Agilent Sample Preparation
The Pesticide Analysis Workflow

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

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Sample Preparation Application Specialist

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
  - Sample extraction, cleanup and dilution
  - EMR-Lipid dispersive SPE
- Future Investigations, Aspects and Considerations

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

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

Analyte	Chemical Abstract Services (CAS) Registry number	Action level ppm
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
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*	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†	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

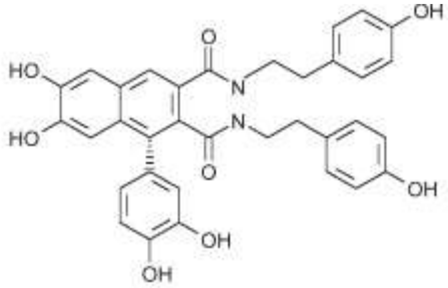
* Permethrins should be measured as cumulative residue of cis- and trans-permethrin isomers (CAS numbers 54774-45-7 and 51877-74-8).

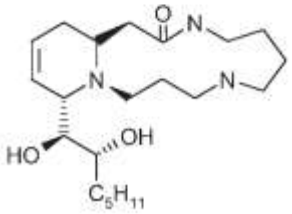
† Pyrethrins should be measured as the cumulative residues of pyrethrin 1, cinerin 1 and jasmolin 1 (CAS numbers 121-21-1, 25402-06-6, and 4466-14-2 respectively).

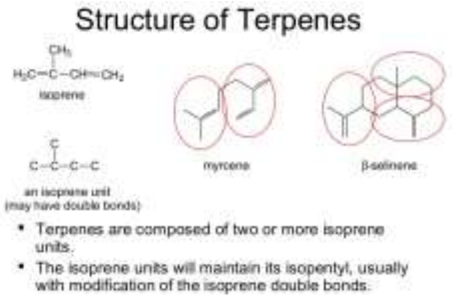
Range 0.1-1 ppm or 100-1000 ppb

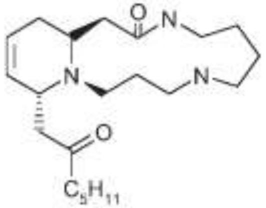
Constituents of Cannabis Plants: Complex

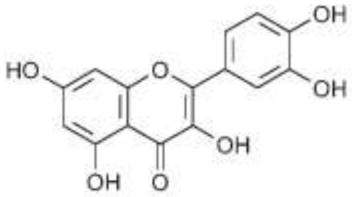
- **Nitrogenous compounds (27 known)**
- Amino acids (18),
- Proteins (3)
- Glycoproteins (6)
- Enzymes (2)
- **Sugars and related compounds (34)**
- Hydrocarbons (50)
- Simple alcohols (7)
- Aldehydes (13)
- Ketones (13)
- Simple acids (21)
- **Fatty acids (22)**
- Simple esters (12)
- Lactones (1)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

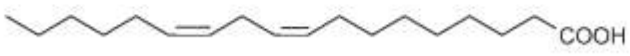


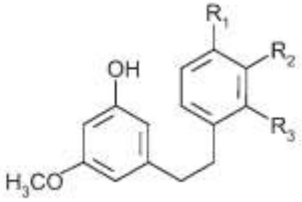










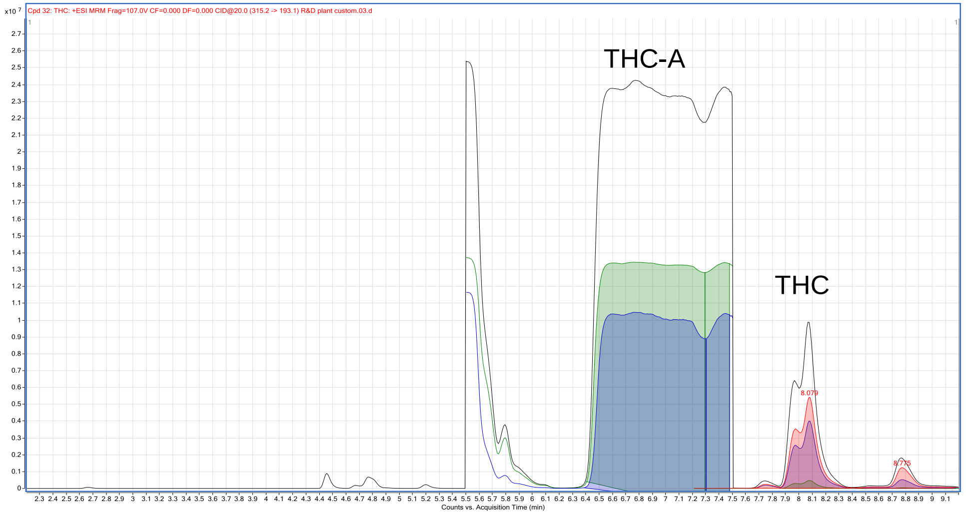


R_1 = OH, R_2 = isoprenyl, R_3 = H

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%)



LC/MSMS analysis after QuEChERS and dSPE cleanup (universal)

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 $MgSO_4$
  - 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

Add 15 mL water and 15 mL ACN (1% acetic acid), shake 30 minutes

Add AOAC QuEChERS salts, shake 2 min, centrifuge 5000 rpm, 5 min

Clean-up transfer aliquot to dispersive SPE

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2nd Clean-up transfer aliquot to dispersive SPE???

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)

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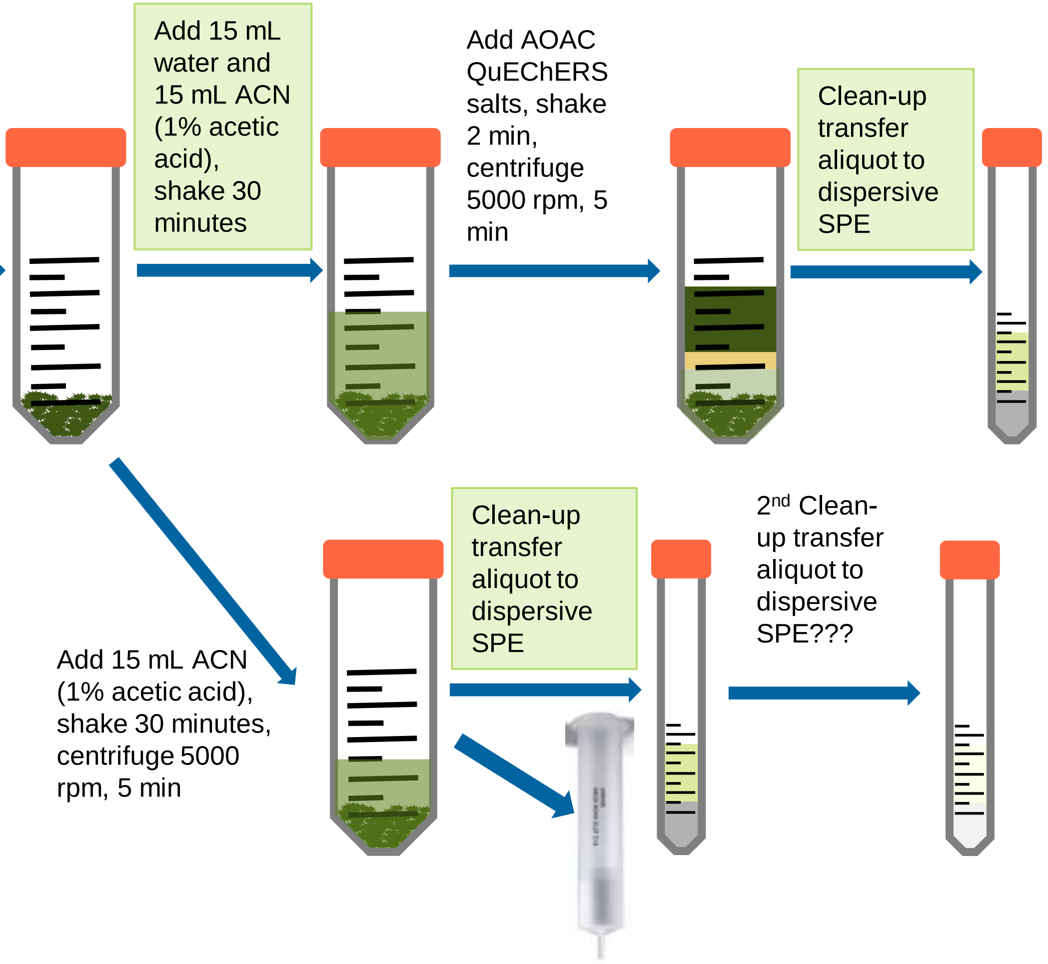

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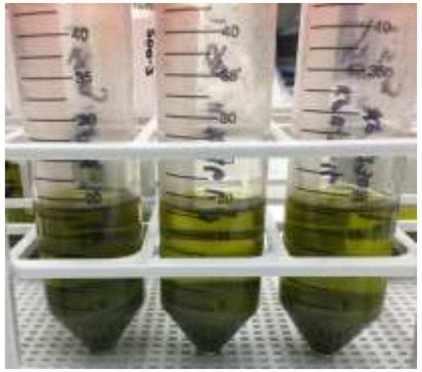

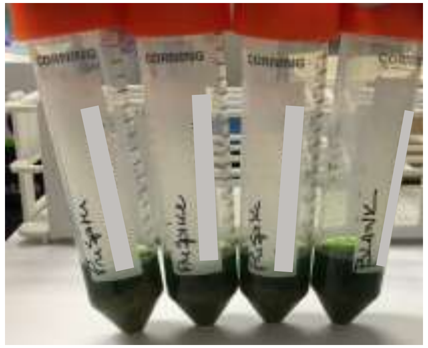

QuEChERS Extraction/Partitioning
or
ACN (1% acetic acid) extraction

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

Step 2:

Clean-up: Dispersive SPE

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

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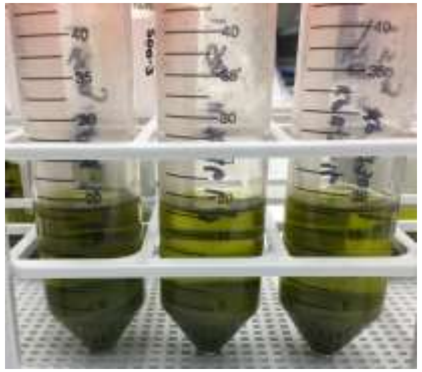

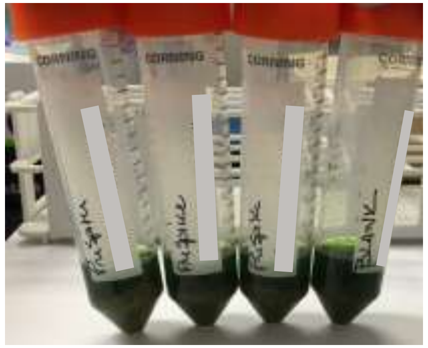

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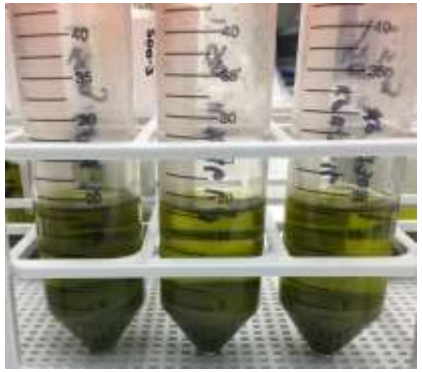

