# Applications of Fused-Core®, Superficially Porous Particles (SPP) in Environmental Analysis

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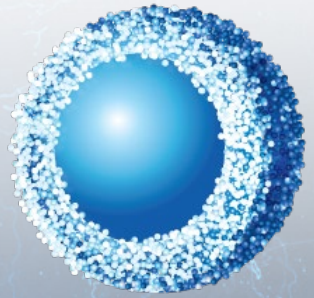
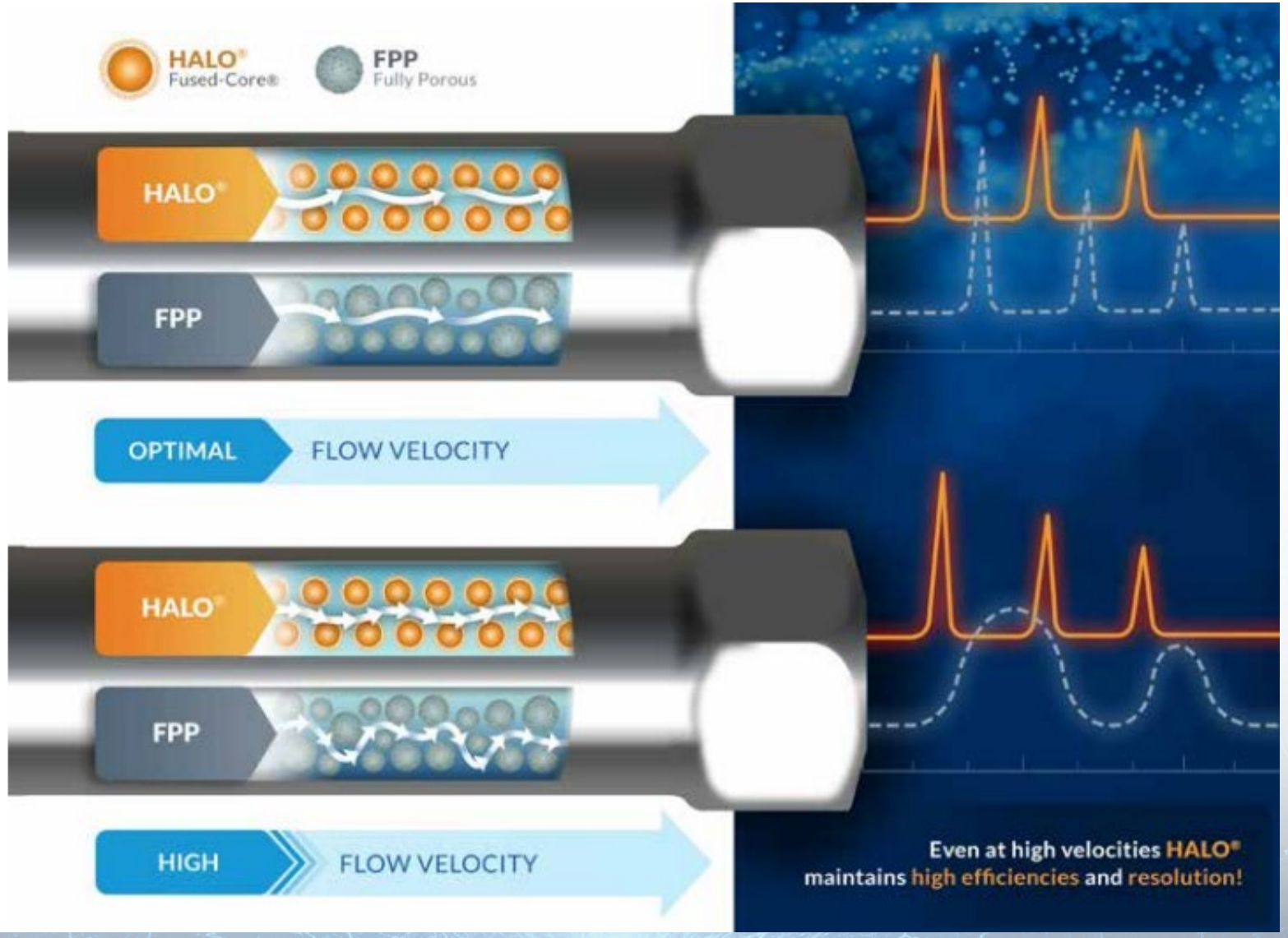


# Outline

- How SPP works
- What are the advantages of using Fused-Core® particle technology
- Versatile columns available for application development

# How SPP Works



# SPP Benefits

- The benefits of superficially porous particles (SPPs) include highly efficient separations in less time and with reduced backpressure requirements.

- In the applied/small molecule market throughput is the one of the most important factors in method development.

# Higher Overall Efficiency (Maximum N)

- Comparison of different columns and maximum plates (theoretical calculations for small molecule at 50% ACN/water)

| Column type | Length (mm) | Flow rate (mL/min) | Plates (N) | Pressure (bar) |
|---|---|---|---|---|
| 5 µm FPP | 150 | 0.6 | 14,600 | 100 |
| 3 µm FPP | 150 | 0.6 | 24,200 | 309 |
| 1.8 µm FPP | 150 | 0.6 | 30,840 | 771 |
| 5 µm SPP | 150 | 0.6 | 28,300 | 78 |
| 2.7 µm SPP | 150 | 0.6 | 38,300 | 284 |

# Applications

- High throughput-Environmental labs, drug screening labs-Screening methods for drugs and mycotoxins. PFP (Pentafluorophenylpropyl) and Biphenyl phase used. Multiple isobaric species

- Triglyceride profiling-Food and beverage industry-analysis of edible oils-C30 phase used. Multiple isobaric species

# Mycotoxins

- Produced by mold actively growing on crop, or can also begin growing during crop storage
- Toxic-crop destruction
- Heat stable to canning
- Danger to humans and animals (feed crops)- secondary metabolites of fungus
- HALO® PFP

TEST CONDITIONS:

Columns: HALO® 90 Å PFP, 2 µm, 2.1 x 50mm
Part Number: 91812-409

Mobile Phase A: Water/2mM ammonium formate/0.1% Formic acid

Mobile Phase B: Methanol/2mM ammonium formate/0.1% Formic acid

Gradient:

| Time | %B |
|---|---|
| 0.01 | 15 |
| 1.0 | 25 |
| 2.0 | 40 |
| 2.50 | 41 |
| 4.50 | 100 |
| 5.50 | 100 |
| 5.51 | 15 |
| 6.50 | Finished |

