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

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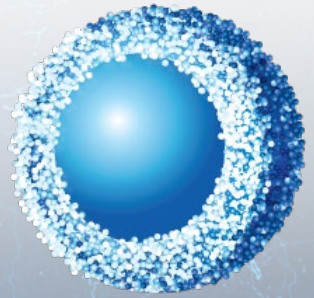
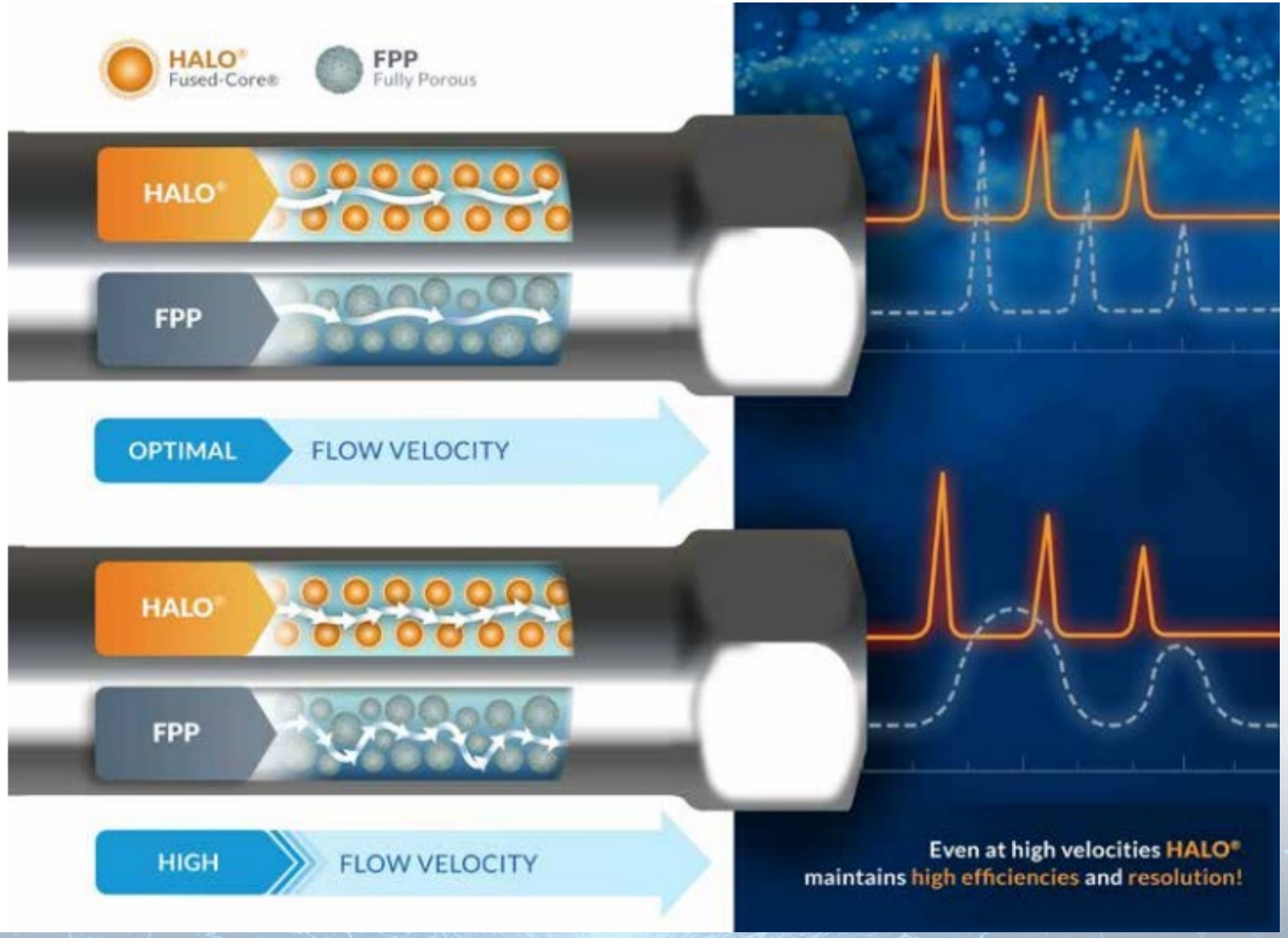


