What you don’t see…

…CAN hurt you

Sample Prep for Today’s Analytical World

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Today’s Agenda

Introduction
Addressing difficult samples
1. Polar analytes
2. High fat samples
3. How to get rid of interfering terpenes
Summary and Conclusions
Objectives of Sample Preparation

- Removal of interferences which would affect detection of analyte
- Removal of interferences that would affect instrument or column lifetime
- Concentration of an analyte to a detectable concentration
With news instruments - more Sample Preparation Techniques can be used

<table>
<thead>
<tr>
<th>Interference Removed</th>
<th>Sample Prep Technique</th>
<th>Dilute &amp; Shoot</th>
<th>Filtration</th>
<th>Liquid/Liquid Extractions</th>
<th>Supported Liquid Extractions (SLE)</th>
<th>Dried Matrix Spotting</th>
<th>Precipitation</th>
<th>QuEChERS</th>
<th>Lipid Removal 'Hybrid' Filtration</th>
<th>Solid Phase Extraction</th>
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<tbody>
<tr>
<td>Lipids</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Some</td>
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<td>Oligomeric Surfactants</td>
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<td>Suggested Agilent Product</td>
<td>Agilent Autosampler Vials</td>
<td>Captiva Syringe Filters</td>
<td>Chem Elut</td>
<td>Captiva ND</td>
<td>Bond Elut QuEChERS</td>
<td>Captiva ND LIPIDS</td>
<td>Bond Elut Silica and Polymeric SPE</td>
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</table>

*Agilent Captiva Filtration Products are recommended for use with any LC or LC-MS method*
Today’s Agenda

Introduction

Addressing difficult samples

1. Polar analytes
2. High fat samples
3. How to get rid of interfering terpenes

Summary and Conclusions
Polar analytes often require advanced solid phase extraction (SPE)

- Analyte is too polar for acetonitrile extraction
- Filtration is not clean enough
- Ion exchange can be used to filter polar interferences away
- We then often need an SPE phase that can bind a wide range of polar analytes that are basic, neutral and acidic
# Bond Elut Packed Bed Phases – Which one!

## Non-polar
- **C18**, **C8**, **C2**, **C1**
- **C18 variations** in carbon load and endcapping
- **EnvirElut**
- **CH** – cyclohexyl
- **CN-E** – endcapped cyano
- **PH** – phenyl
- **ENV, LMS, PPL, Focus, Nexus**
- **Bond Elut Plexa**

## Polar
- **PSA** - primary and secondary amine
- **NH2** - aminopropyl
- **CN-U** – unendcapped cyano
- **DEA** - diethylaminopropyl
- **Diol**
- **Si** - silica

## Cation Exchange
- **SCX** – benzenesulfonic acid
- **PRS** – propylsulfonic acid
- **CBA** – carboxylic acid

## Reversible Covalent
- **PBA** – phenylboronic acid

## Specialty Phases
- **AccuCAT**
- **Atrazine**
- **Etc..**

## Anion Exchange
- **SAX** – quaternary amine
- **PSA** – primary and secondary amine
- **NH2** – aminopropyl
- **DEA** – diethylaminopropyl

## Mixed mode IEX/NP
- **Certify** – SCX/C8
- **Certify II** – SAX/C8
- **Plexa PCX**
- **Plexa PAX**

## Alumina
- **Alumina** – aluminum oxide

## Florisil
- **Florisil** – magnesium-silica

## Carbon
- **Carbon**

## Carbon/NH2
Bond Elut Plexa - A Different Type of Polymeric Sorbent

- The hydroxylated ligands exist on the surface and in the outer regions of the pores.
- Deep inside the pore is pure S/DVB.
- There is no amide functionality (N-vinylpyrolidone) which might contribute to retaining matrix interferences.

