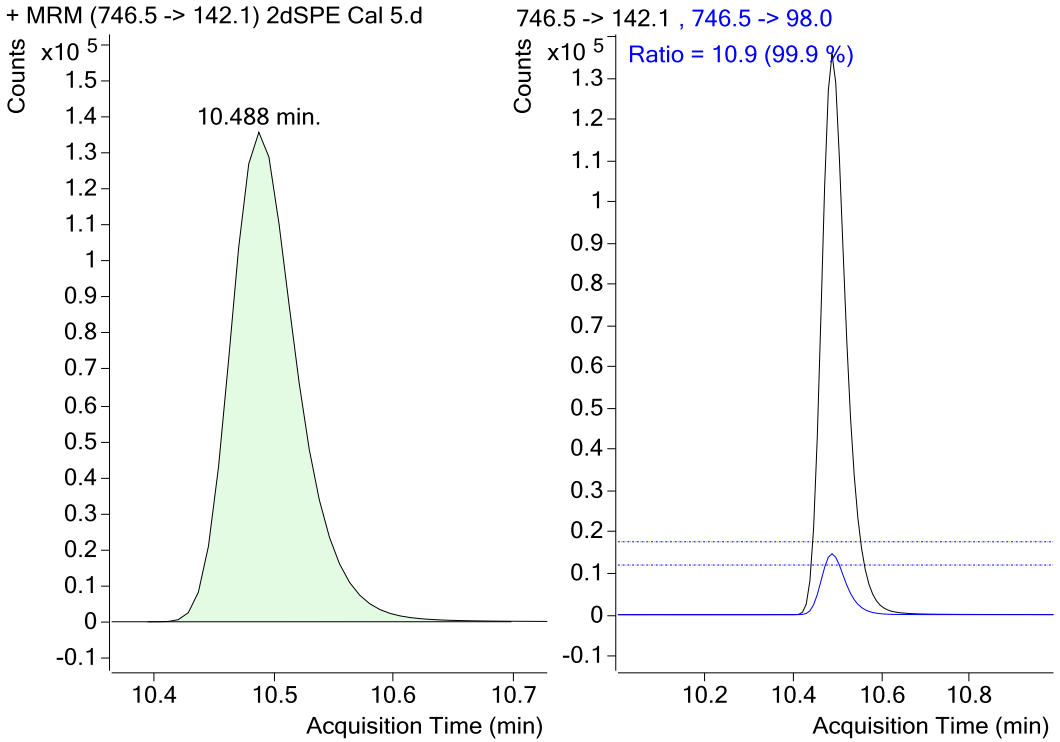
FY 2016	(Sample Results Reported 7/1/15 Thru 06/30/16)			
	Samples Tested:	599		
	Samples with Detects:	391	65%	
	Non-detect Samples:	208	35%	
	Azadirachtins Only:	97	16%	
	Compounds Detected			**# of Detects**
	Myclobutanil			190
	Azadirachtins			165
	Imidacloprid			77
	Spiromesifen			72
	Bifenazate			43
	Avermectins			44
	Etoxazole			27
	Piperonyl Butoxide			15
	Azoxystrobin			13
	Spinosyn A			15
	Spinosyn D			6
	Pyrethrins			3
	Propiconazole			3
	Spirotetramat			1
	Chlorfenapyr			2