Outline

- 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

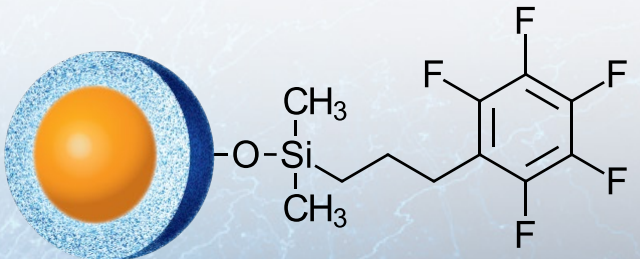
Injection Volume: 1 µL

Sample Solvent: 95/5 water/methanol

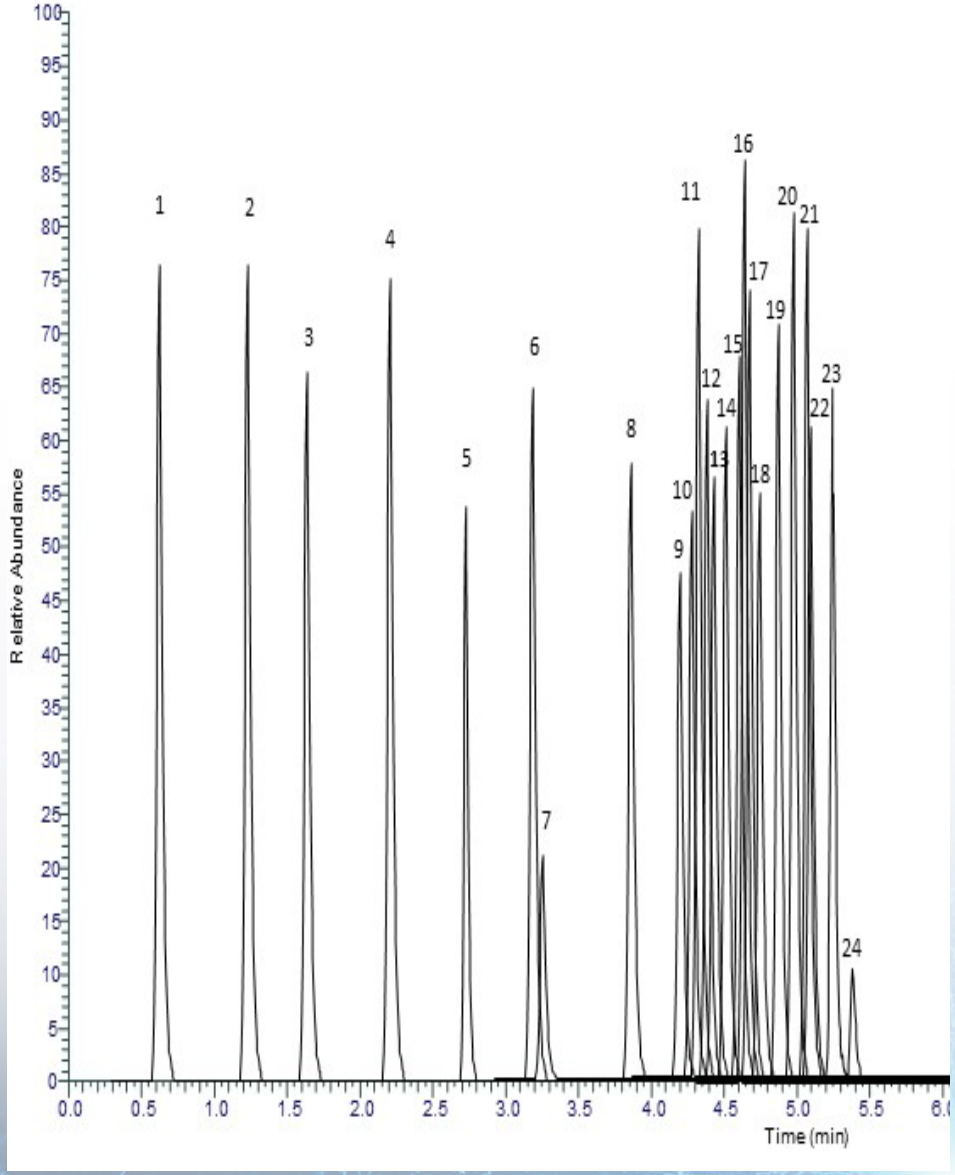
LC System: Shimadzu Nexera X2

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

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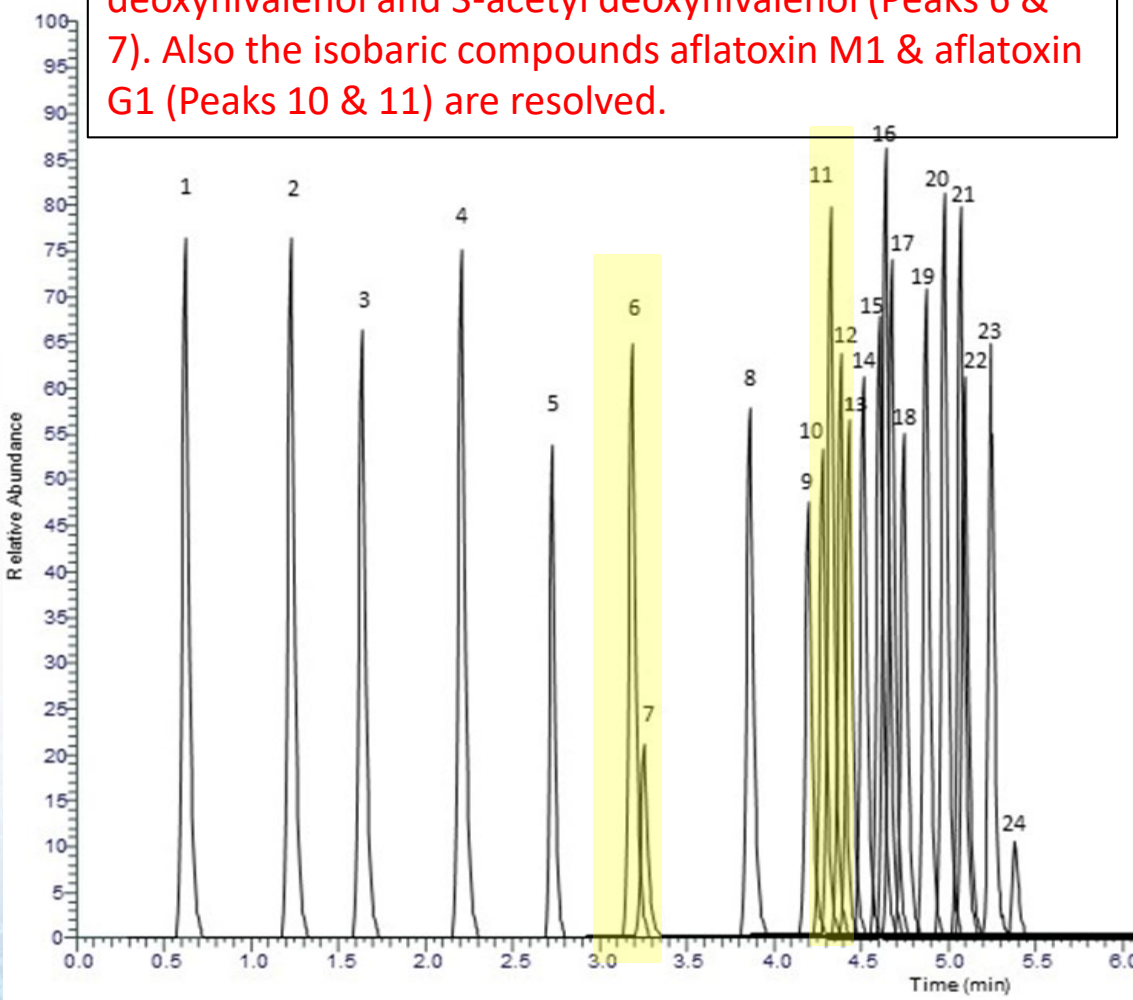
Notice the resolution of the isomers, 15-acetyl deoxynivalenol and 3-acetyl deoxynivalenol (Peaks 6 & 7). Also the isobaric compounds aflatoxin M1 & aflatoxin G1 (Peaks 10 & 11) are resolved.