Polar and non polar drugs bind in the SDVB hydrophobic end of the pore structure.
Plexa Recoveries Versus HLB – good results with a wide range of compounds

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Plexa</th>
<th>HLB</th>
<th>pKa</th>
<th>LogP</th>
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<tbody>
<tr>
<td>Albuterol</td>
<td>97.9</td>
<td>115.4</td>
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<td>Atenolol</td>
<td>97.0</td>
<td>94.0</td>
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<td>Loratadine</td>
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<td>Pravastatin</td>
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<tr>
<td>Propranolol</td>
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<td>35.0</td>
<td>4.9</td>
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<tr>
<td>Zolpidem</td>
<td>93.0</td>
<td>96.8</td>
<td>9.9</td>
<td>3.4</td>
</tr>
</tbody>
</table>

Plexa gives equivalent or better absolute recoveries than Oasis HLB for a wide range of pKa and logP compounds

Conditions: Absolute Recovery from human plasma
Basic load conditions, n=6, 200 ng/ml
Sorbent Conditioning - Why Do It?

Hydrophobic/hydrophilic boundary
Drying a silica-based Cartridge (ex: C18)

Over drying / lack of conditioning before sample application can contribute to

- low recoveries
- poor flow
- irreproducibility (high RSDs)
Examples on how polymeric SPE perform
Bond Elut Plexa / Phenols from Drinking Water

Efficient Preparation of Phenols in Drinking Water

Advantage Statement: Varian's Bond Elut Plexa solid phase extraction (SPE) column has a very high extraction capacity for analytes and efficient removal of contaminants, making it ideal for extraction of phenols from drinking water. Plexa features a unique structure never before seen in standard styrene divinyl benzene polymer SPE columns, whereby polarity is separated between a hydrophilic exterior and hydrophobic core (Figure 1).

Sample preparation method of phenols in drinking water using Varian Bond Elut Plexa (200 mg)

- Water sample (500 mL) adjusted with HCl to pH 2
- Bond Elut Plexa 6 mL (200 mg)
  1. Condition with 10 mL ethyl acetate
  2. Sample application 10 mL methanol
  3. Rinse with 10 mL water.
- Drying
  1. Dry for 30 or more min. via either N₂ gas or using a vacuum manifold.
- Elution
  1. Elute with 5 mL ethyl acetate.
- Removing the water
  1. Dry using a few crystals of anhydrous sodium sulfate.
- Concentration of extract
  1. 4 mL of eluted solution is gently concentrated down to 0.8 mL under N₂ gas.
- Derivatization
  1. Add 100 µL N,N-di(trimethylsilyl)trifluoroacetamide and let stand for an hour.
  2. Add 20 µL of internal standard.
  3. Make up to 1 mL with ethyl acetate.
- GC/MS/MS analysis
Method 528 provides procedures for the determination of phenols in finished drinking water. The method may be applicable to untreated source waters but has not been evaluated for these uses. The method is applicable to a variety of phenols that are efficiently partitioned from the water sample into a modified PSDB SPE sorbent (Varian’s PPL) and is sufficiently volatile and thermally stable for gas chromatography.

Extraction is performed by passing a 1L water sample through a SPE cartridge with 500mg modified PSDB (PPL). The phenols are eluted with methylene chloride.

<table>
<thead>
<tr>
<th>ANALYTE</th>
<th>CAS NUMBER</th>
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<tbody>
<tr>
<td>phenol</td>
<td>108-95-2</td>
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<tr>
<td>2-chlorophenol</td>
<td>95-57-8</td>
</tr>
<tr>
<td>2-methylphenol (o-cresol)</td>
<td>95-48-7</td>
</tr>
<tr>
<td>2-nitrophenol</td>
<td>88-75-5</td>
</tr>
<tr>
<td>2,4-dimethylphenol</td>
<td>105-67-9</td>
</tr>
<tr>
<td>2,4-dichlorophenol</td>
<td>120-83-2</td>
</tr>
<tr>
<td>4-chloro-3-methylphenol</td>
<td>59-50-7</td>
</tr>
<tr>
<td>2,4,6-trichlorophenol</td>
<td>88-06-2</td>
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<tr>
<td>2,4-dinitrophenol</td>
<td>51-28-5</td>
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<tr>
<td>4-nitrophenol</td>
<td>93951-79-2</td>
</tr>
<tr>
<td>2-methyl-4,6-dinitrophenol</td>
<td>534-52-1</td>
</tr>
<tr>
<td>pentachlorophenol</td>
<td>87-86-5</td>
</tr>
</tbody>
</table>
Chlorinated organophosphates are used as flame retardants in insulation foams, paints, coatings, plastics, and textiles while the non-chlorinated ones are mostly used as plasticizers. The compounds have been detected in air, sediment and soil, sewage sludge, streams and lakes. Particularly the chlorinated organophosphates are known to persist in the aquatic environment.