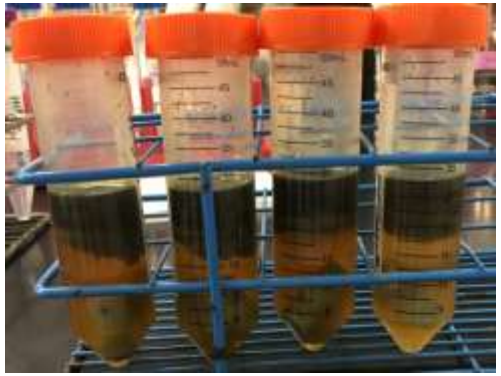

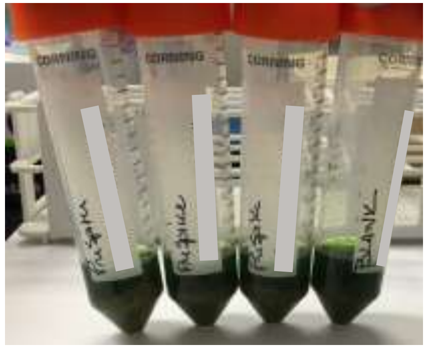

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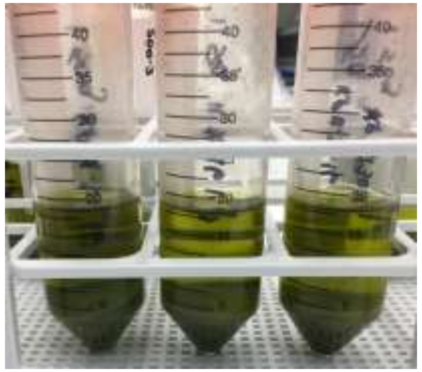

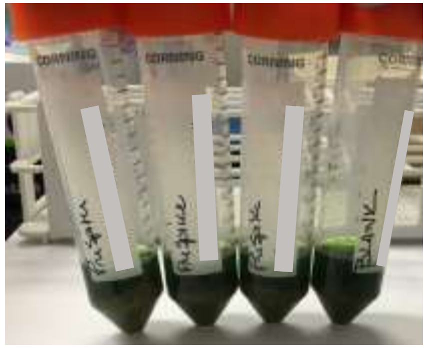

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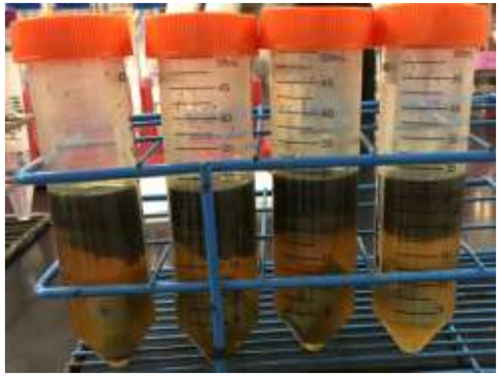

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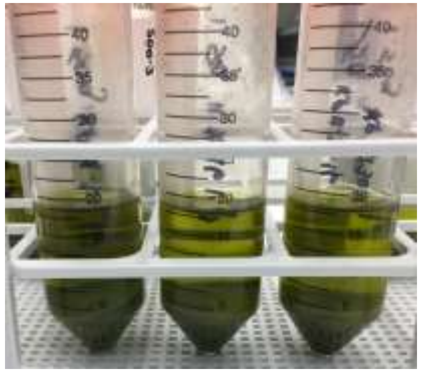

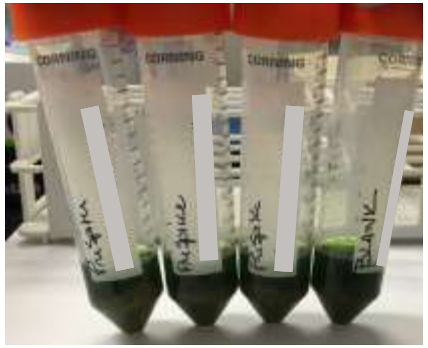

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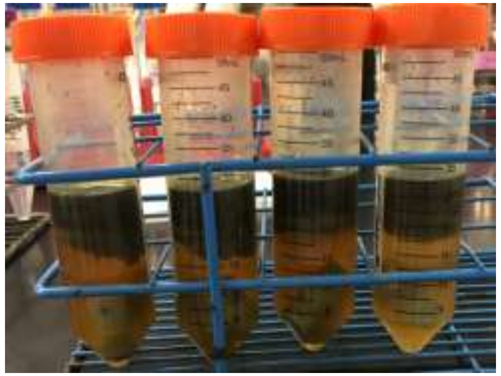

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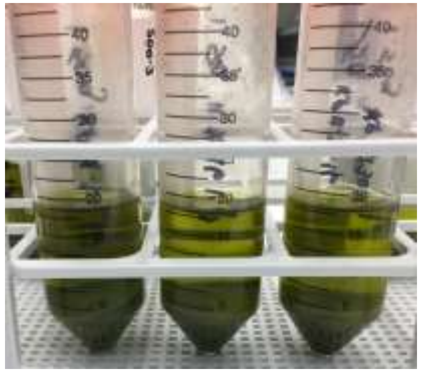

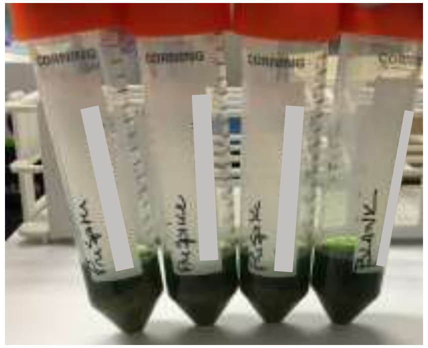

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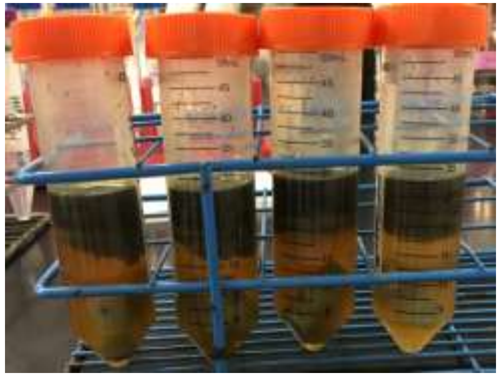

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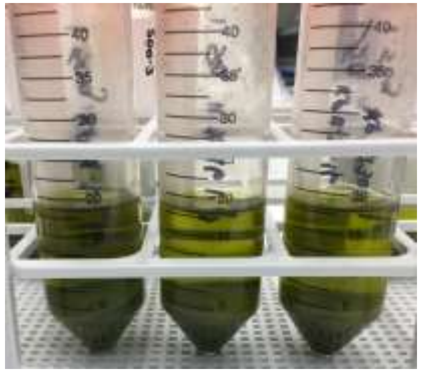

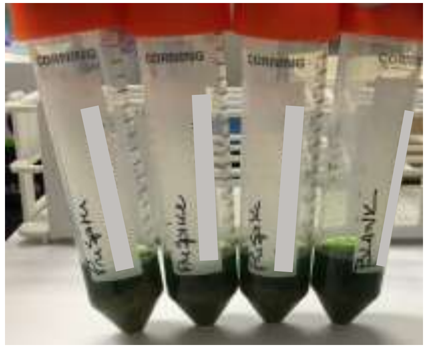

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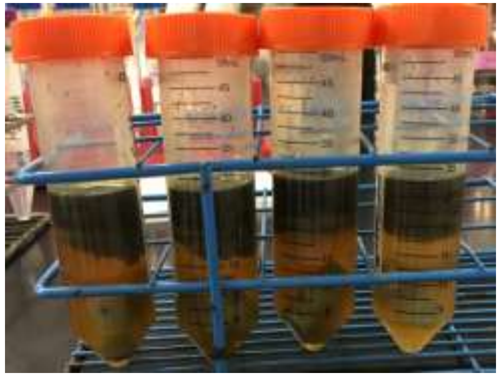

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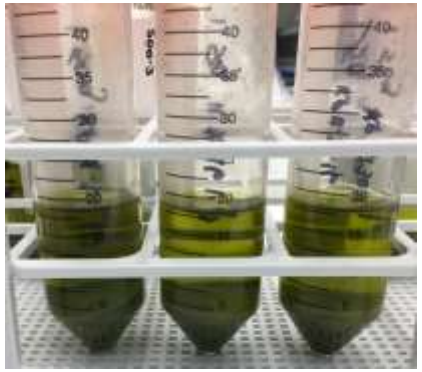

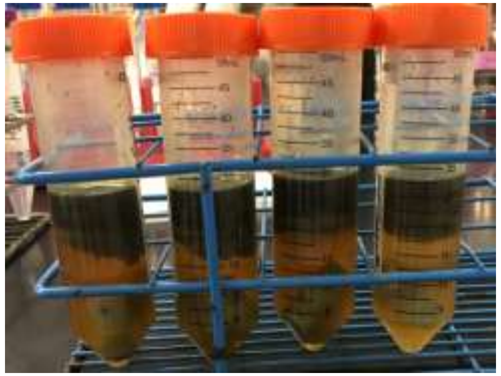

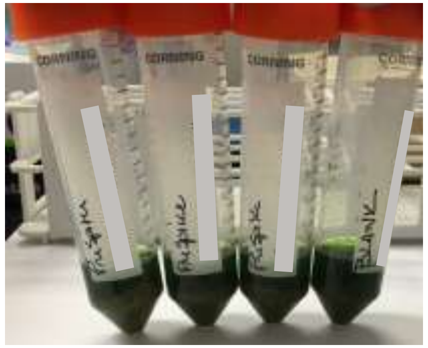

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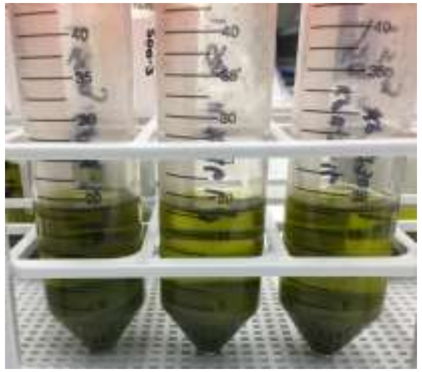

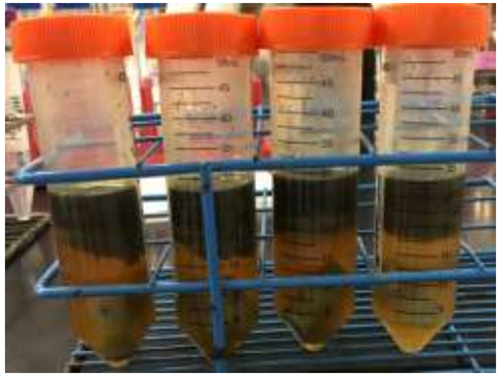

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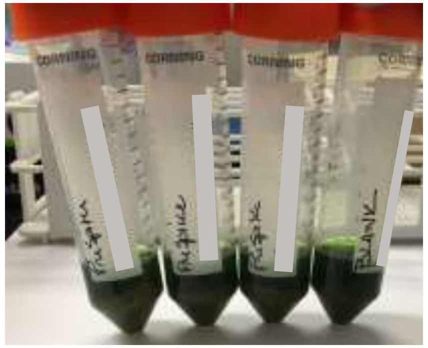

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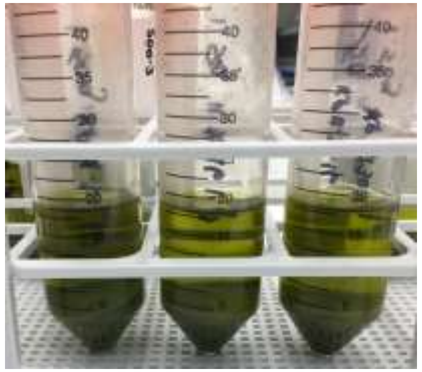