SAMHSA-5 Panel

- SAMHSA-The Substance Abuse and Mental Health Services Administration

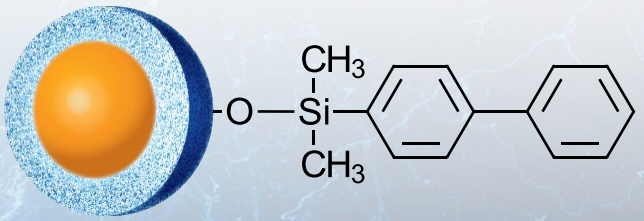
- Amphetamines (Amphetamines, Methamphetamines, Speed)
- Cocaine (Cocaine, Crack, Benzoylecognine)
- Marijuana – THC Tetrahydrocannabinol (Cannabinoids, Hash)
- Opiates (Heroin, Opium, Codeine, Morphine)
- Phencyclidine (PCP)

- HALO® Biphenyl

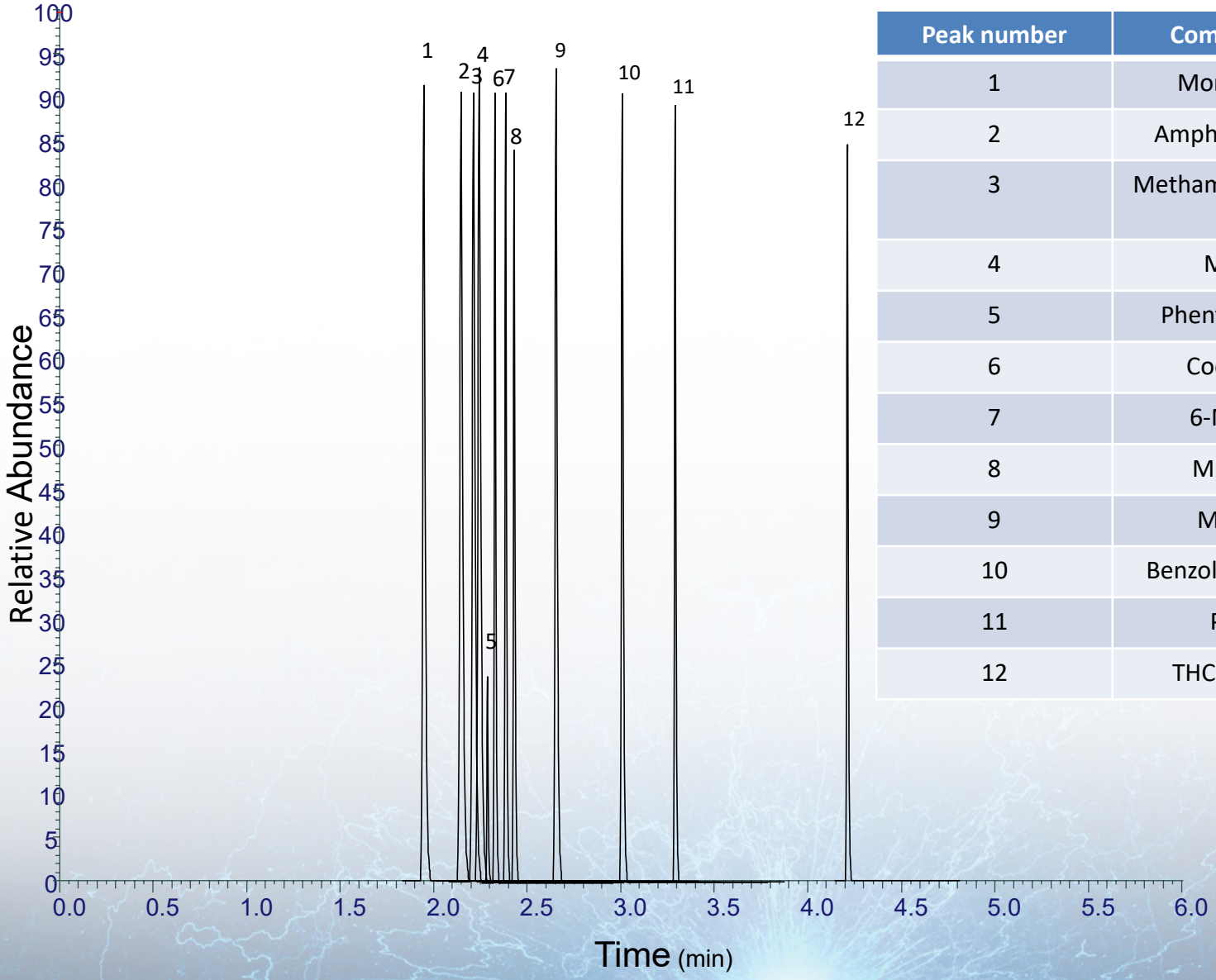
Columns: HALO® 90 Å Biphenyl, 2 µm, 2.1 x 100
Part Number: 91812-611
Mobile Phase A: Water/0.1% Formic acid
Mobile Phase B: Methanol/0.1% Formic acid