Flow Rate: 0.4 mL/min

Initial Pressure: 485 bar

Temperature: 40 ° C

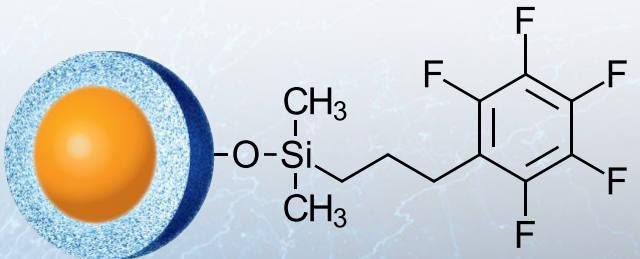
Injection Volume: 1 µL

Sample Solvent: 95/5 water/methanol

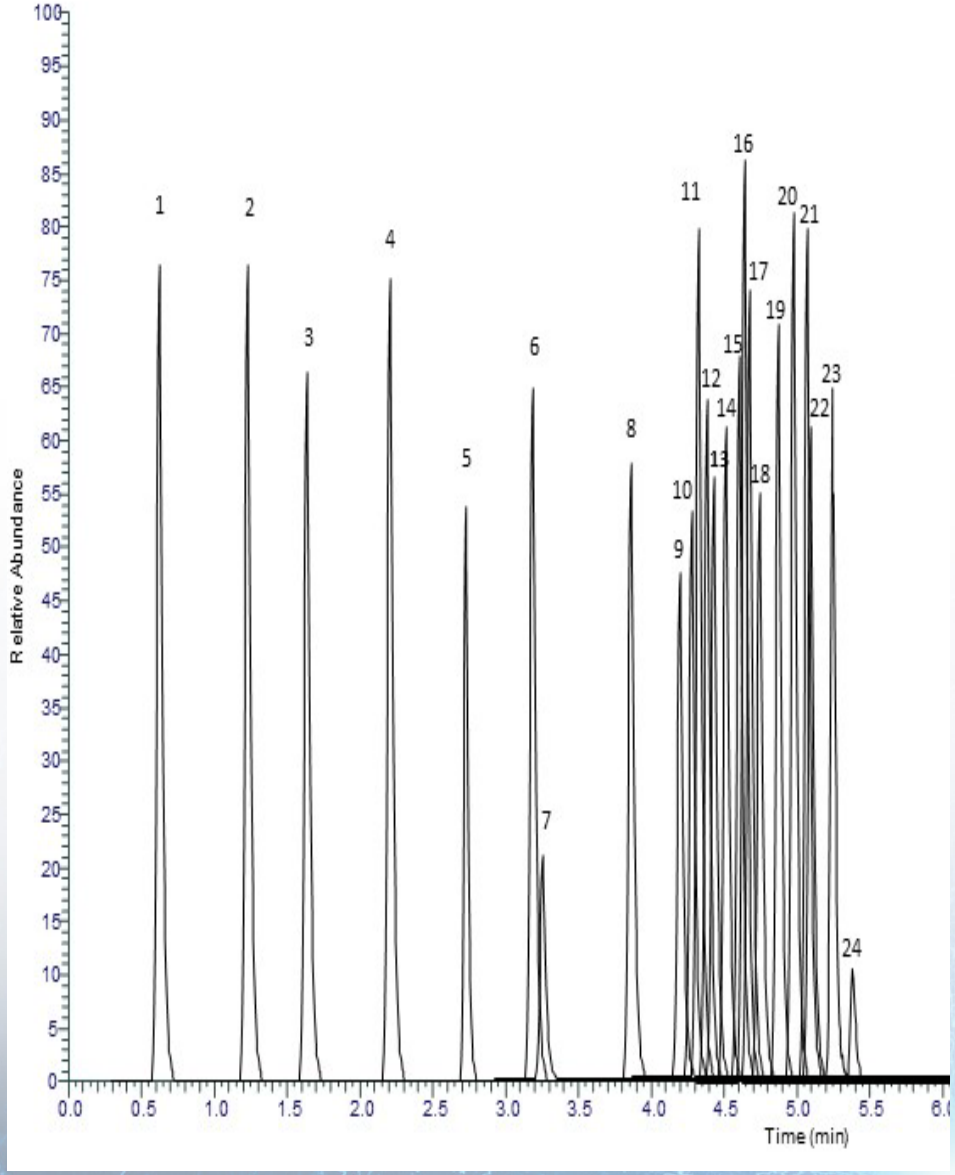
LC System: Shimadzu Nexera X2

Detection: +ESI MS/MS

MS: Orbitrap Exactive mass spectrometer



# Mycotoxins




| Peak Number | Compound | Retention Time | Precursor Ion | Product Ion |
|---|---|---|---|---|
| 1 | Nivalenol | 0.71 | 313.1235 | 175.10 |
| 2 | Deoxynivalenol | 1.38 | 297.1335 | 249.09 |
| 3 | Deoxynivalenol-3-glucoside | 1.70 | 459.1850 | 193.10 |
| 4 | Fusarenon X | 2.37 | 355.1387 | 247.10 |
| 5 | Neosolaniol | 2.87 | 383.1702 | 365.16 |
| 6 | 15-Acetyldeoxynivalenol | 3.33 | 339.1378 | 321.15 |
| 7 | 3-Acetyldeoxynivalenol | 3.36 | 339.1378 | 231.15 |
| 8 | Gliotoxin | 3.97 | 327.0436 | 196.08 |
| 9 | Aflatoxin G2 | 4.27 | 331.0759 | 312.97 |
| 10 | Aflatoxin M1 | 4.39 | 329.0604 | 273.12 |
| 11 | Aflatoxin G1 | 4.40 | 329.0601 | 242.90 |
| 12 | Aflatoxin B2 | 4.44 | 315.0820 | 284.87 |
| 13 | HT-2 + Na | 4.47 | 447.1934 | 345.10 |
| 14 | Diacetoxyscirpenol | 4.49 | 367.2637 | 307.15 |
| 15 | Aflatoxin B1 | 4.52 | 313.0662 | 286.99 |
| 16 | Ochratoxin A | 4.67 | 404.0855 | 238.99 |
| 17 | T-2 +Na | 4.72 | 489.2049 | 245.09 |
| 18 | Ochratoxin B | 4.88 | 370.1321 | 324.15 |
| 19 | Citrinin | 4.96 | 251.0860 | 233.09 |
| 20 | Zearalenone | 5.11 | 319.1491 | 283.08 |
| 21 | Patulin +MEOH | 5.11 | 187.0723 | 98.95 |
| 22 | Fumonisin B1 | 5.24 | 722.3868 | 334.25 |
| 23 | Fumonisin B3 | 5.41 | 706.3901 | 336.25 |
| 24 | Fumonisin B2 | 5.44 | 704.3901 | 336.25 |