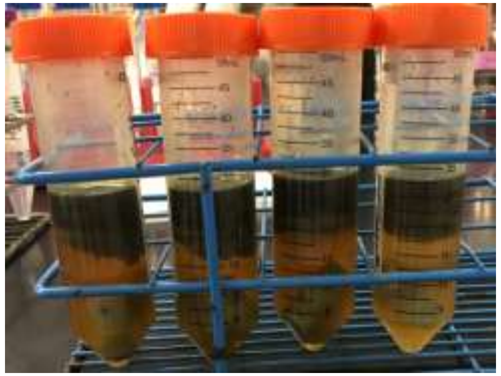

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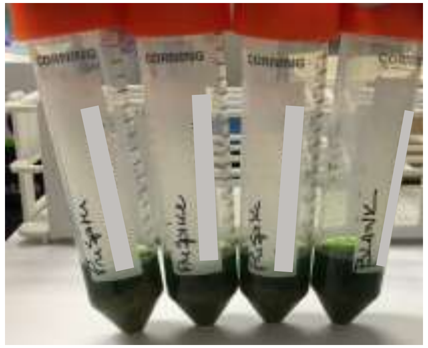

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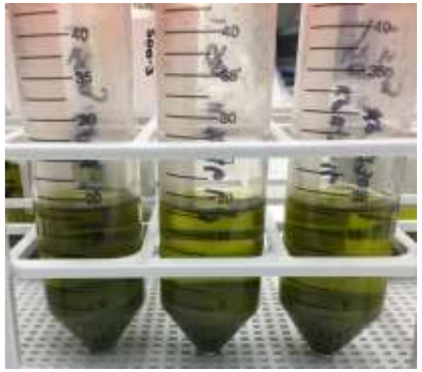

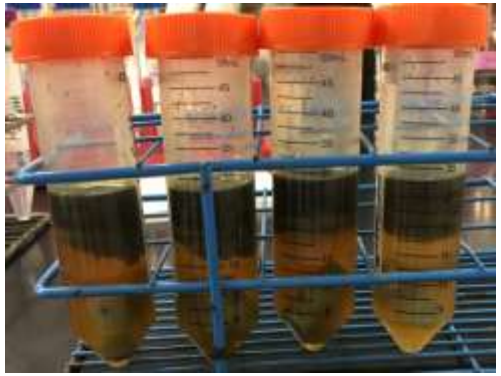

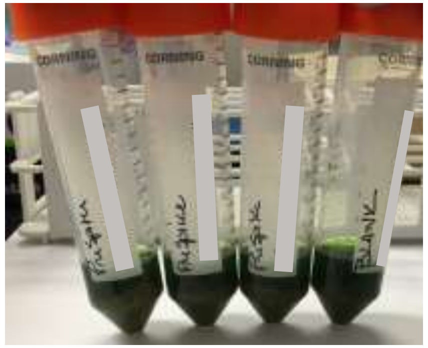

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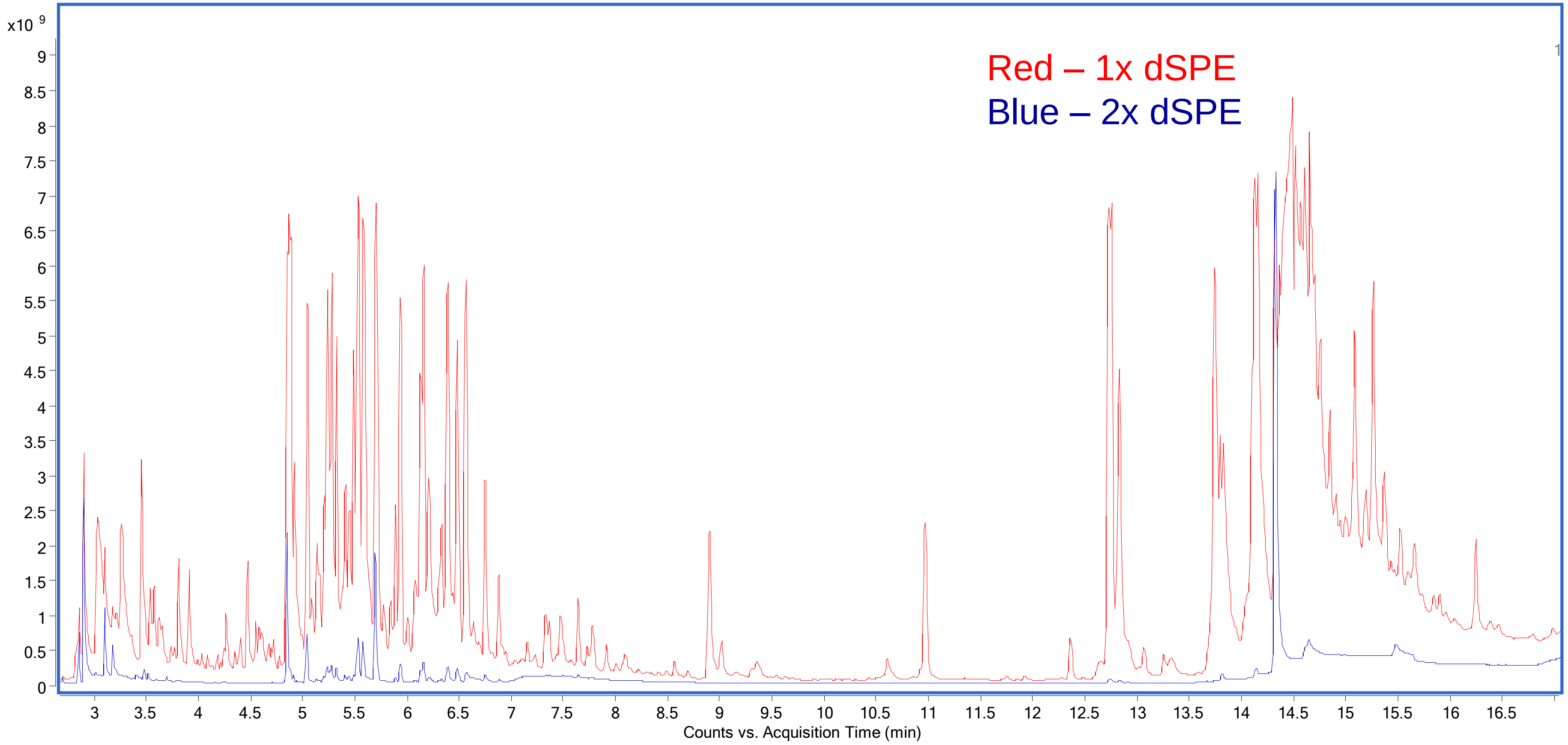
Cannabis mixture cleanup – QuEChERS with 1x dSPE vs. 2 x dSPE custom dispersive steps



GC MS 7010 Triple Quad TIC Scan, 0.5 uL injection, Gain 10

Custom dispersive: Mixture of PSA, C18-EC, GCB, additional sorbent, and $MgSO_4$

Sample Preparation: Insights and Observations

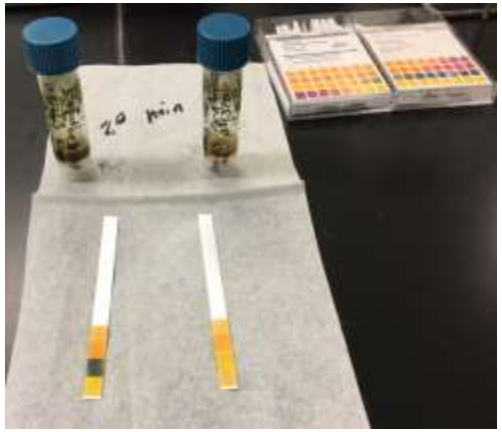
Step 1: QuEChERS or ACN Extraction

- Addition of Water, vortex 15-30 minutes

5 min

Water 50 mM Na Acetate pH 3.5

10 min



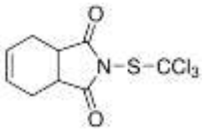
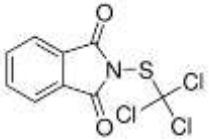
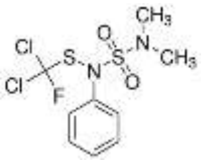
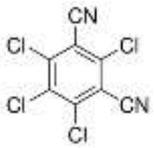
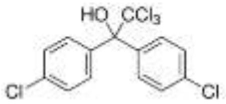
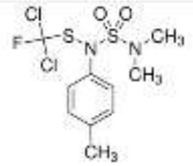
20 min with indicator pH range

5 min with indicator pH range

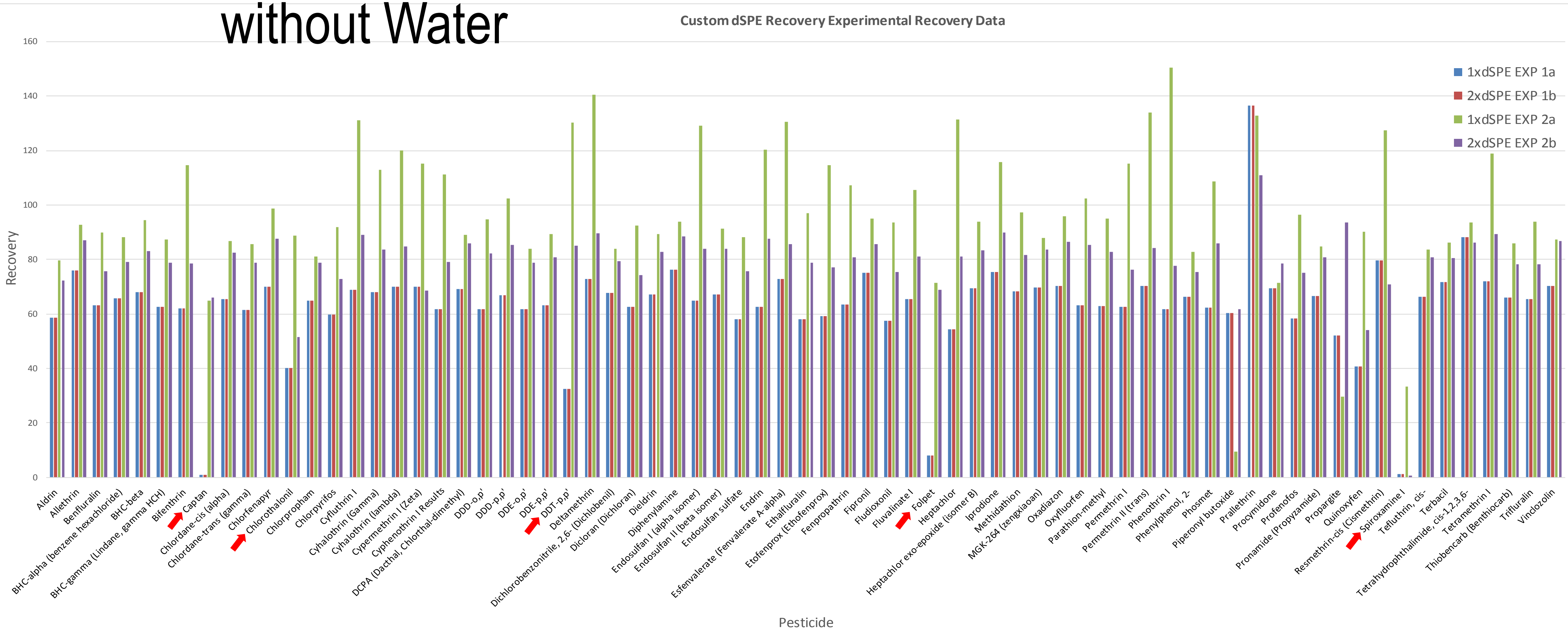
Water/ACN (1:1 v/v)