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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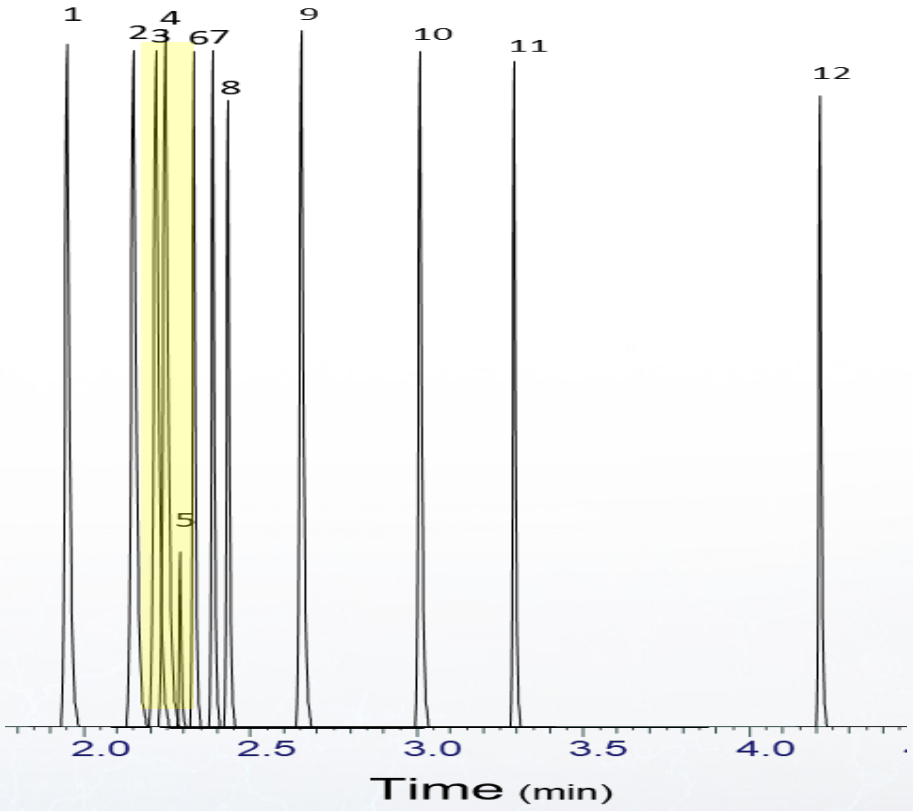


Notice the resolution of the isomers, methamphetamine and phentermine (Peaks 3 and 5)