# Mycotoxin Isomeric resolution

| Peak Number | Compound | Retention Time | Precursor Ion | Product Ion |
|---|---|---|---|---|
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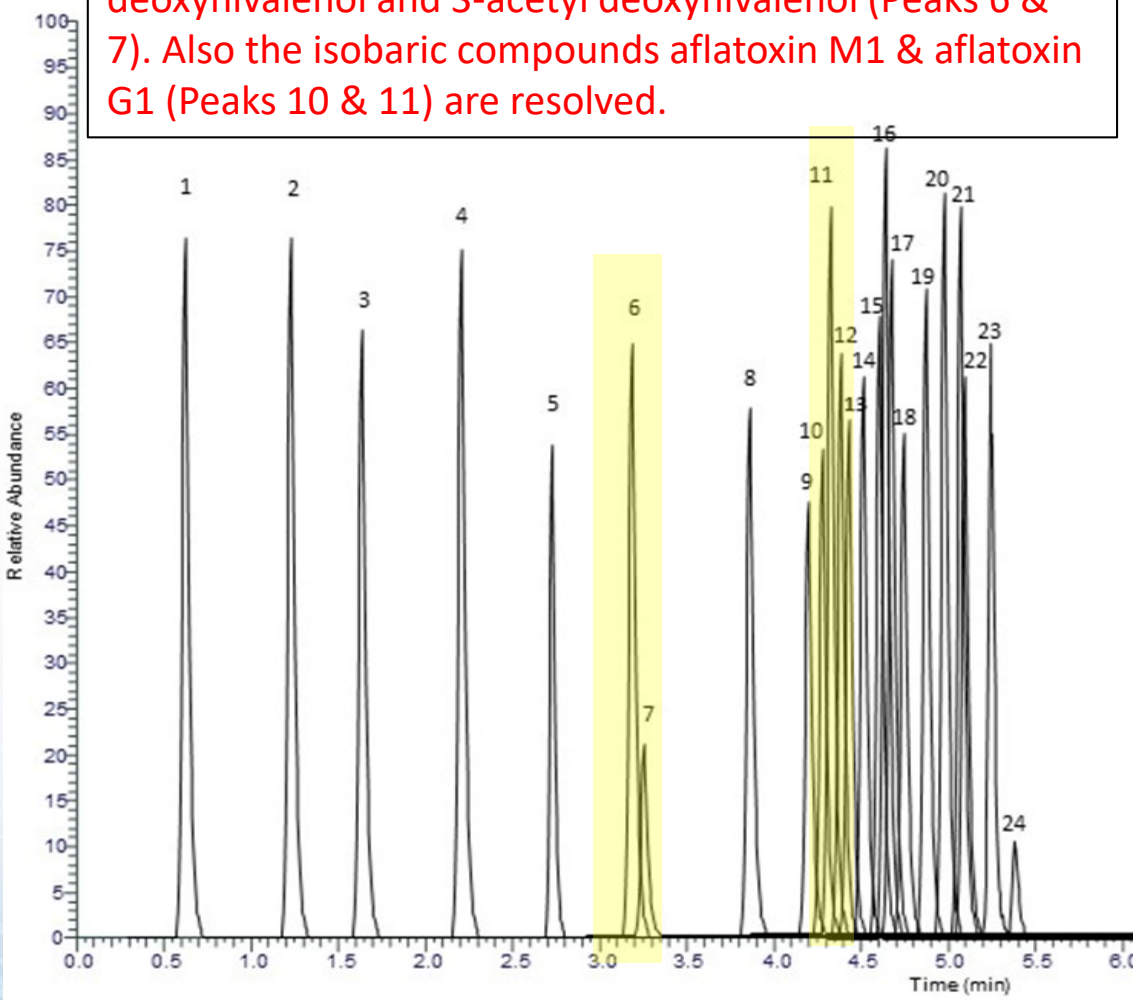
Notice the resolution of the isomers, 15-acetyl deoxynivalenol and 3-acetyl deoxynivalenol (Peaks 6 & 7). Also the isobaric compounds aflatoxin M1 & aflatoxin G1 (Peaks 10 & 11) are resolved.



# SAMHSA-5 Panel

- SAMHSA-The Substance Abuse and Mental Health Services Administration

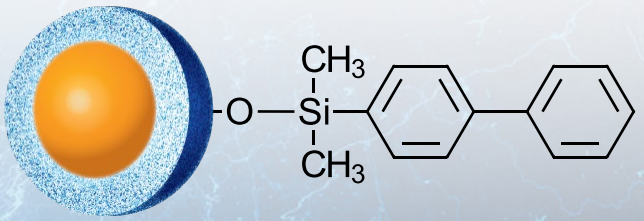
- Amphetamines (Amphetamines, Methamphetamines, Speed)
- Cocaine (Cocaine, Crack, Benzoylecognine)
- Marijuana – THC Tetrahydrocannabinol (Cannabinoids, Hash)
- Opiates (Heroin, Opium, Codeine, Morphine)
- Phencyclidine (PCP)