Overlay MRM of 15 Pesticide Screen in Marijuana

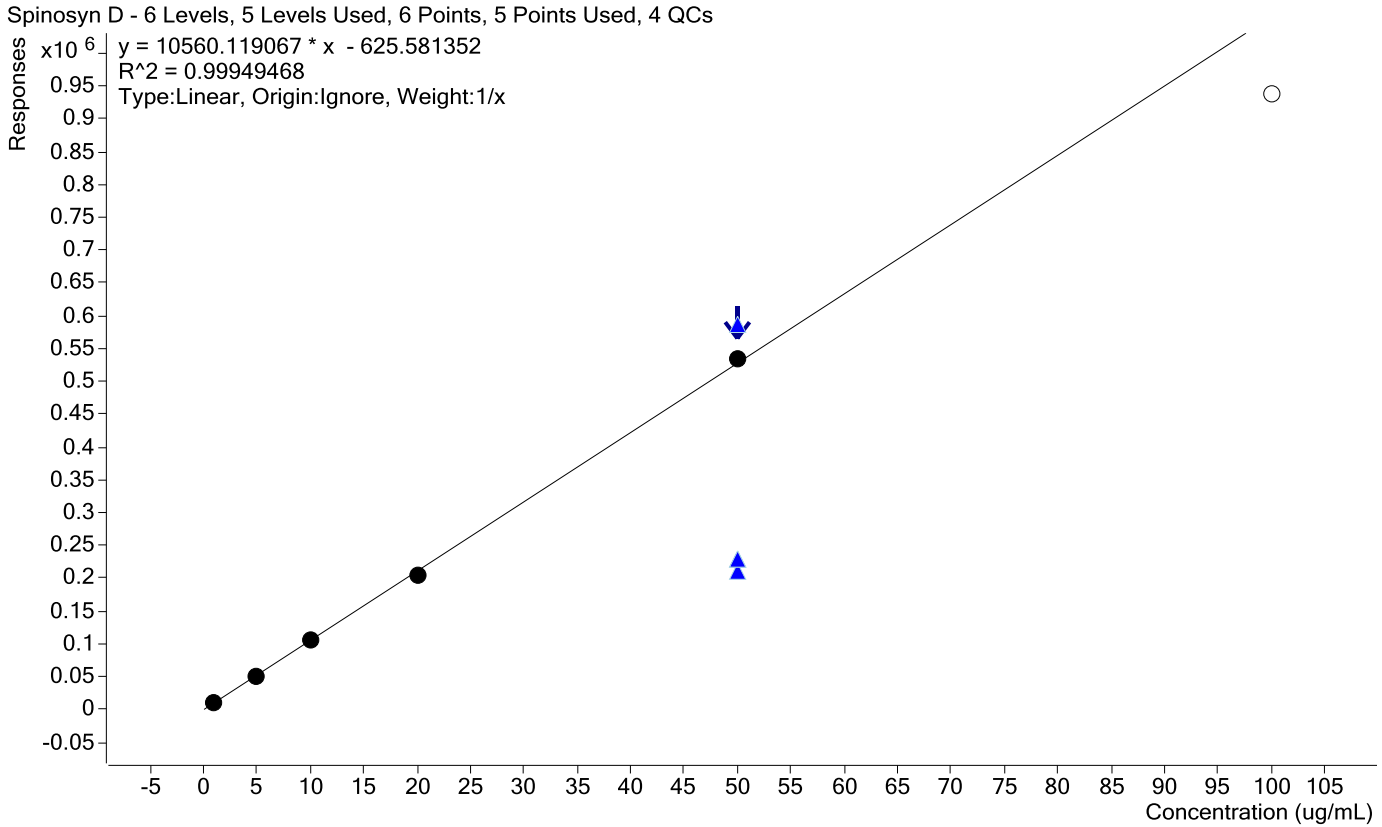


Pesticides Screened in Marijuana

Spinosyn D MRM and Qualifier in Marijuana



Calibration Curve of Spinosyn D in Marijuana



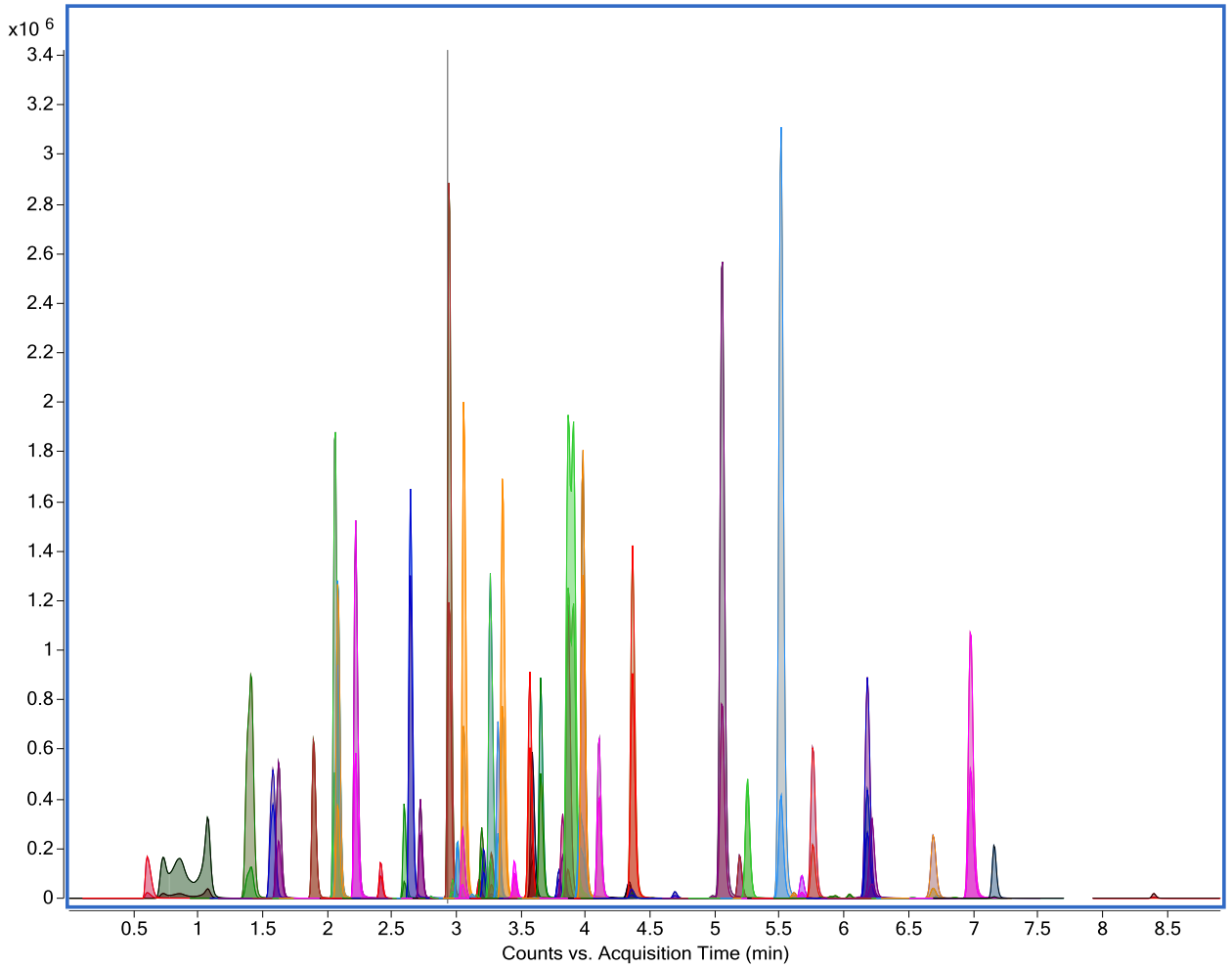
62 Common Pesticides in Marijuana

State of Oregon Pesticide List

Acephate	Clofentezine	Hexythiazox	Piperonyl butoxide
Acequinocyl	Cyfluthrin	Imazalil	Prallethrin
Acetamiprid	Cypermethrin	Imidacloprid	Propiconazole
Aldicarb	Daminozid	Jasmolin I	Pyrethrin
Avermectin B1a	Diazinon	Kresoxim methyl	Pyridaben
Avermectin B1b	Dibrom Naled	Malathion	Spinosyn A
Azoxystrobin	Dichlorvos	Metalaxyl	Spinosyn D
Bifenazate	Dimethoate	Methiocarb	Spiromesifen
Bifenthrin	Ethoprophos	Methomyl	Spirotetramat
Boscalid	Etofenprox	MGK-264	Spiroxamine
Carbaryl	Etoxazole	Myclobutanil	Tebuconazole
Carbofuran	Fenoxycarb	Oxamyl	Thiacloprid
Chlorantraniliprole	Fenpyroximate	Paclobutrazol	Thiamethoxam
Chlorfenapyr	Fipronil*	Parathion-methyl	Trifloxystrobin
Chlorpyrifos	Flonicamid	Permethrin	
Cinerin I	Fludioxonil*	Phosmet	