TAG profiles of Edible oils on a C30

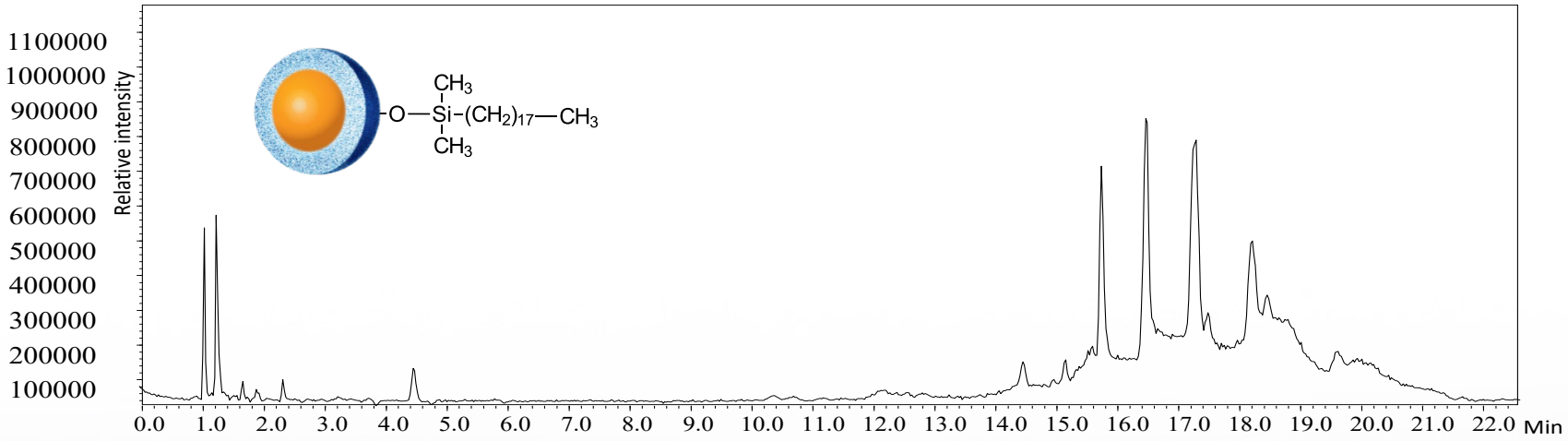
- Long chain fatty acids and esters
- Triglycerides & diglycerides
- Hydrophobic

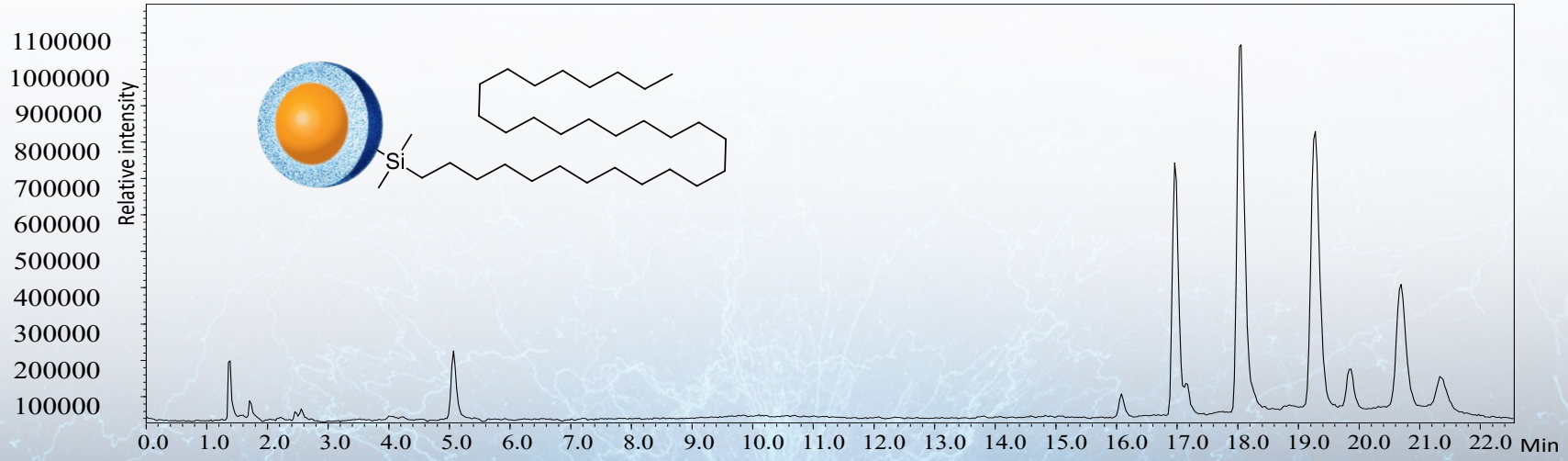
Columns: HALO® 160 Å C30, 2.7 µm, 2.1 x 150
Part Number: 92115-730
Mobile Phase A: MEOH 10mM ammonium formate/0.1% formic acid
Mobile Phase B: IPA/0.1% Formic acid