- HALO® Biphenyl

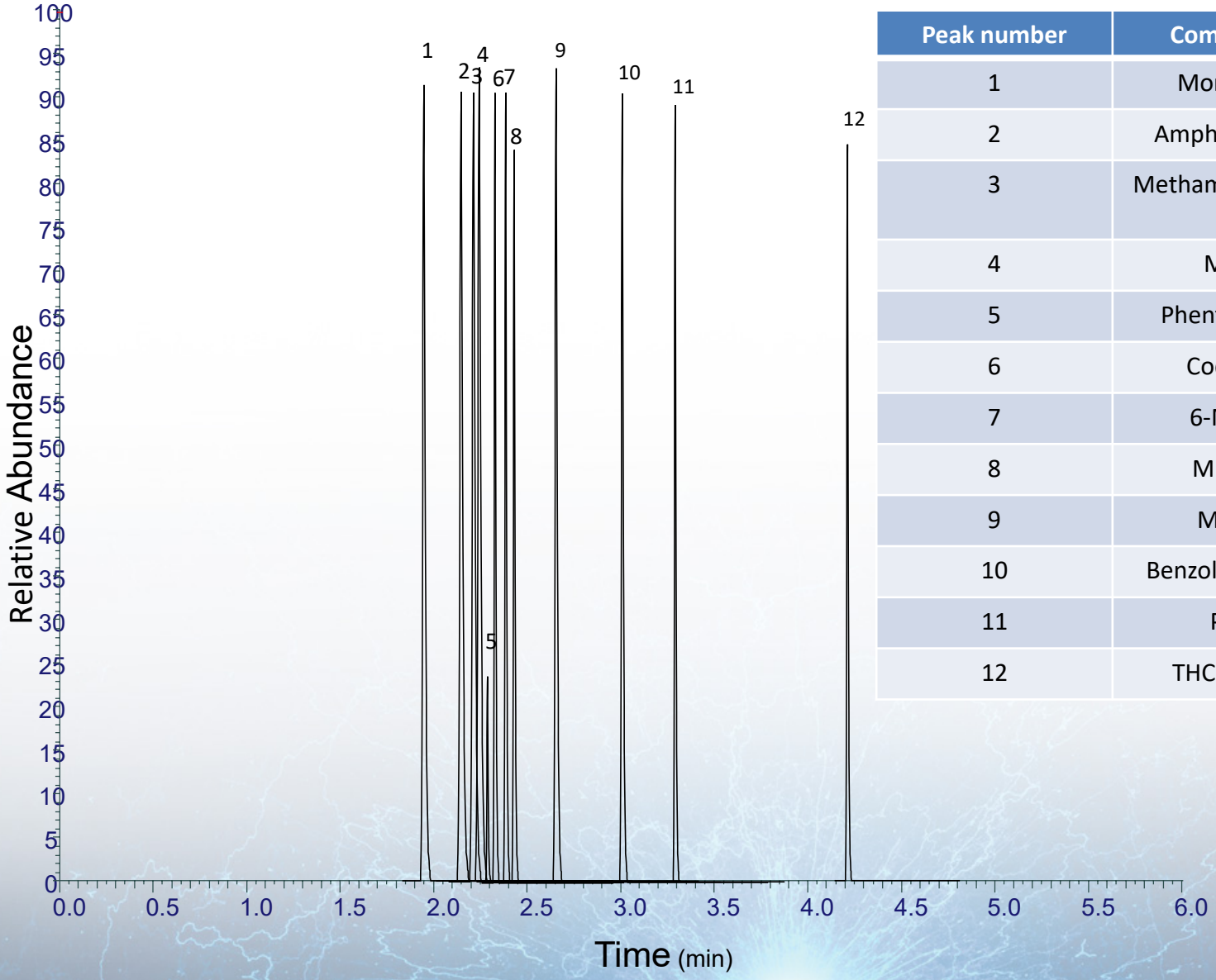
Columns: HALO® 90 Å Biphenyl, 2 µm, 2.1 x 100
Part Number: 91812-611
Mobile Phase A: Water/0.1% Formic acid
Mobile Phase B: Methanol/0.1% Formic acid

| Gradient: | Time | %B |
|---|---|---|
| | 0.0 | 5 |
| | 4.00 | 98 |
| | 5.00 | 98 |
| | 5.01 | 5 |
| | 7.00 END | |

Flow Rate: 0.4 mL/min
Initial Pressure: 325 bar
Temperature: 40 ° C
Injection Volume: 2 µL
Sample Solvent: 95/5 MEOH/Water
LC System: Shimadzu Nexera X2
MS: Orbitrap Exactive mass spectrometer



# SAMHSA-5 Panel




| Peak number | Compound | Observed ion |
|---|---|---|
| 1 | Morphine | 286.341 m/z |
| 2 | Amphetamine | 136.206 m/z |
| 3 | Methamphetamine | 150.237 m/z |
| 4 | MDA | 180.221 m/z |
| 5 | Phentermine | 150.233 m/z |
| 6 | Codeine | 300.364 m/z |
| 7 | 6-MAM | 328.380 m/z |
| 8 | MDMA | 194.246 m/z |
| 9 | MDEA | 208.271 m/z |
| 10 | Benzolecgonine | 290.331 m/z |
| 11 | PCP | 244.387 m/z |
| 12 | THC-COOH | 345.415 m/z |

# SAMHSA-5 Panel isomeric resolution

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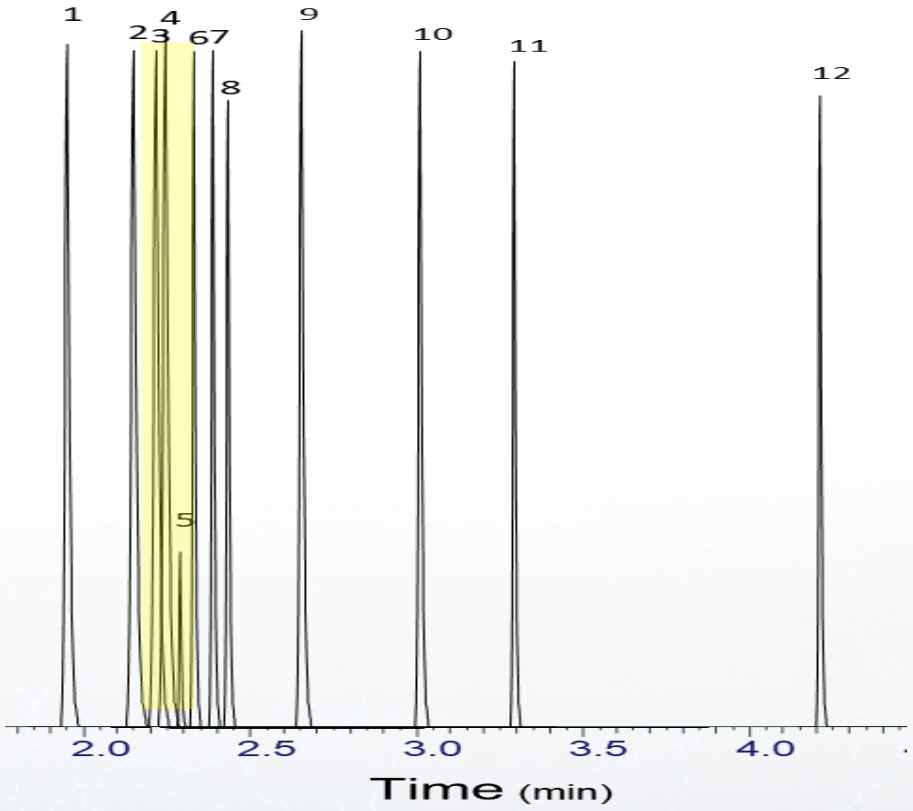