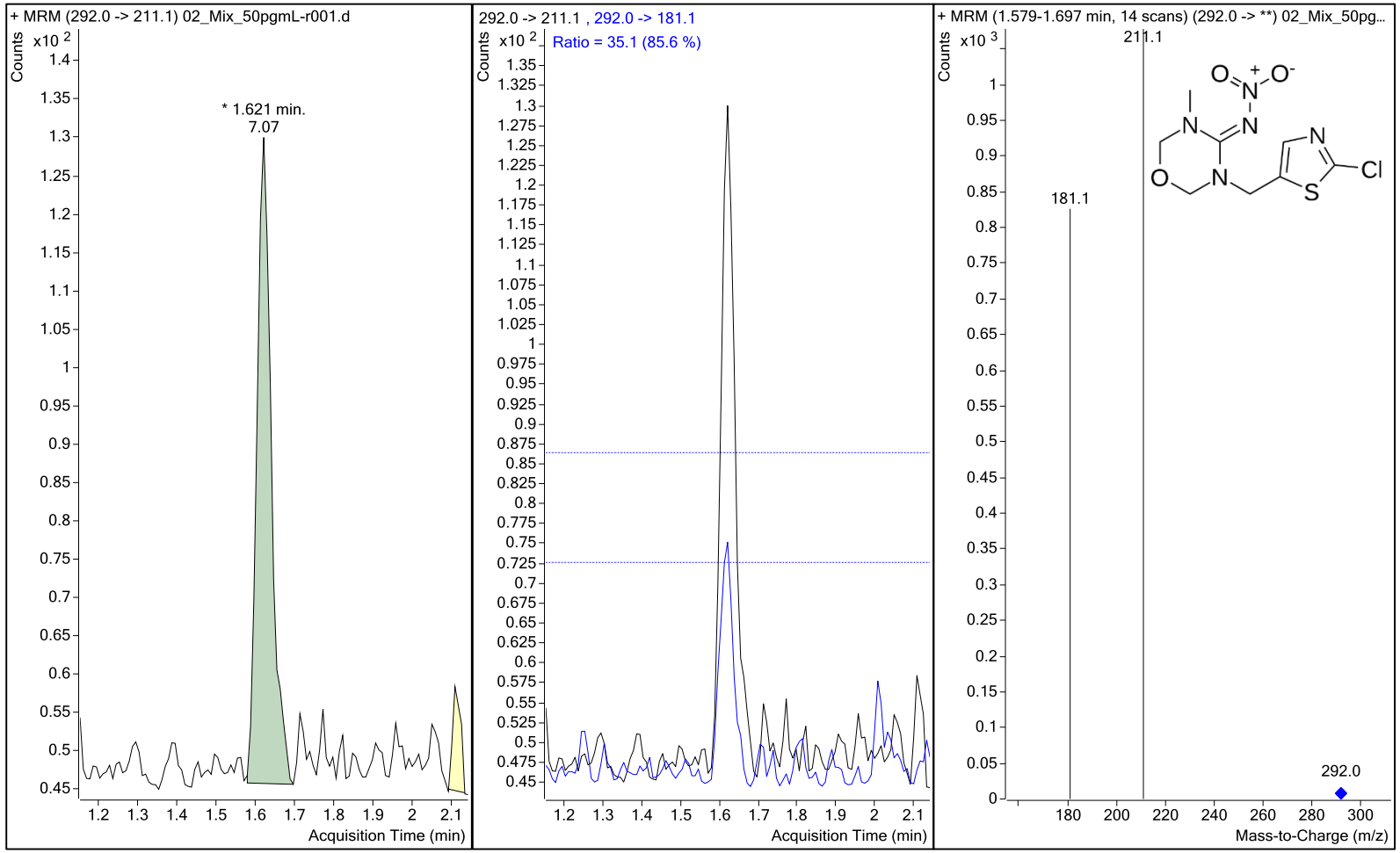
* Negative mode

Overlaid MRM Transitions of 62 Pesticides in 10 min