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

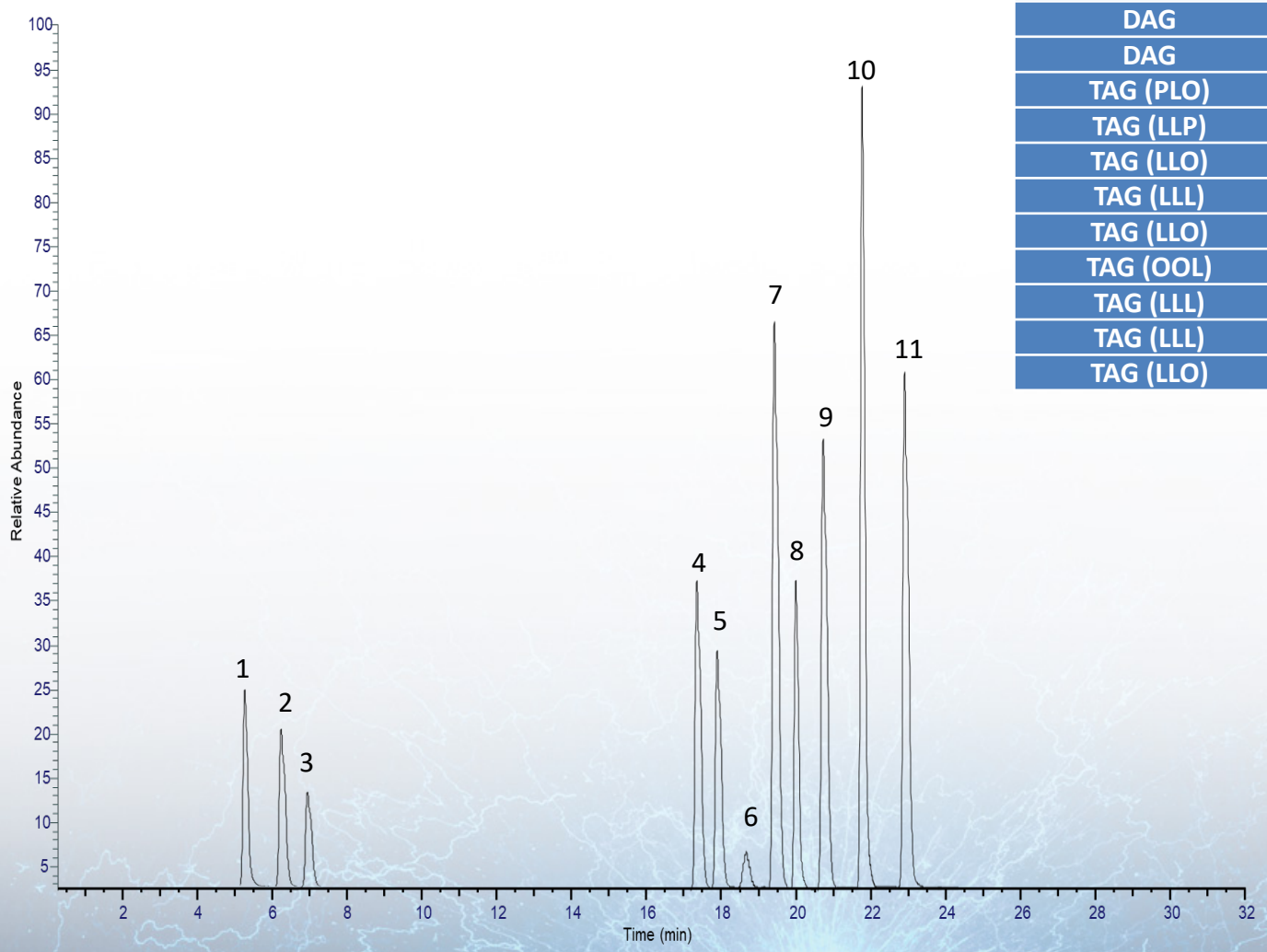
Flow Rate: 0.4 mL/min
Initial Pressure: 325 bar
Temperature: 40 ° C
Injection Volume: 2 µL
Sample Solvent: MEOH
LC System: Shimadzu Nexera X2
MS: Orbitrap Exactive mass spectrometer