- Because of the low concentration of the analytes in the water sample an enrichment step is necessary.
**SPE Conditions**

Cartridge: Bond Elut PPL, 100 mg sorbent in 1 mL cartridge

Condition: 1 mL methanol, 1 mL methanol / acetonitrile (1/1)

Apply 1.5 - 2.5 L water sample

Dry the cartridge using nitrogen

Elution with 3 x 333 µL methanol / acetonitrile (1/1)
Results

The simple clean-up and enrichment with SPE has the advantage that up to 20 water samples can be extracted simultaneously without using complex apparatus. Bond Elut PPL has been proven to be a robust sorbent with high capacity for the extraction of polar and medium polar analytes.

Table 1. Recoveries and LODs of organophosphates; extracted from the water sample with SPE.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Recovery (%)</th>
<th>LOD (ng/L)</th>
<th>Quantifying ion m/z</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tris(1-chloro-2-propyl)-phosphate (TCP)</td>
<td>91</td>
<td>1</td>
<td>99</td>
</tr>
<tr>
<td>Tris(2-chloroethyl)-phosphate (TCEP)</td>
<td>95</td>
<td>2</td>
<td>63</td>
</tr>
<tr>
<td>Tris(1,3-dichloro-2-propyl)-phosphate (TDCP)</td>
<td>99</td>
<td>1</td>
<td>75</td>
</tr>
<tr>
<td>Tri-n-butylphosphate (TnBP)</td>
<td>89</td>
<td>1</td>
<td>99</td>
</tr>
<tr>
<td>Tri-isobutylphosphate (TiBP)</td>
<td>85</td>
<td>2</td>
<td>99</td>
</tr>
<tr>
<td>Tris(2-butoxyethyl)-phosphate (TBEP)</td>
<td>93</td>
<td>3</td>
<td>125</td>
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</tbody>
</table>
Conclusion

Novel polymeric SPE phases are recommended:

1. They have double the binding capacity over silica
2. They can bind a wide range of polar analytes
3. They don’t decondition/dry out on the SPE manifold
4. They provide more robust methods
Today’s Agenda

Introduction
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Summary and Conclusions
Remove interferences...

- To acquire desired sensitivity/selectivity
- To reduce contamination/carryover issues
- Use of sensitive and expensive instruments: *Protect your investment!!!*

Pesticides in Avocado *without* SP

Pesticides in Avocado with SP
Lipids
Matrices and Approximate Total Lipid Content

- **Low (<2%)**
  - Spinach (0%)
  - Strawberry (0%)
  - Onion (0%)
  - Paprika (1%)
  - Cumin (2%)
  - Rice (2%)
  - Hops (2%)
  - Tilapia (2%)
  - Sea Bass (3%)
  - Wheat (3%)

- **Med (4-12%)**
  - Soy Milk (4%)
  - Beef Liver (4%)
  - Pork Liver (4%)
  - Corn (4%)
  - Chocolate (8%)
  - Canned Pet Food (~10%)
  - Cow’s Milk (5%)

- **High (>12%)**
  - Nut Butters (16%)
  - Avocado (21%)
  - Pork (8%)
  - Trout (8%)
  - Soy Oil (100%)
  - Avocado Oil (100%)