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



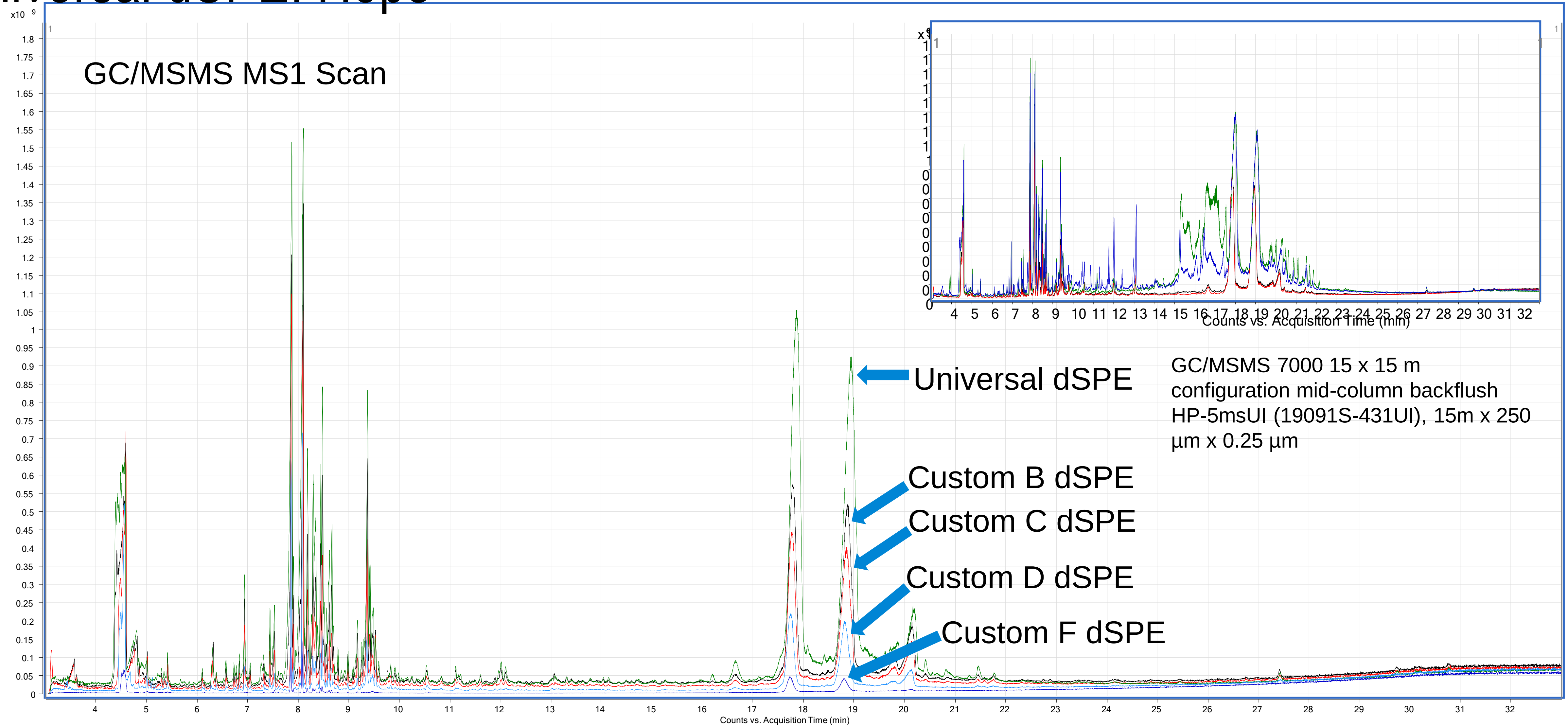
EXP 1a: AOAC QuEChERS: ACN (1%AA) 13.75 mL, dSPE custom, 1xdSPE

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

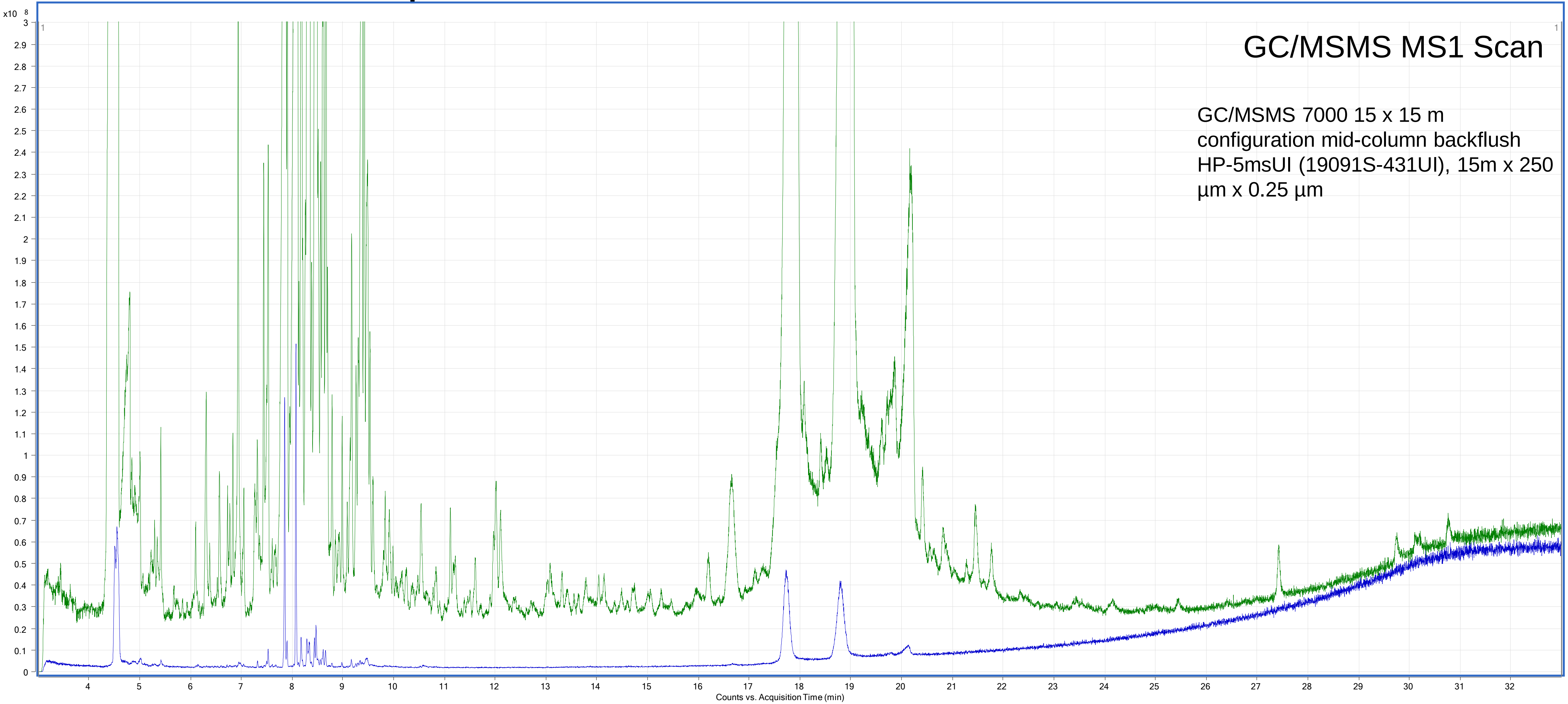
EXP 2a: ACN (1%AA) 13.75 mL only no QuEChERS salts and no H2O, dSPE custom, 1xdSPE

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

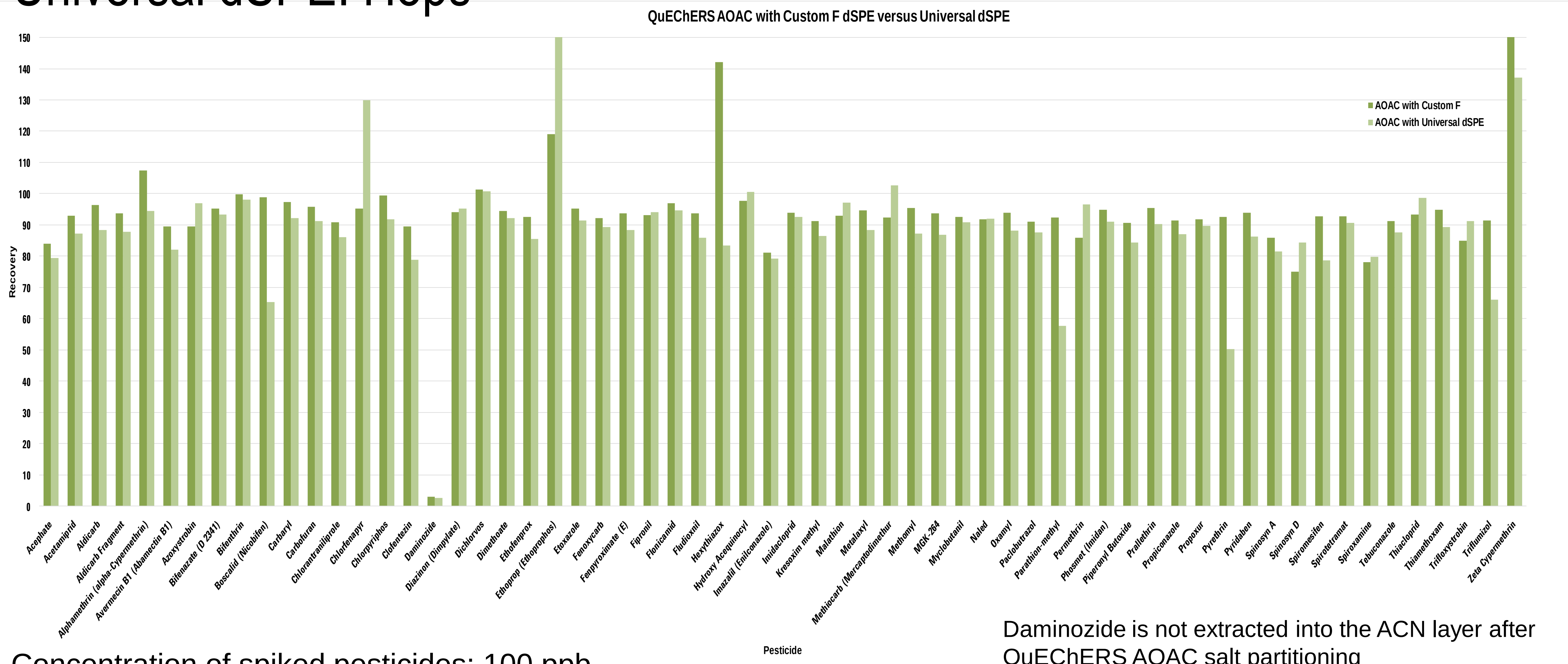
QuEChERS AOAC Extraction/Partitioning with Custom dSPE versus Universal dSPE: Hops



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

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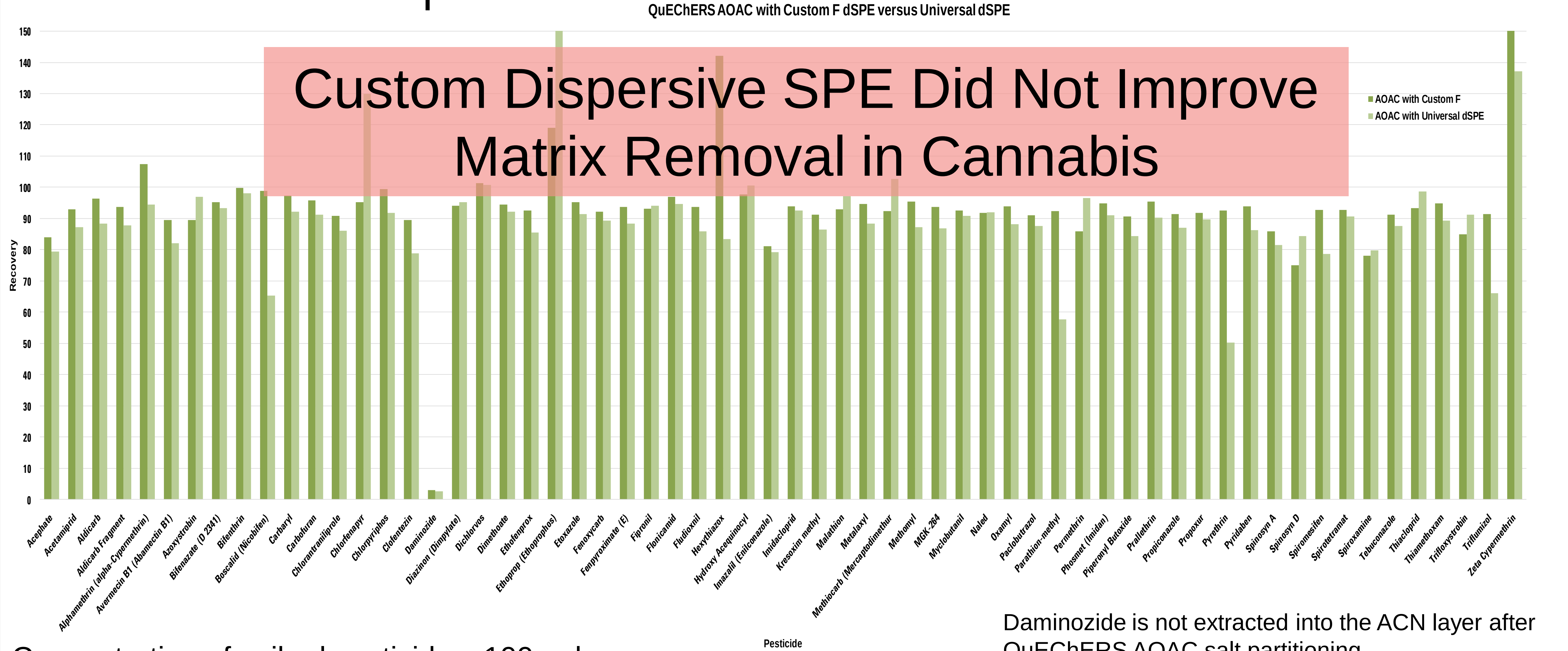