C18 vs C30 comparison



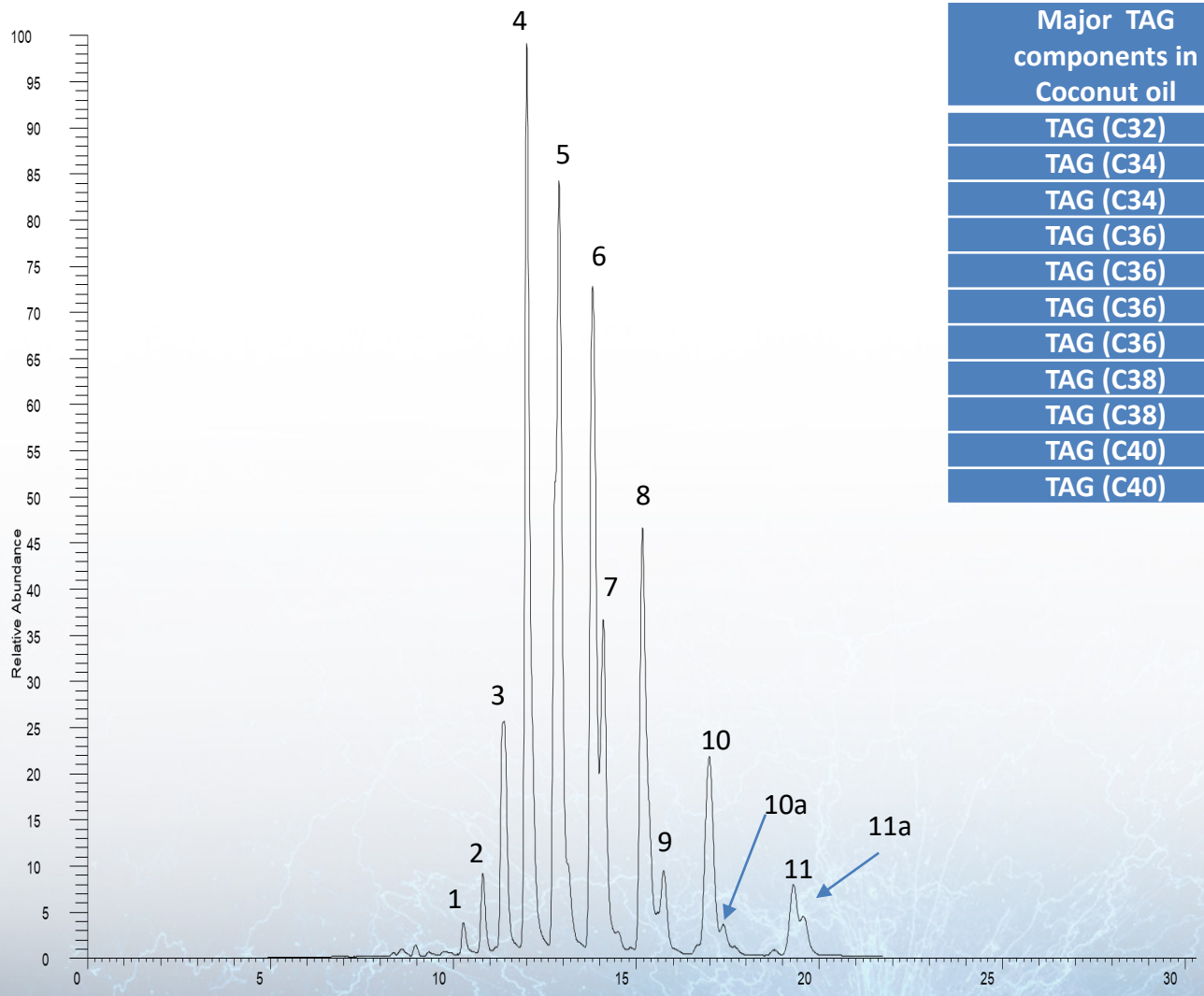


Corn oil



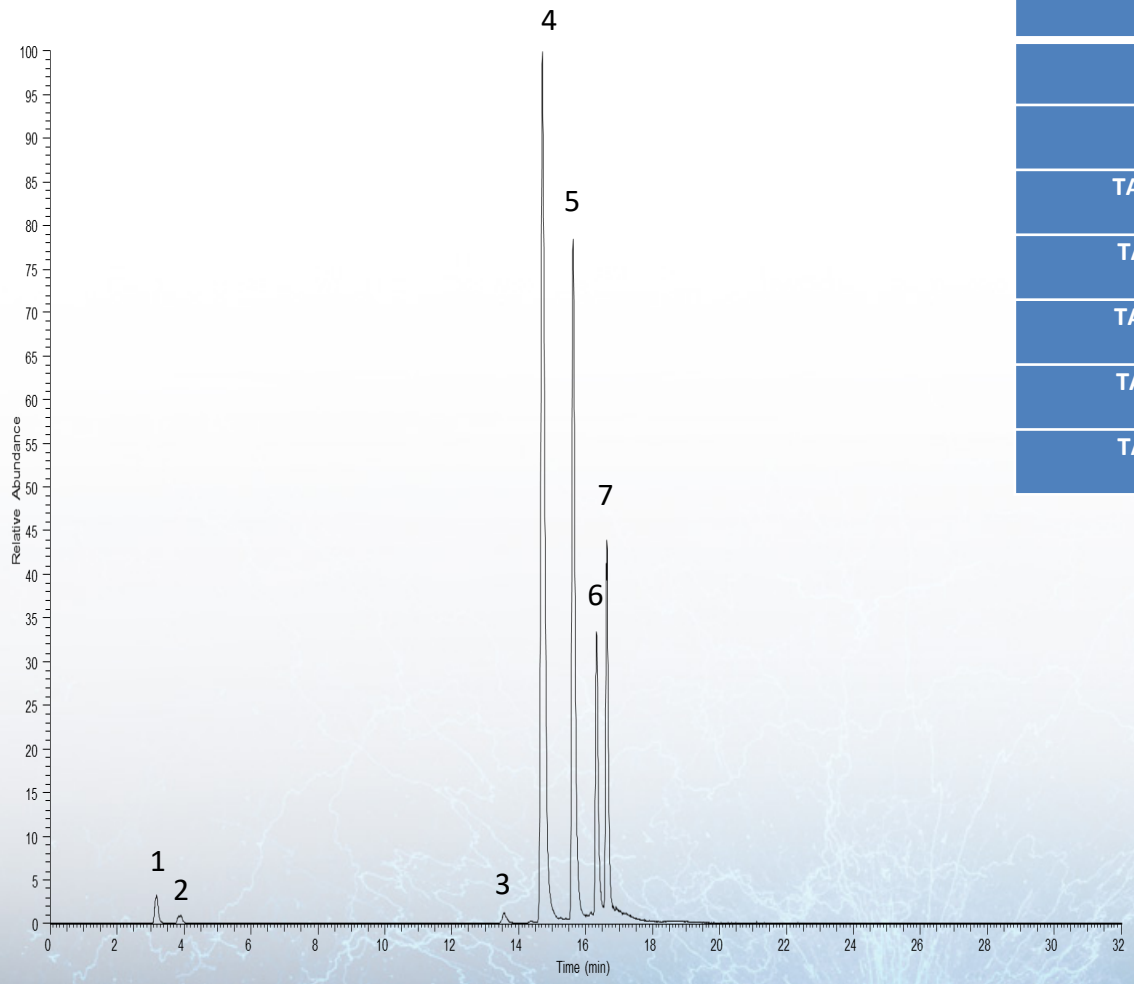
Major TAGS in Corn Oil	Observed Ions	Peak Number
DAG	634.325 m/z	1
DAG	636.375 m/z	2
DAG	638.405 m/z	3
TAG (PLO)	856.751 m/z	4
TAG (LLP)	858.767 m/z	5
TAG (LLO)	872.755 m/z	6
TAG (LLL)	879.405 m/z	7
TAG (LLO)	881.465 m/z	8
TAG (OOL)	885.432 m/z	9
TAG (LLL)	898.358 m/z	10
TAG (LLL)	900.478 m/z	11
TAG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