Notice the resolution of the isomers, methamphetamine and phentermine (Peaks 3 and 5)

# TAG profiles of Edible oils on a C30

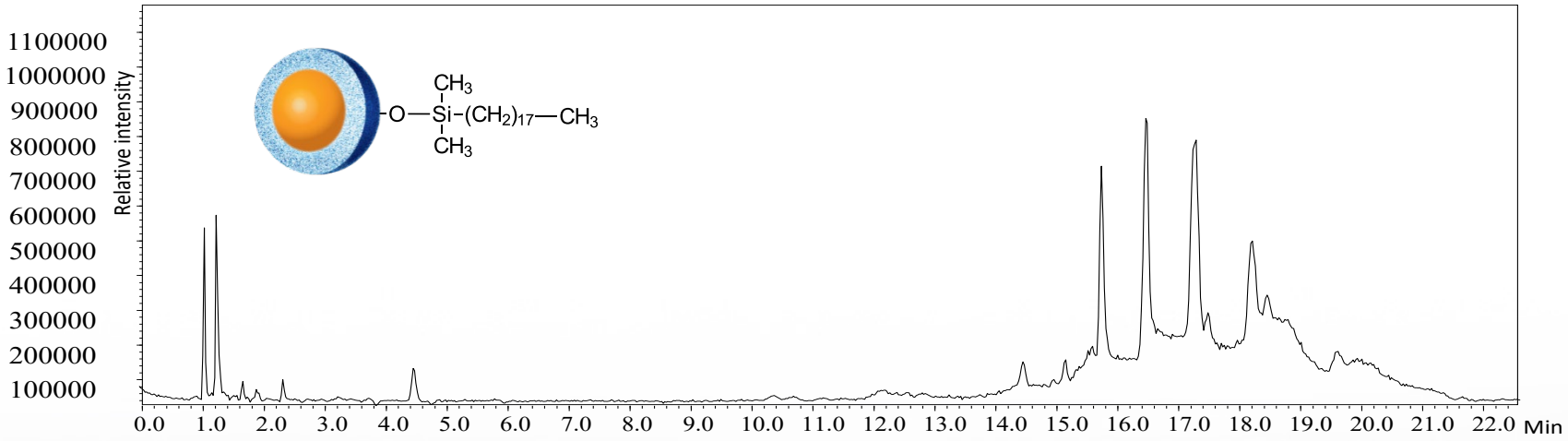
- Long chain fatty acids and esters
- Triglycerides & diglycerides
- Hydrophobic

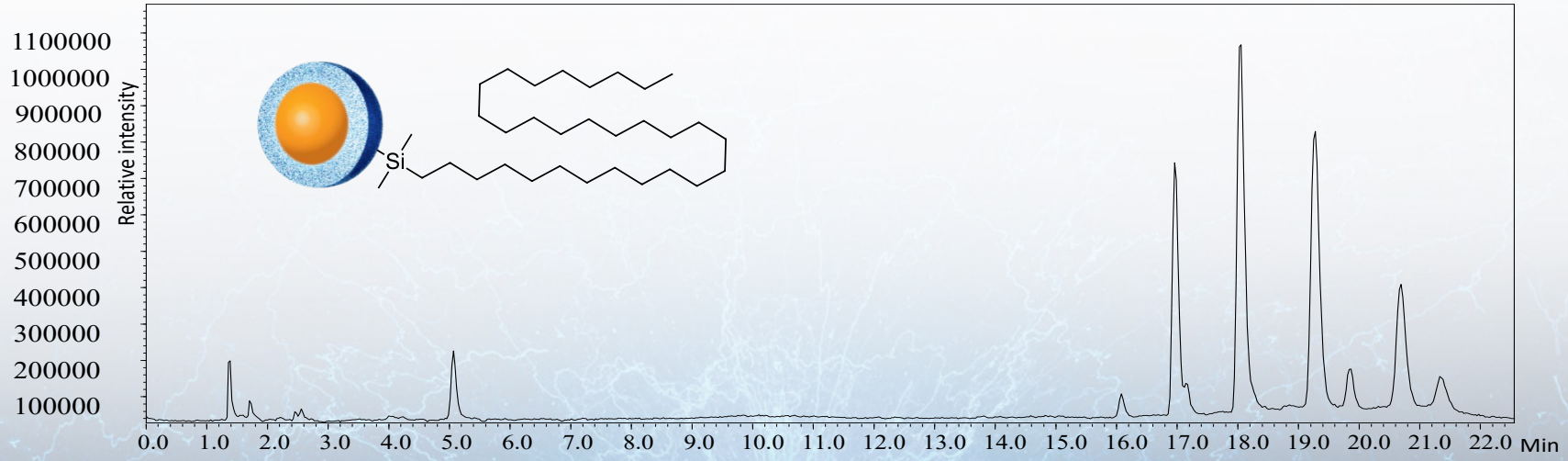
Columns: HALO® 160 Å C30, 2.7 µm, 2.1 x 150
Part Number: 92115-730
Mobile Phase A: MEOH 10mM ammonium formate/0.1% formic acid
Mobile Phase B: IPA/0.1% Formic acid

| Gradient: | Time | %B |
|---|---|---|
| | 0.0 | 5.0 |
| | 10.00 | 20 |
| | 15.00 | 80 |
| | 25.00 | 80 |
| | 25.50 | 5.0 |
| | 32.00 | END |