Custom Dispersive SPE Did Not Improve Matrix Removal in Cannabis

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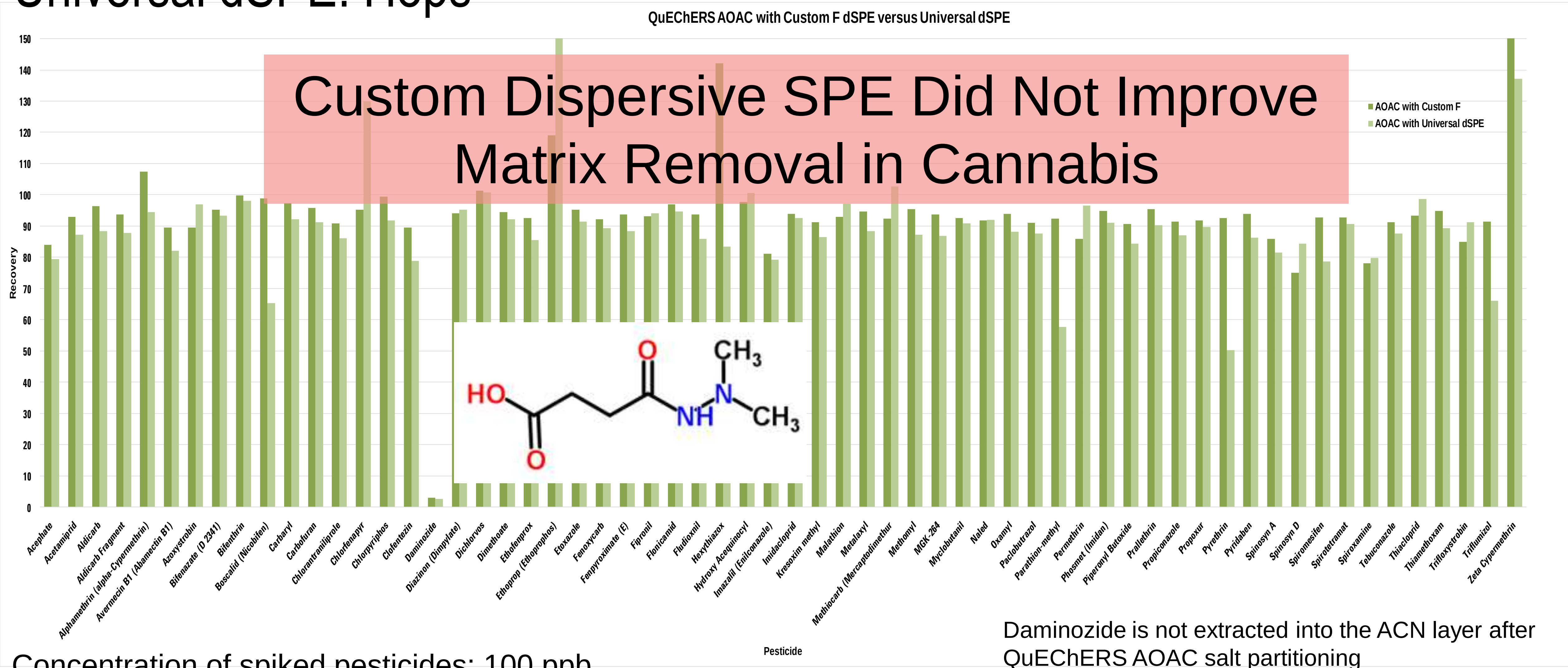

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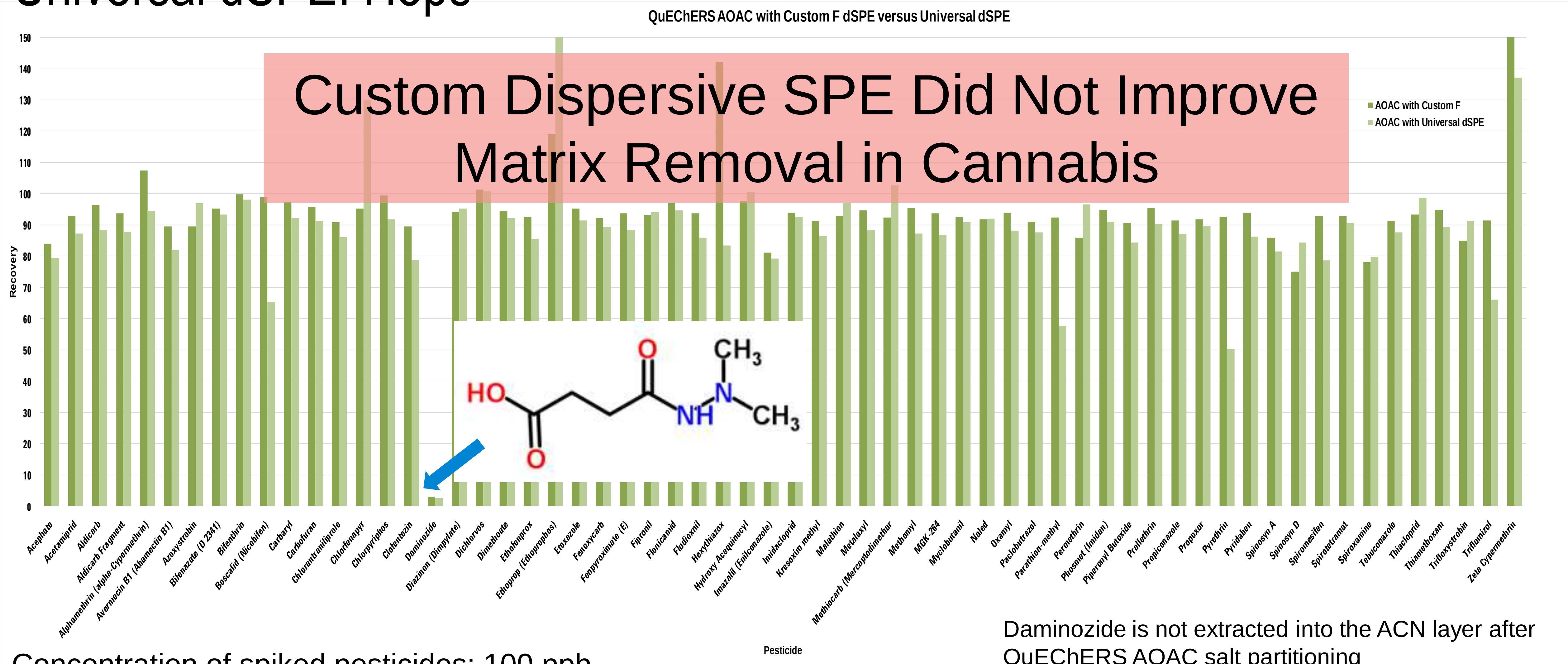

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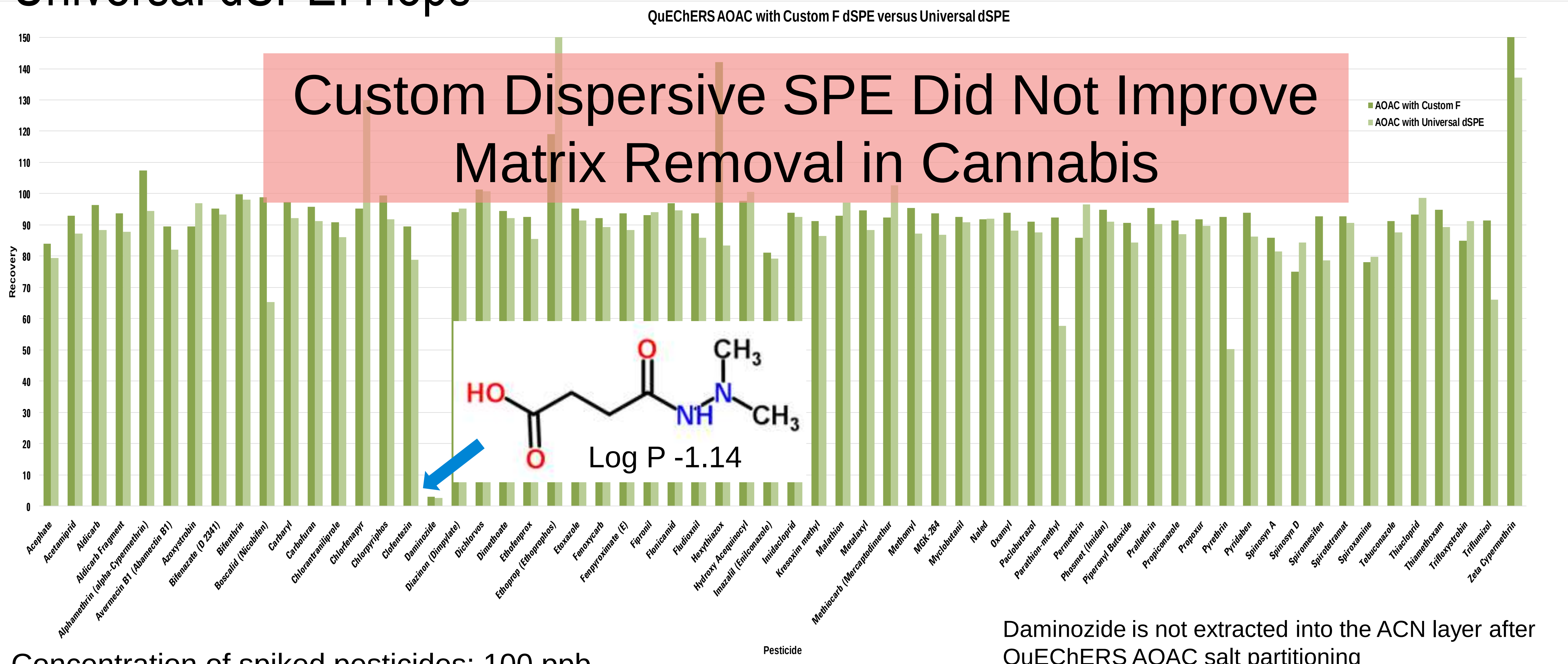