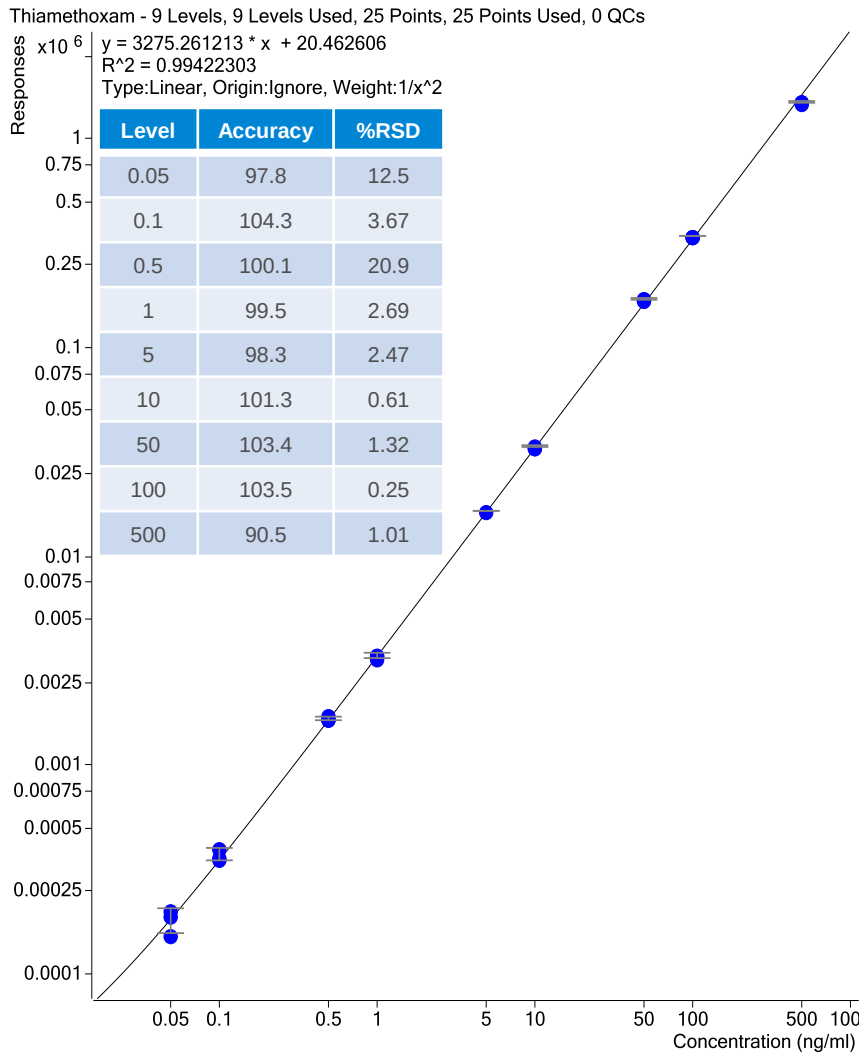


Excellent Sensitivity and Linearity

Thiamethoxam, 100 