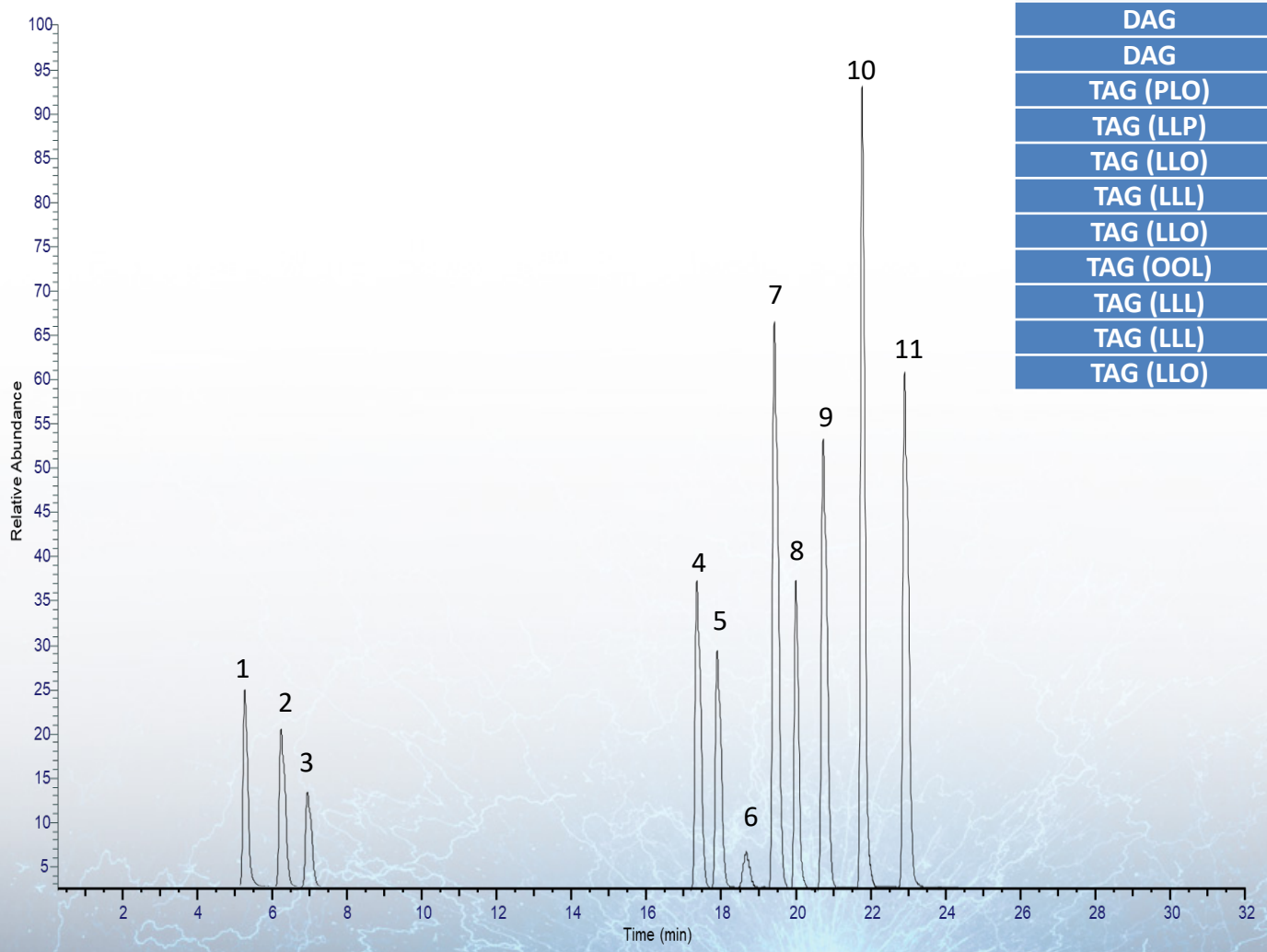
Flow Rate: 0.4 mL/min
Initial Pressure: 325 bar
Temperature: 40 ° C
Injection Volume: 2 µL
Sample Solvent: MEOH
LC System: Shimadzu Nexera X2
MS: Orbitrap Exactive mass spectrometer