- **Plasma or whole blood (10 – 20 %)**
# Current Procedures for Lipid Removal

<table>
<thead>
<tr>
<th>Method</th>
<th>How Lipids are removed</th>
<th>Weakness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dilute and Shoot</td>
<td>No lipid removal, only dilution</td>
<td>No lipid removal</td>
</tr>
<tr>
<td>Protein Precipitation</td>
<td>PPT followed by centrifugation</td>
<td>Insufficient lipid removal</td>
</tr>
<tr>
<td></td>
<td>PPT followed by filtration with or without sorbent</td>
<td>Insufficient lipid removal; low analyte recovery</td>
</tr>
<tr>
<td>QuEChERS</td>
<td>PSA/C18 sorbent (dSPE)</td>
<td>Not selective; insufficient lipid removal; analyte loss</td>
</tr>
<tr>
<td></td>
<td>Zr-containing sorbent</td>
<td>Low total lipid capacity; analyte recovery</td>
</tr>
<tr>
<td></td>
<td>Freeze sample</td>
<td>Time needed; loss of analyte</td>
</tr>
<tr>
<td>SPE/SLE</td>
<td>Load and elute</td>
<td>Time needed; solvent usage; extensive method development</td>
</tr>
<tr>
<td>SEC/GPC</td>
<td>Chromatographic separation</td>
<td>Uses copious amounts of solvent and time; capital expense</td>
</tr>
</tbody>
</table>
EMR-Lipid
Enhanced Matrix Removal

EMR: As easy to use as QuEChERS; as clean as SPE
EMR Product offering

EMR fits into current sample preparation workflows

EMR-Lipid (p/n 5982-1010)

EMR-Polish (p/n 5982-0102)

Extraction Tube
EMR Sorbent - What is it?

When “activated” by water…

- The materials selective hydrophobic interactions increase.
- Suspension of nano particles (high surface area).
- Rapidly interacts with straight chain, “lipid-like” functional groups.

Centrifugation preferably used to separate precipitate from solution (not filtration).

EMR-Lipid Mechanism – Size exclusion and hydrophobic interaction.
... and what does it do?

**EMR sorbent removes Lipids**

What are Lipids?

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

- Free Fatty Acids
- Triglycerides
- Cholesterol
- Phospholipids
What Does EMR *NOT* Interact With?

EMR does *NOT* remove analytes of interest

Exceptions?

Compounds containing long aliphatic functional groups (e.g. prostaglandins)

- Fluoroquinolones
- Organochlorine Pesticides
- Tetracyclines
- PAHs
- Imidazole pesticides
- Vitamin D
- Prostaglandins
- Fluoroquinolones
- Tetracyclines
- PAHs
- Imidazole pesticides
- Vitamin D
- Prostaglandins
EMR Fits into Existing Workflows

QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe)

- Easy-to-use sample preparation for food testing, solid samples (e.g. vegetables, fruits, meat, seafood, etc.)

  **EMR Applications:** - Pesticide Residues in Avocado,

Modified Liquid Extraction (Protein Precipitation)

- Proteins are removed by a “crash” step prior to injection or cleanup (e.g. milk, meat, seafood, etc.)

  **EMR Applications:** - PAHs in Salmon,  
  - Veterinary Drugs in Bovine Liver
Improving dSPE in QuEChERS

1. Extraction
   - Pros
     • Fast and inexpensive
     • Takes minimal experience
     • Doesn’t require special equipment
     • Accommodates multiple matrices
     • Accommodates large analyte groups
   - Cons
     • Large amount of coextractives

2. Dispersive SPE
   - Pros
     • Same as extraction
   - Cons
     • Minimal cleanup provided
     • Can remove analytes
     • Lipids are challenging to remove selectively
EMR – Lipid – dSPE Cleanup

A. QuEChERS or Liquid Extract
B. Add H₂O to EMR tube (“activation”)
C. Transfer extract
D. Vortex and centrifuge
E. Supernatant (1:1; extract: H₂O)
A. Add supernatant to EMR – Polish tube
B. Vortex immediately
C. Phase separation after centrifuge
D. Transfer upper layer for analysis
E. Final samples split for GC and LC analysis
F. Extra dry step recommended prior to GC
EMR Workflow