fg on column



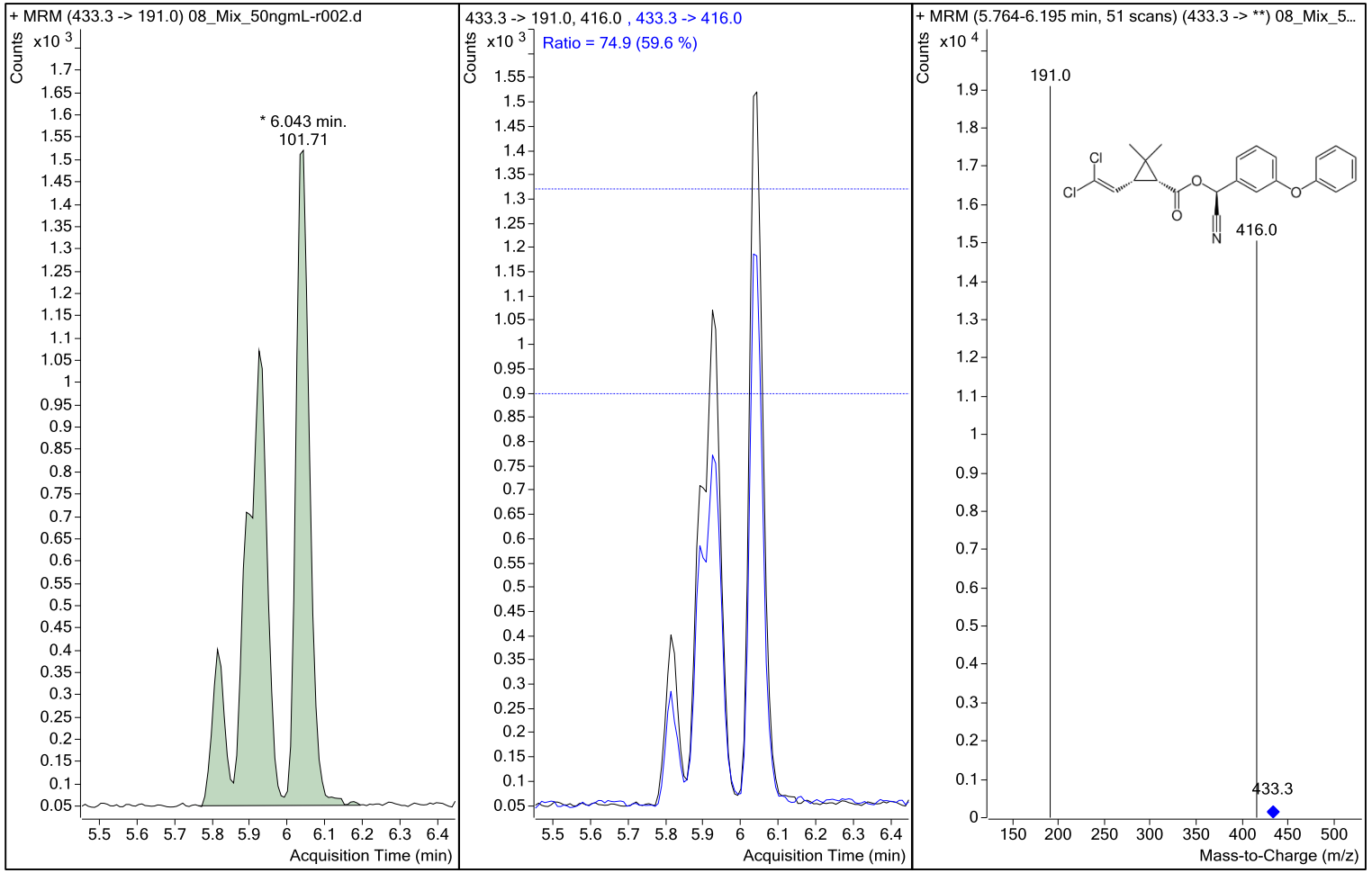
Log-Log Plot from 0.05 ng/mL to 500 ng/mL