# C18 vs C30 comparison



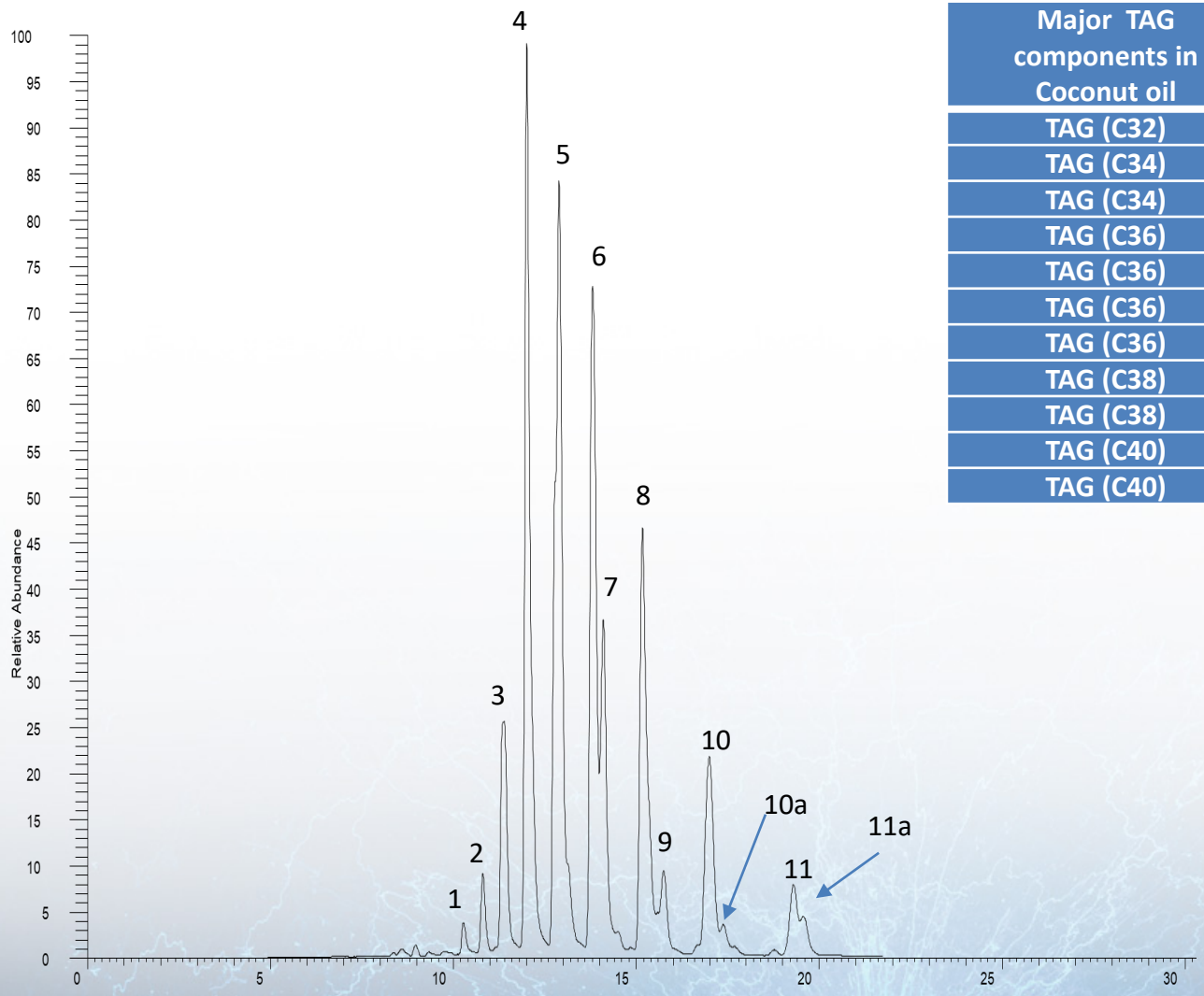



# Corn oil



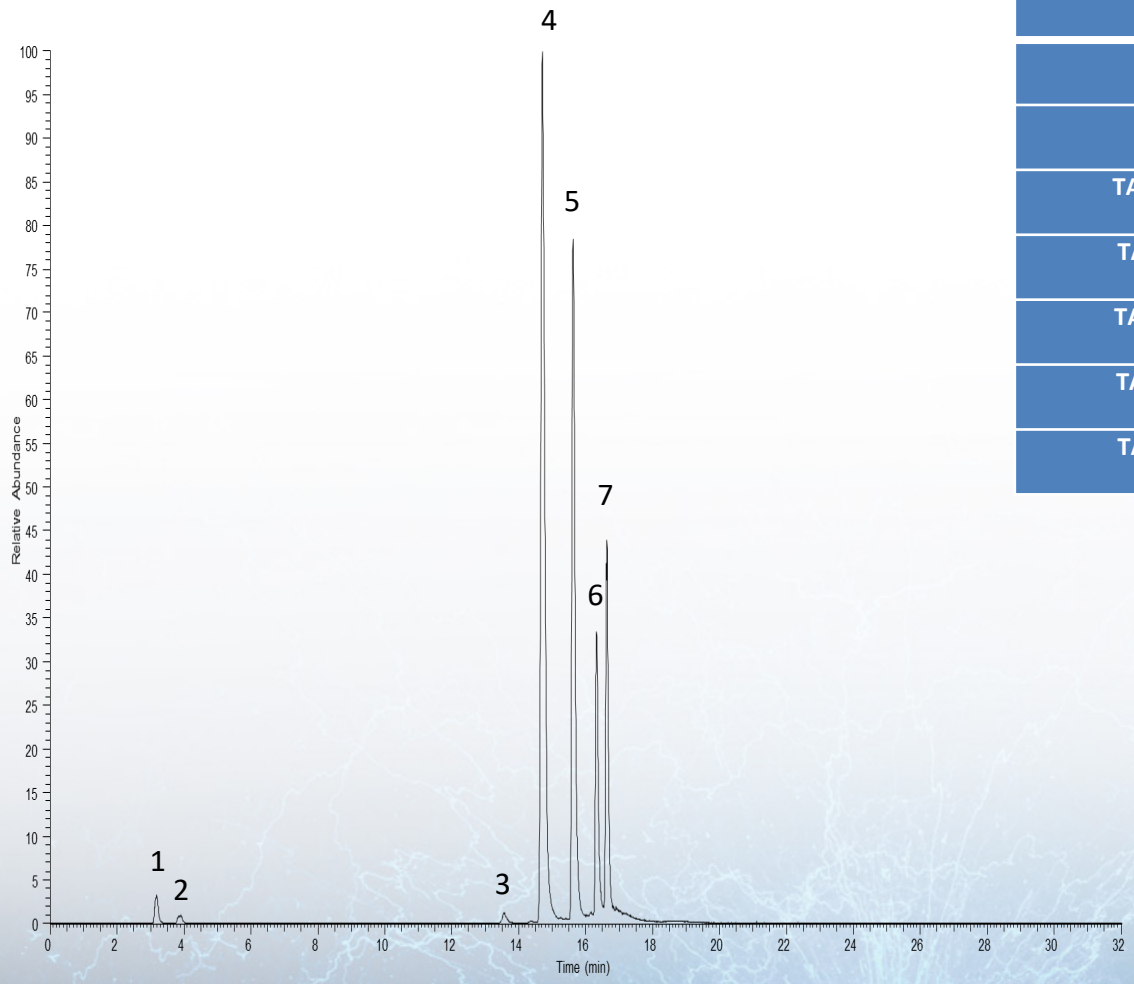

| Major TAGS in Corn Oil | Observed Ions | Peak Number |
|---|---|---|
| DAG | 634.325 m/z | 1 |
| DAG | 636.375 m/z | 2 |
| DAG | 638.405 m/z | 3 |
| TAG (PLO) | 856.751 m/z | 4 |
| TAG (LLP) | 858.767 m/z | 5 |
| TAG (LLO) | 872.755 m/z | 6 |
| TAG (LLL) | 879.405 m/z | 7 |
| TAG (LLO) | 881.465 m/z | 8 |
| TAG (OOL) | 885.432 m/z | 9 |
| TAG (LLL) | 898.358 m/z | 10 |
| TAG (LLL) | 900.478 m/z | 11 |
| TAG (LLO) | 902.499 m/z | 12 |