Coconut oil



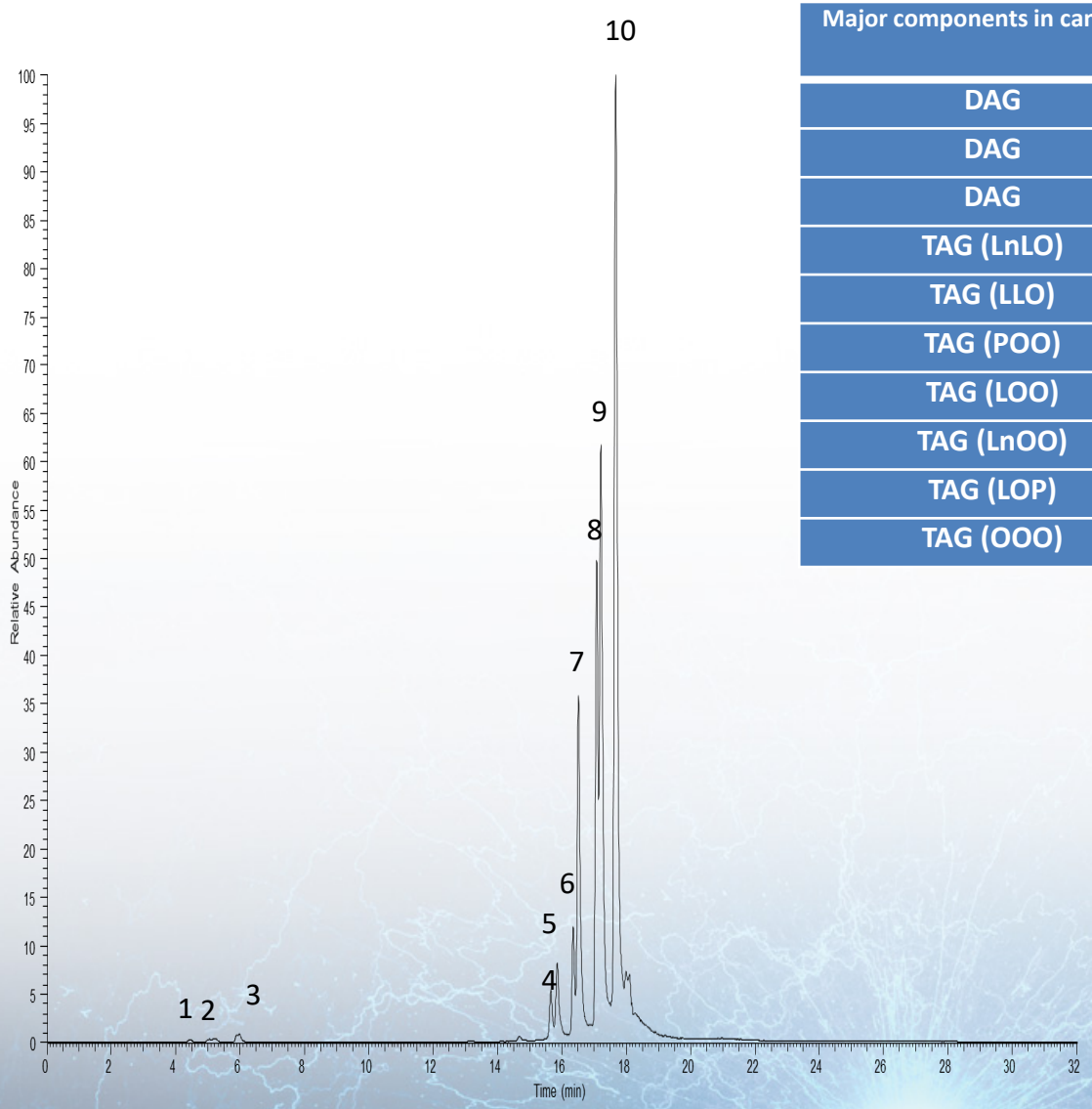
Major TAG components in Coconut oil	Observed Ions	Peak Number
TAG (C32)	516.506 m/z	1
TAG (C34)	544.554 m/z	2
TAG (C34)	572.668 m/z	3
TAG (C36)	600.502 m/z	4
TAG (C36)	628.616 m/z	5
TAG (C36)	656.713 m/z	6
TAG (C36)	684.774 m/z	7
TAG (C38)	712.598 m/z	8
TAG (C38)	740.518 m/z	9
TAG (C40)	768.566 m/z	10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