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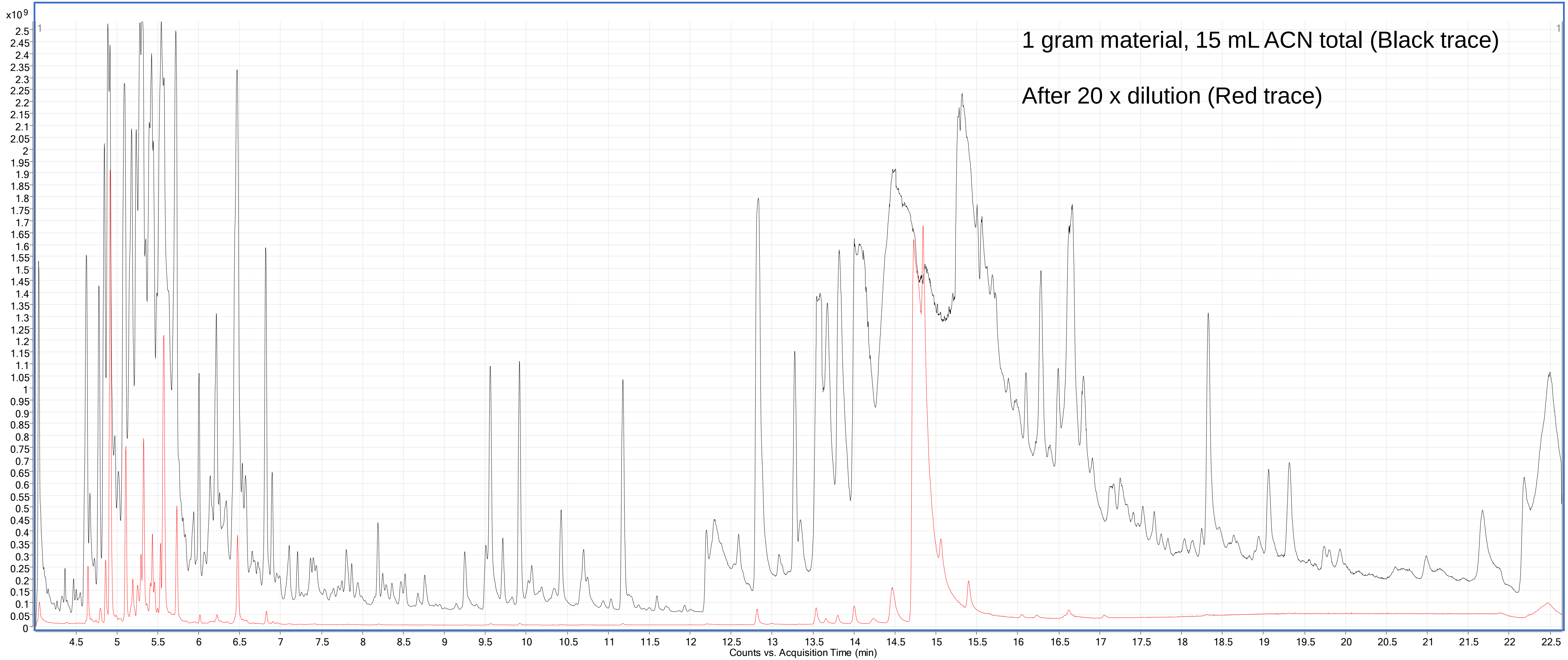
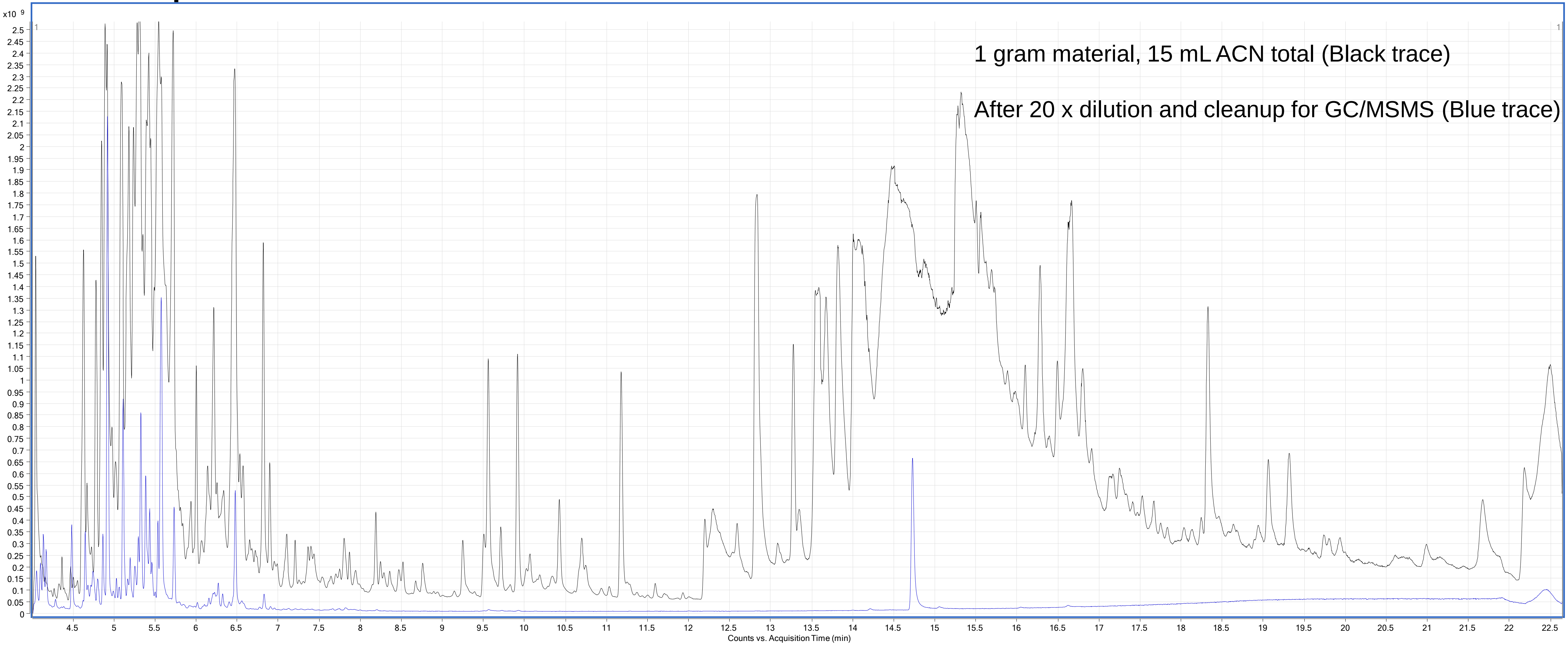
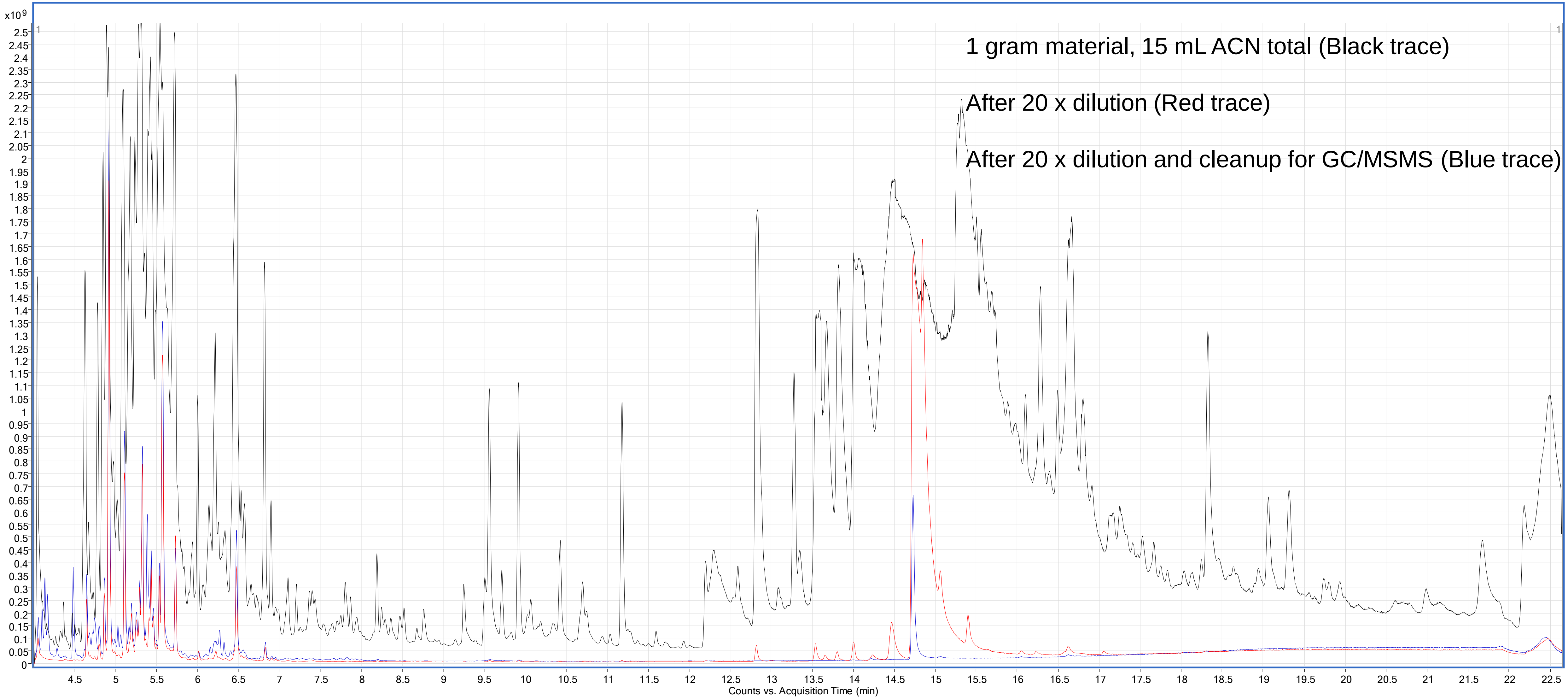
Comparison of Cannabis Extract versus Extract with 20 x Dilution Full Scan GC/MSMS



Comparison of Cannabis Extract versus Extract 20 x Dilution with Cleanup for GCMSMS



Overlay of Cannabis Extracts Full Scan GC/MSMS



Dilution better for Quantitation & Recovery?

Analyte	No dilution	1:5 Dilution	1:10 Dilution	1:20 Dilution	1:50 Dilution	1:100 Dilution
Acetamiprid	47.1 ± 3.7	88.5 ± 2.3	94.6 ± 3	99.2 ± 6.8	104.5 ± 6.4	116.7 ± 5.9
Aflatoxin B1	31.1 ± 0.9	80 ± 1.3	90 ± 3.8	92.1 ± 7.7	110.2 ± 8.8	112 ± 3.4
Buprofezin	4.1 ± 0.3	16.6 ± 1.1	32.1 ± 1.2	47.3 ± 5.8	84.6 ± 9	109.8 ± 9.9
Chlorantraniliprole	8.2 ± 0.7	31.1 ± 1.1	50.9 ± 5.2	65.5 ± 10.9	94.2 ± 10.2	101.9 ± 20.4
Lenacil	16.9 ± 1.8	57.7 ± 5.9	75.7 ± 5.4	82.4 ± 3.6	97.5 ± 9.2	106.9 ± 11.5
Methomyl	24.9 ± 1.9	51.3 ± 3.5	65.3 ± 6.5	77.1 ± 7.5	107.3 ± 10.7	113.3 ± 14.6
Profenofos	2.6 ± 0.3	12.5 ± 0.6	20.4 ± 2.1	34.2 ± 1.7	59 ± 8.3	88.5 ± 15.5
Propamocarb	105.9 ± 8	102.4 ± 3.4	101 ± 3	100.2 ± 2.8	114.5 ± 3.4	111.5 ± 6.8
Proquinazid	11.4 ± 0.6	42.8 ± 0.9	57.9 ± 1	68.3 ± 3.9	96 ± 11.6	111.3 ± 6.8
Sudan red 7B	19.8 ± 2.5	57.4 ± 4.2	61 ± 6.9	70.5 ± 2.4	84 ± 5	81.5 ± 7.2