# Coconut oil



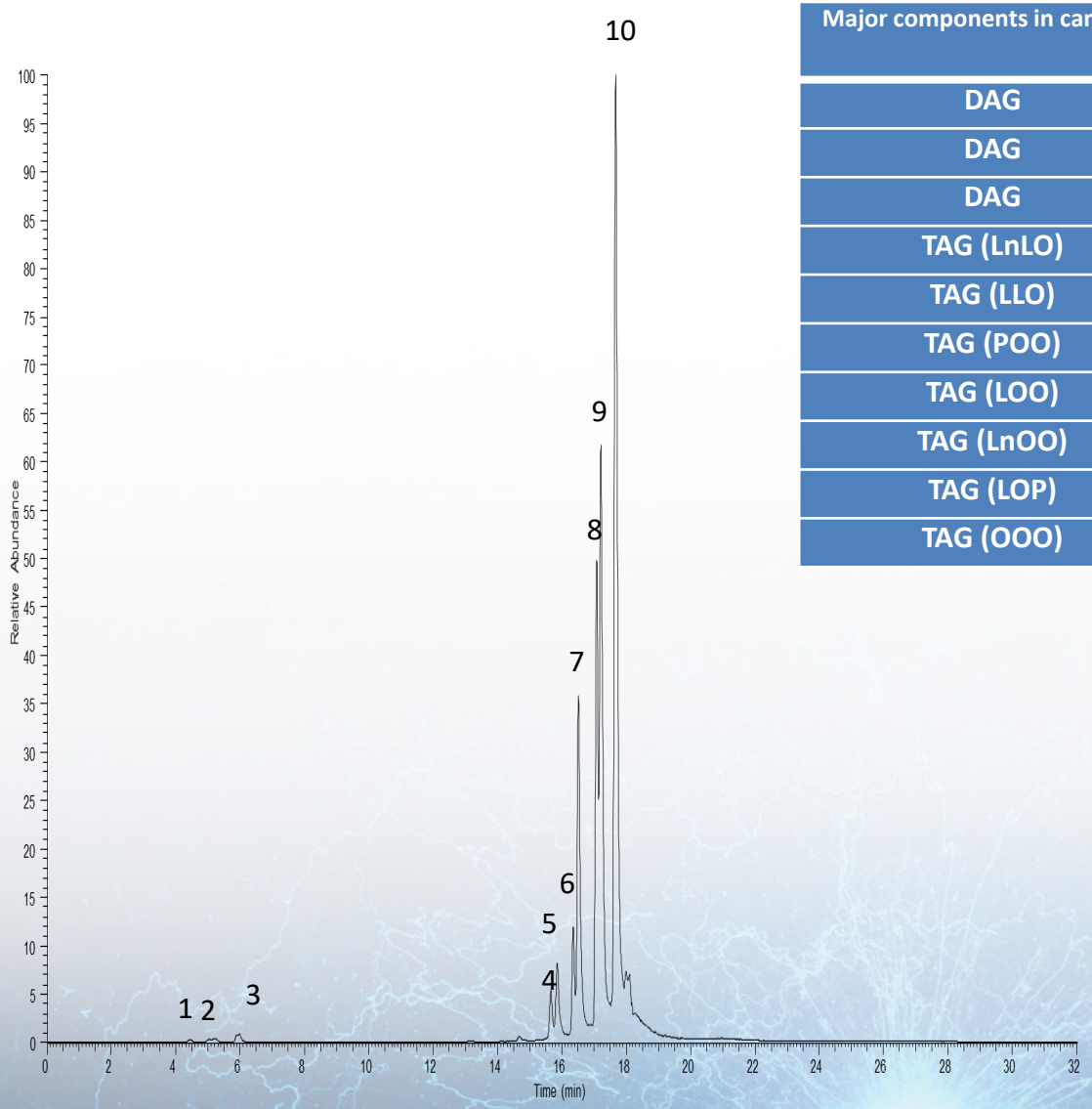

| Major TAG components in Coconut oil | Observed Ions | Peak Number |
|---|---|---|
| TAG (C32) | 516.506 m/z | 1 |
| TAG (C34) | 544.554 m/z | 2 |
| TAG (C34) | 572.668 m/z | 3 |
| TAG (C36) | 600.502 m/z | 4 |
| TAG (C36) | 628.616 m/z | 5 |
| TAG (C36) | 656.713 m/z | 6 |
| TAG (C36) | 684.774 m/z | 7 |
| TAG (C38) | 712.598 m/z | 8 |
| TAG (C38) | 740.518 m/z | 9 |
| TAG (C40) | 768.566 m/z | 10 |
| TAG (C40) | 796.614 m/z | 11 |

# Grape Seed oil




| Major TAG components in Grape seed oil | Observed Ions | Peak Number |
|---|---|---|
| DAG | 634.475 m/z | 1 |
| DAG | 636.491 m/z | 2 |
| TAG (POL) | 894.718 m/z | 3 |
| TAG (LLL) | 896.358 m/z | 4 |
| TAG (OLL) | 898.471 m/z | 5 |
| TAG (PLL) | 900.385 m/z | 6 |
| TAG (LLL) | 900.485 m/z | 7 |

# Rapeseed/Canola Oil




| Major components in canola oil | Observed Molecular mass (m/z) | Peak Number |
|---|---|---|
| DAG | 634.425 m/z | 1 |
| DAG | 636.495 m/z | 2 |
| DAG | 638.437 m/z | 3 |
| TAG (LnLO) | 858.767 m/z | 4 |
| TAG (LLO) | 872.755 m/z | 5 |
| TAG (POO) | 874.355 m/z | 6 |
| TAG (LOO) | 885.432 m/z | 7 |
| TAG (LnOO) | 896.432 m/z | 8 |
| TAG (LOP) | 900.734 m/z | 9 |
| TAG (OOO) | 902.499 m/z | 10 |

# Conclusion

- HALO columns are an attractive option for applications requiring high throughput and high selectivity.
- In addition, the columns exhibit excellent cross market appeal (Including biologics) by enabling separation and resolution of hydrophobic, as well as isomeric compounds.

# Acknowledgements


- Scientists at Advanced Materials Technology

  Conner McHale, Stephanie Schuster, William Johnson