Extract  dSPE  Polish  Dry

In acetonitrile
Or acetone
GC-MS Fullscan Avocado

Avocado – Untreated
GC-MS Fullscan Avocado

Avocado – Untreated
Avocado – EMR Treated
GC-MS Fullscan Pet Food

Pet Food – Untreated
GC-MS Fullscan Pet Food

Pet Food – Untreated
Pet Food – EMR Treated
GC-MS Full Scan– Avocado Oil

Avocado Oil Extract
EMR – Lipid Extract
GC-MS Spinach Fullscan

Abundance

Time-->
GC-MS Red Pepper Fullscan

Red Pepper matrix blank w/ Q-Universal dSPE cleanup

Red Pepper matrix blank w/ Q-General dSPE cleanup

Red Pepper matrix blank w/o cleanup

Red Pepper matrix blank w/ EMR cleanup

Agilent Technologies
Benefits to Instrumental Flowpath
Comparison of Analytes Response Consistency over Multiple Avocado Sample Injections

TPP Peak Area over 100 Injections

- EMR cleanup
- Z-Sep+ cleanup
- C18 dSPE cleanup

TPP
Commonly used IS
RT: 18.3 min

Response drops ~ 4% after 100 avocado sample injections
Response drops ~ 50% after 100 avocado sample injections
Response increases ~ 35% after 100 avocado sample injections
Analytes Responses Reproducibility on GC/MS/MS over 100 Injections of Avocado Samples

<table>
<thead>
<tr>
<th>Pesticides</th>
<th>EMR Cleanup</th>
<th>C18/PSA cleanup</th>
<th>Zirconia sorbent cleanup</th>
<th>EMR over 50 injections</th>
<th>C18/PSA cleanup</th>
<th>Zirconia sorbent cleanup</th>
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<td>Dichlorvos</td>
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MS Source Critical Tuning Parameters

MS (GC/MS) Performance Monitoring over Multiple Avocado Sample Injections

- **Relative Abundance for Ion 219**, Minimum of 70 needed
- **Relative Abundance for Ion 502**, minimum of 5 needed
- **EM voltage**, higher than 1400 indicated needs to replace

Injections of Avocado Samples with EMR cleanup on GC/MS

NACRW 2015
# 67 Pesticides Analysis in Avocado by LC and GC-QQQ

<table>
<thead>
<tr>
<th>Representative Pesticide</th>
<th>Chemical Class</th>
<th>Pesticide Group</th>
<th>Detection Technique</th>
<th>Representative Pesticide</th>
<th>Chemical Class</th>
<th>Pesticide Group</th>
<th>Detection Technique</th>
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<th>Chemical Class</th>
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</tbody>
</table>
QuEChERS-EMR Protocol for Multi-residue Analysis of Pesticides in Avocado

1. Accurately weigh 15 g comminuted Avocado sample in 50 mL centrifuge tube.
2. Add 15 mL of 1% Acetic Acid in Acetonitrile, and AOAC QuEChERS extraction kit.
3. Cap and shake vigorously on mechanical shaker for 2 min.
4. Centrifuge @ 5000 rpm for 5 min.
5. Transfer 5 mL of upper ACN extract and 5 mL of water to EMR dSPE 15 mL tubes.
6. Add 15 mL of 1% Acetic Acid in Acetonitrile, and AOAC QuEChERS extraction kit.
7. Cap and shake vigorously on mechanical shaker for 2 min.
8. Vortex and Centrifuge.
9. Transfer 5 mL supernatant to empty tube, add content of Polish Pouch.
10. Vortex, centrifuge, and transfer the upper ACN layer to another vial.
11. Combine 200 µL of ACN extract and 800 µL water, vortex, and centrifuge if needed.

Samples are ready for GC-QQQ analysis after dry step. Samples are ready for LC-QQQ analysis.
## Comparison of Avocado Co-extractives by Weight

<table>
<thead>
<tr>
<th>Cleanup</th>
<th>Amount of co-extractives (mg, n= 3)</th>
<th>Amount of co-extractives removed by cleanup (mg, n = 3)</th>
<th>% of matrix co-extractives removed by cleanup</th>
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<tbody>
<tr>
<td>No further cleanup</td>
<td>14.7</td>
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<tr>
<td>C18/PSA Cleanup</td>
<td>9.5</td>
<td>5.2</td>
<td>35.4</td>
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<td>EMR-Lipid Cleanup</td>
<td>4.2</td>
<td>10.5</td>
<td>71.4</td>
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<tr>
<td>Zirconia sorbent Cleanup</td>
<td>7.0</td>
<td>7.7</td>
<td>52.4</td>
</tr>
</tbody>
</table>