Compound recoveries in black pepper
extremely complex matrix

Agilent 6495B: LC/MSMS

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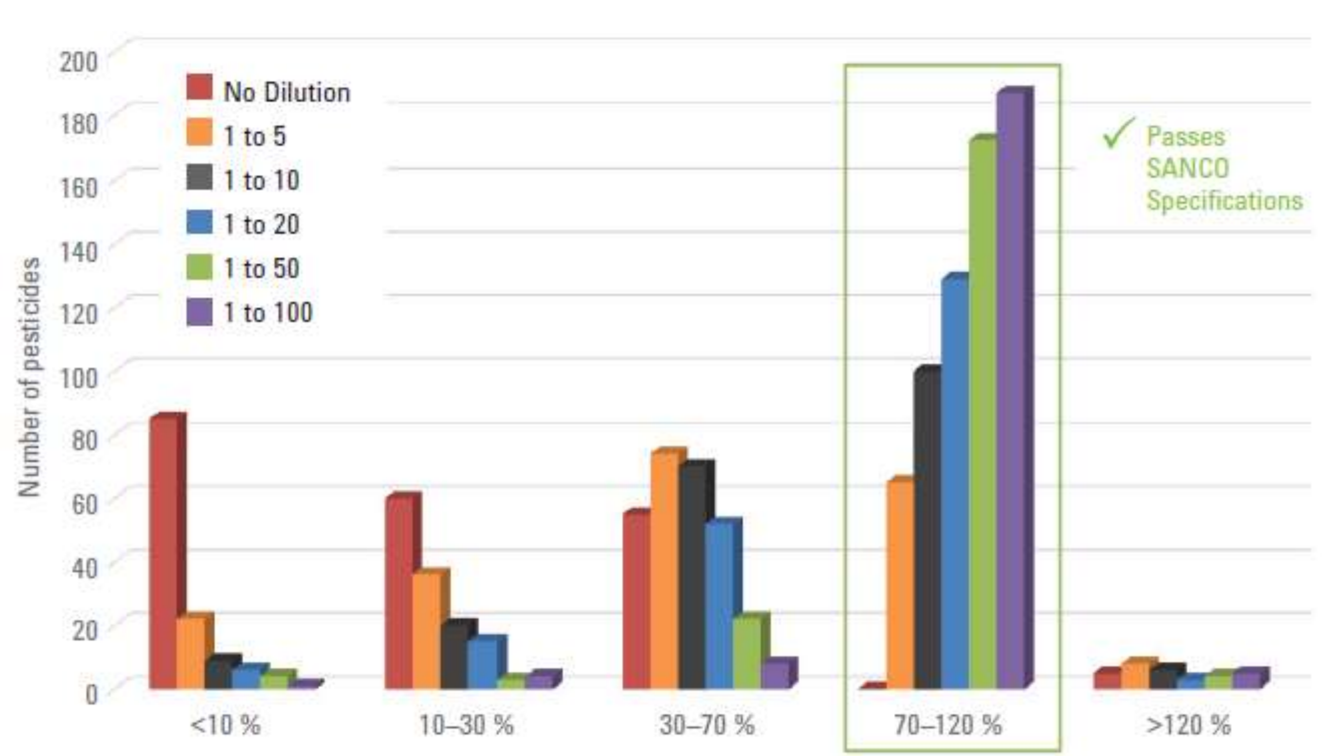


Agilent 6495B: LC/MSMS

1:100 dilution required for acceptable recoveries >90% of the compounds

Recovery of Problematic Pesticides from Cannabis Extraction with 20 x Dilution LC/MSMS

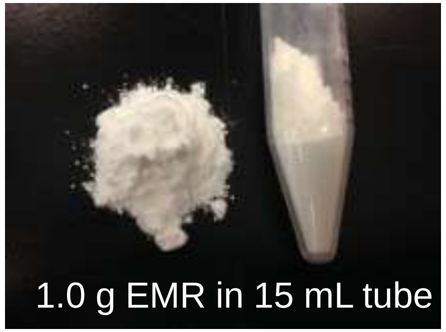
Compound	Recovery%	Average ng/mL
Abamectin	79	0.63
Acephate	86	0.69
Acequinocyl-Hydroxy	87	0.69
Azoxystrobin	88	0.70
Bifenazate	87	0.69
Daminozide	95	0.76
Hexythiazox	78	0.62
Piperonyl butoxide	89	0.72
Prallethrin	92	0.74
Pyrethrin I	88	3.50
Pyrethrin II	91	3.64
Spinosyn A	82	3.64
Spinosyn D	74	0.66
Spirotetramat	82	0.66
Spiroxamine	94	0.75

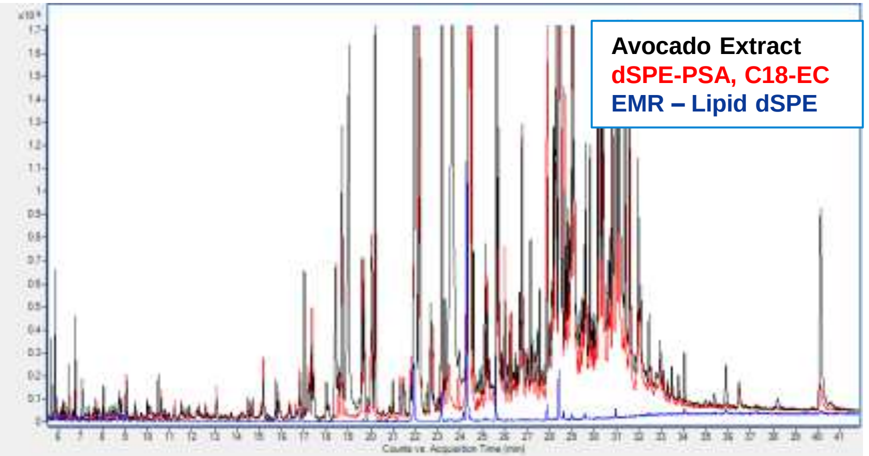
N=5

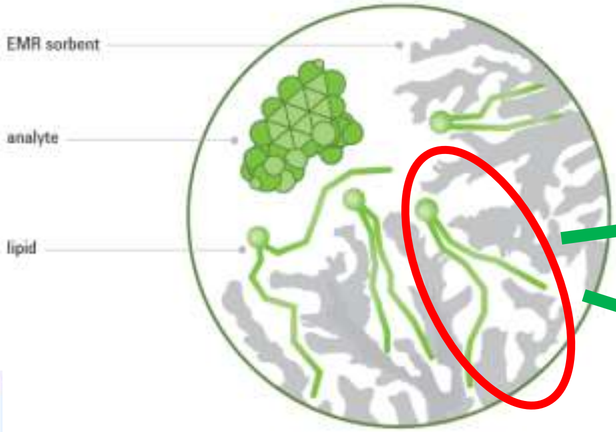
THC and CBD High Fat Products

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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Cannabis-Based Products: Unique Dispersive SPE

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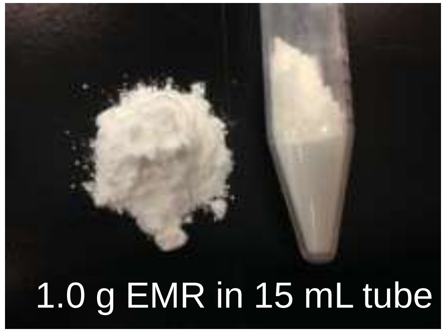

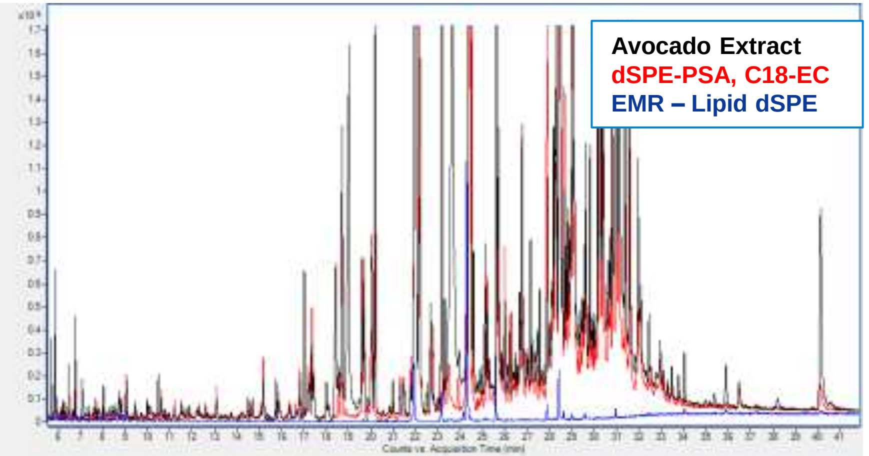

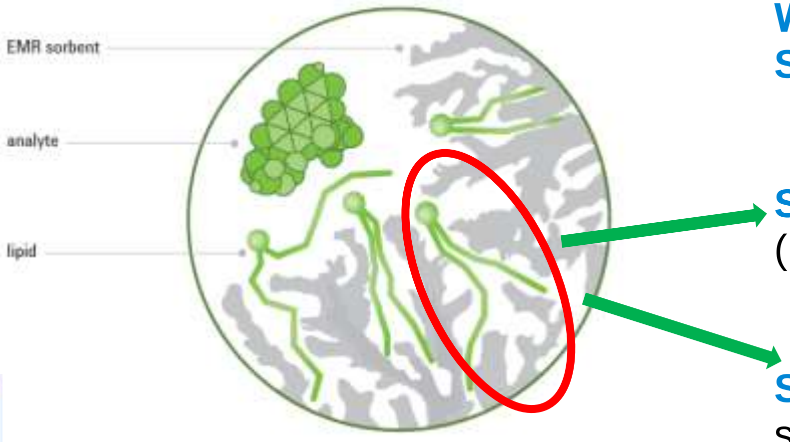

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EMR-Lipid Mechanism – Size exclusion and hydrophobic interaction

… and what does it do?

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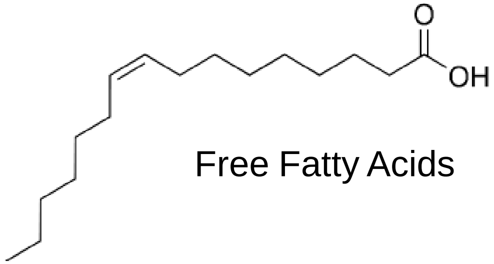
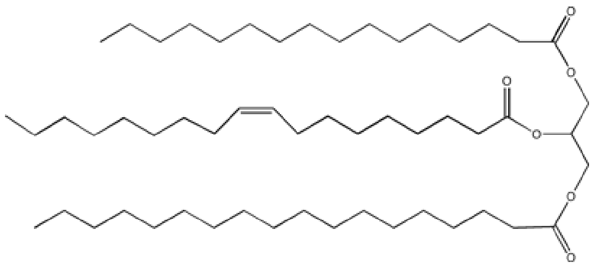
EMR sorbent removes LIPIDS it interacts with the long aliphatic chain NOT the functional groups

What are Lipids?