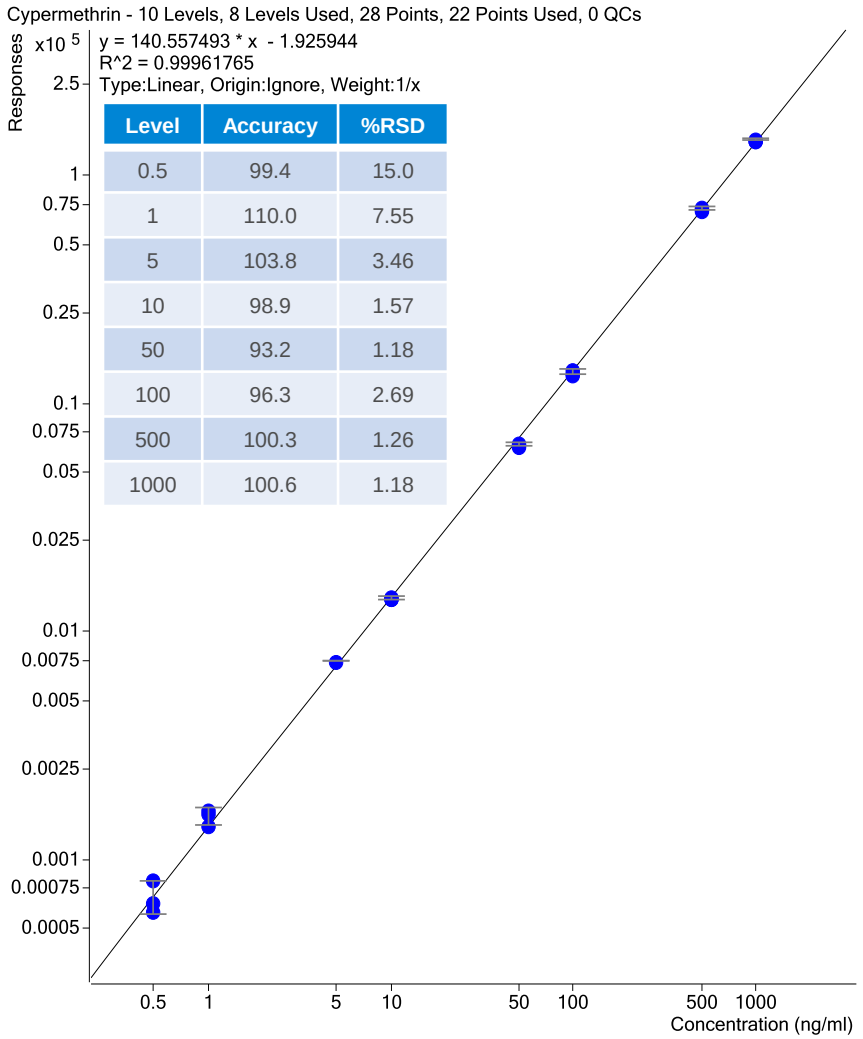
Level	Accuracy	%RSD
0.05	97.8	12.5
0.1	104.3	3.67
0.5	100.1	20.9
1	99.5	2.69
5	98.3	2.47
10	101.3	0.61
50	103.4	1.32
100	103.5	0.25
500	90.5	1.01

Excellent Sensitivity and Linearity

Cypermethrin: 1 pg on column



Log-Log Plot from 0.5 ng/mL to 1000 ng/mL



Level	Accuracy	%RSD
0.5	99.4	15.0
1	110.0	7.55
5	103.8	3.46
10	98.9	1.57
50	93.2	1.18
100	96.3	2.69
500	100.3	1.26
1000	100.6	1.18

Thank You Very Much!

Questions?