% Matrix Co-extractives Removed by Cleanup = \[
\frac{\text{Amount of Co-extractives Removed after Cleanup}}{\text{Amount of Co-extractives without Cleanup}} \times 100%
\]

The use of EMR material cleanup removes extra 20-30% of Avocado co-extractives in comparison to traditional QuEChERS and/or competitor’s cleanup.
The use of EMR material cleanup provides significantly cleanup chromatographic sample background.
Comparison of GC/MS/MS MRM Chromatogram for Matrix Background

Counts vs. Acquisition Time (min)

- Matrix blank w/ C18/PSA cleanup
- Matrix blank w/ zirconia sorbent cleanup
- Matrix blank w/ EMR cleanup
# Chromatographic Benefits of Matrix Removal Provided by EMR Cleanup

<table>
<thead>
<tr>
<th>Benefit</th>
<th>Example</th>
<th>EMR-Lipid cleanup</th>
<th>Zirconia sorbent cleanup</th>
<th>C18/PSA cleanup</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduced matrix suppression</td>
<td>EPN in Avocado on LC-QQQ</td>
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<tr>
<td>Improved S/N ratio</td>
<td>Captan in Avocado on GC-QQQ</td>
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<td></td>
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<tr>
<td>Less interferences for accurate integration</td>
<td>Permethrin in Avocado on GC-QQQ</td>
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</tbody>
</table>

**Example**

- **EMR-Lipid cleanup**
  - EPN in Avocado on LC-QQQ
  - Captan in Avocado on GC-QQQ
  - Permethrin in Avocado on GC-QQQ

**Zirconia sorbent cleanup**

**C18/PSA cleanup**
Selected Problematic Pesticides for Recovery Comparison

Selected Pesticides Recovery Comparison from Multi-residue Pesticides Analysis in Avocado by LC/QQQ or GC/QQQ

- EMR cleanup
- C18/PSA cleanup
- Zirconia sorbent cleanup

Analytes Recovery % (n=6) in 50ppb pesticides fortified Avocado samples
Statistical Recovery Results Comparison

Statistical Recovery Results for Multi-residue Pesticides (67) Analysis in Avocado by LC/QQQ and GC/QQQ

- EMR-Lipid cleanup
- C18/PSA cleanup
- Zirconia sorbent cleanup

- REC < 70%
- REC in 70-120%
- REC > 120%
Method Accuracy and Precision

Analysis of Pesticides in Avocado by LC/MS/MS & GC/MS/MS -- Overall Accuracy and Precision

Error bar = 95% CI

Overall Analyte Accuracy Results for QC’s Spiked at 5 ng/g, 50 ng/g, and 150/300 ng/g in Avocado (n = 18)
Summary and Conclusion

- EMR-Lipid provides the most complete lipid removal of any sorbent on the market.
- Achieve SPE cleanliness with dSPE simplicity.
- EMR is a one size fits all sorbent for a variety of sample types and analytes.
- Key applications were validated with EMR and demonstrate better recovery, better precision, and decreased matrix impact to the instrument and results.
Today’s Agenda

Introduction

Addressing difficult samples
1. Polar analytes
2. High fat samples
3. How to get rid of interfering terpenes

Summary and Conclusions
Constituents of Cannabis and Hop: 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 (86),
- Vitamins (1) [Vitamin A],
- Pigments (2),
- Elements (9).
QuEChERS AOAC Extraction/Partitioning with Custom dSPE versus Universal dSPE: Hops

GC/MSMS MS1 Scan

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

GC/MSMS MS1 Scan
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.
Conclusion

Addressing difficult samples
1. Polar analytes: Explore new polymeric SPE phases
2. High fat samples: Consider EMR Lipids as a dSPE
3. How to get rid of interfering terpenes: stay tuned 😊
Thank You!