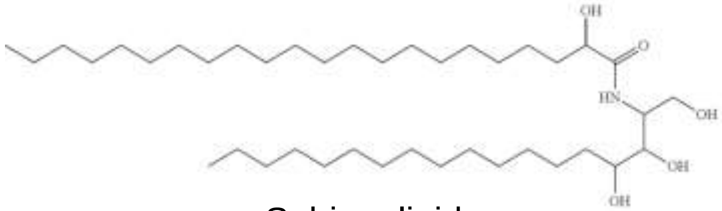
A class of naturally occurring hydrocarbon containing compounds commonly known as fats and oils



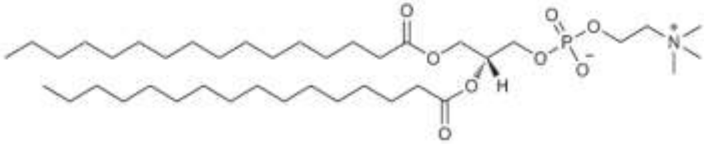
Free Fatty Acids



Triglycerides



Sphingolipids



Phospholipids

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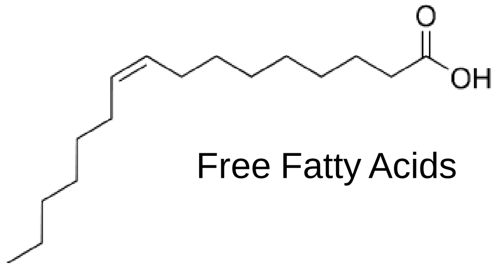

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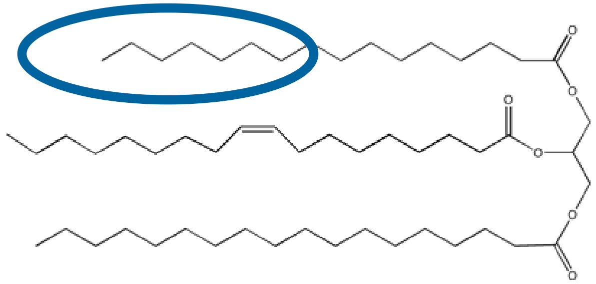

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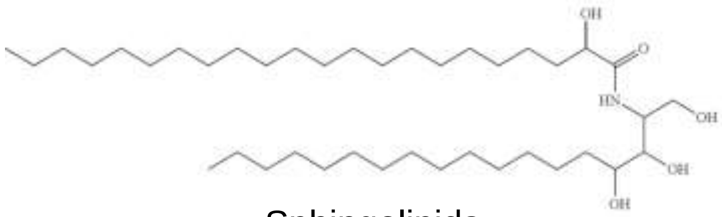

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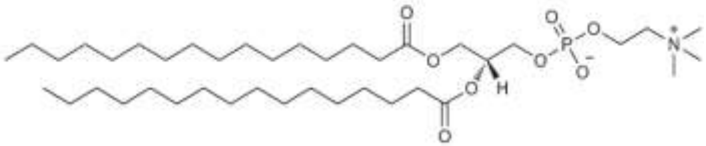
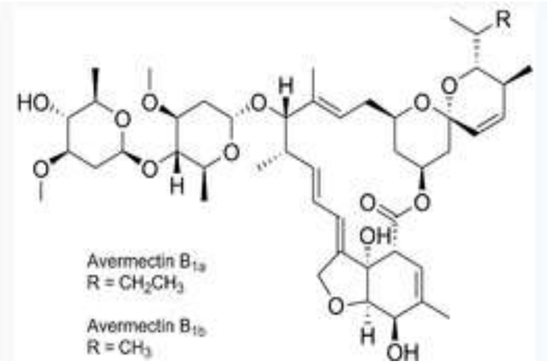


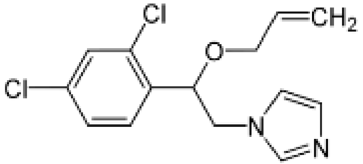
Phospholipids

What Does EMR *NOT* Interact With?

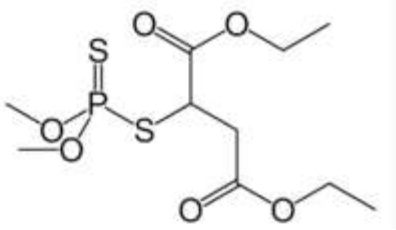
EMR does NOT remove analytes of interest



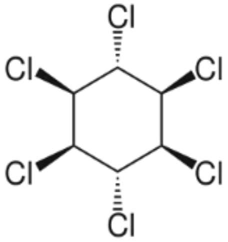
Insecticides and anthelmintics



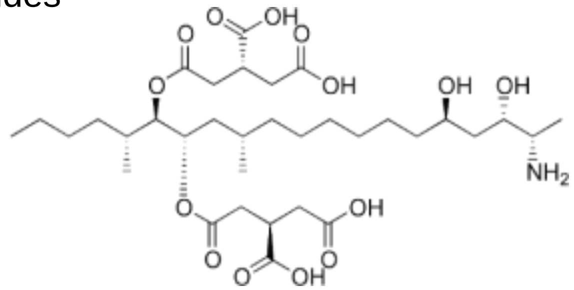
Imidazole pesticides



Organophosphate Pesticides



Organochlorine Pesticides

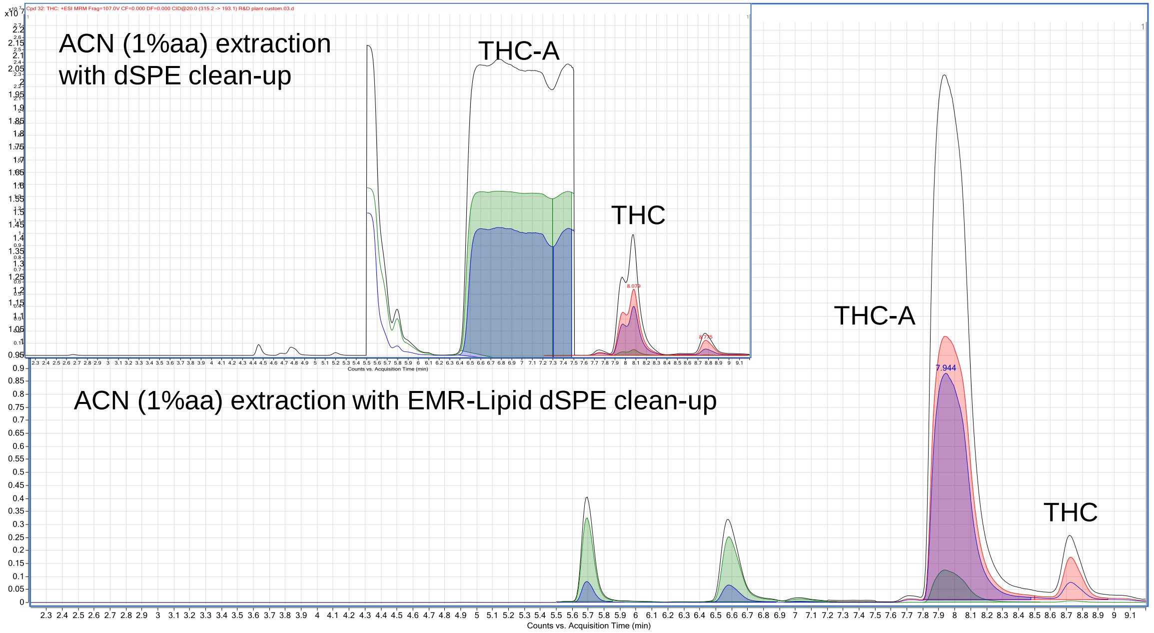


Fumonisin B2

Aflatoxins and Mycotoxins

Cannabis-Based Product: THC-Butter

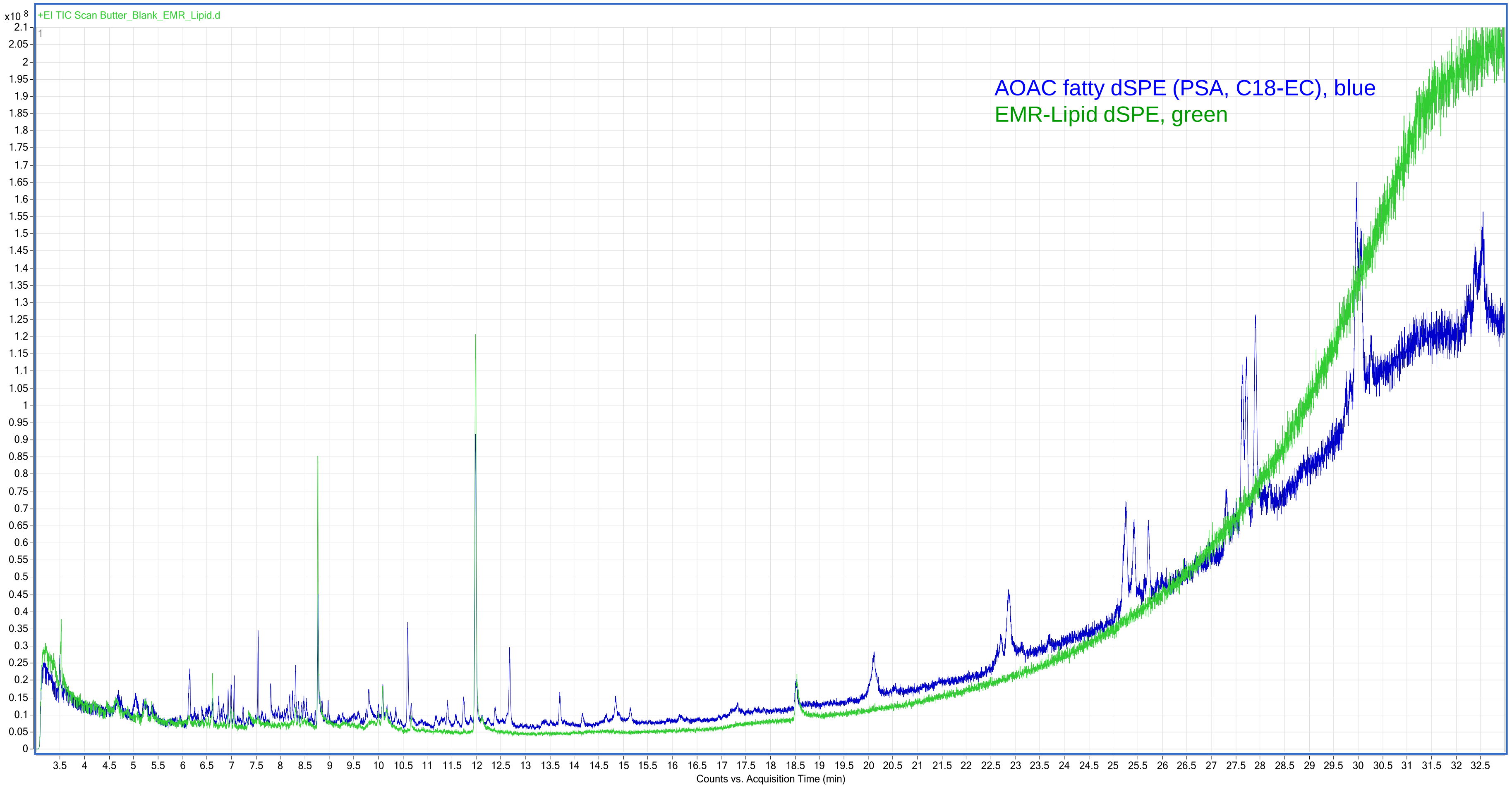
- 1.5 g of THC-butter was added to a 50 mL centrifuge tube
- Add 15 mL of ACN (1%acetic acid)
- Vortex 30 minutes, centrifuge 5000 rpm, 5 min
- Add 5 mL water to EMR-Lipid, vortex immediately 1 min
- Add 5 mL of ACN (1% acetic acid) extract, vortex immediately 2 min
- Centrifuge 5000 rpm, 5 min
- Analyze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

Extraction of Butter: MS1 Scan



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

EMR-Lipid Cartridge Formate

- SPE format with gravity flow through design
- Accommodate mLs of extract
- Very applicable to LC/MSMS work flow
- Continuing the work with high fat products for pesticide analysis
- Oils, cookies, chocolate based products
- Application work available at product release early fall

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- Dispersive SPE or SPE will remove some of the matrix co-extractives

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- Do not use QuEChERS EN method, water and ACN mix will be too basic for labile compounds based on the weaker buffering capacity of citrate salts used in the QuEChERS EN
- QuEChERS methodology: Salting/Partitioning and dSPE may negatively affect recovery; e.g. Daminozide
- Promising results with ACN (1% acetic acid) extraction, no QuEChERS salts
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- Is the existing clean-up techniques enough for long term analysis, issues with analysis and instrument maintenance, check you sample prep methodology by full scan both LC and GC/MSMS