SUPELCO
Solutions within.

New Developments in Chromatography at Supelco

L.M.Sidisky

May 19, 2015

ISCC, Fort Worth, TX

sigma-aldrich.com/analytical

SIGMA-ALDRICH

New Products

- **GC**
- **SPME**

New Products

- **GC**
- **SLB-35ms**
- **SLB-IL111 and SP-2560, 200 meter versions**
- **SLB-IL60 Fusel Alcohols Application**
- **SLB-IL D3606**
- **SLB-IL PAH**
- **Ionic Liquids for Water and Alcoholic Beverage Analysis**

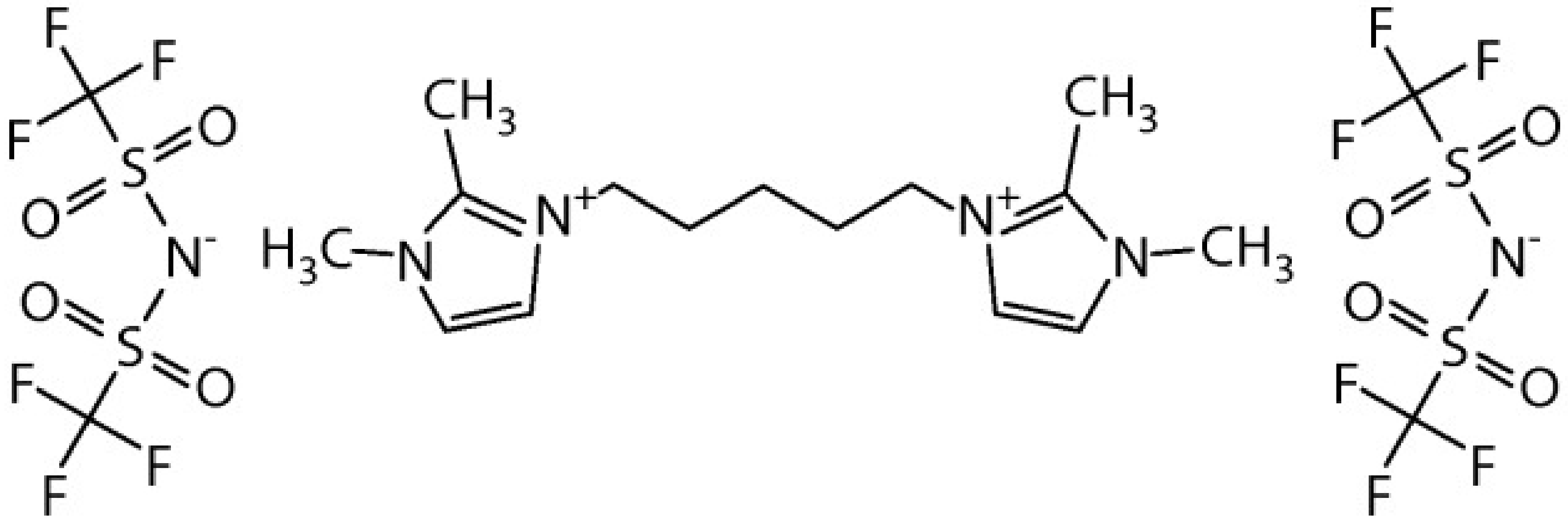
New Products

- **SLB-35ms**
- **A new line of 35% phenyl capillary columns with a**
- **maximum temperature of 350°C**

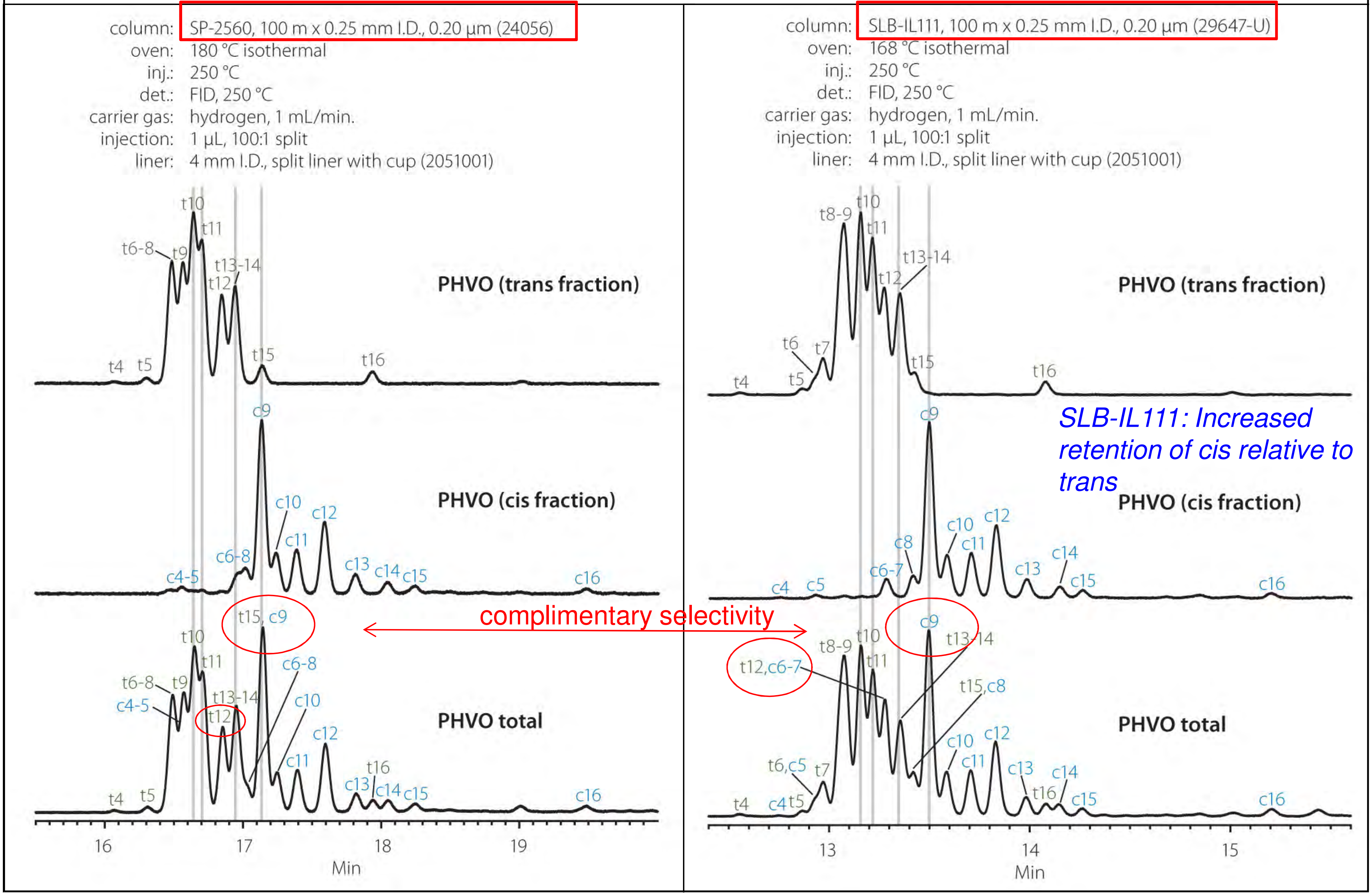
SLB-IL111

Phase Structure

1,5-Di(2,3-dimethylimidazolium)pentane bis(trifluoromethylsulfonyl)imide



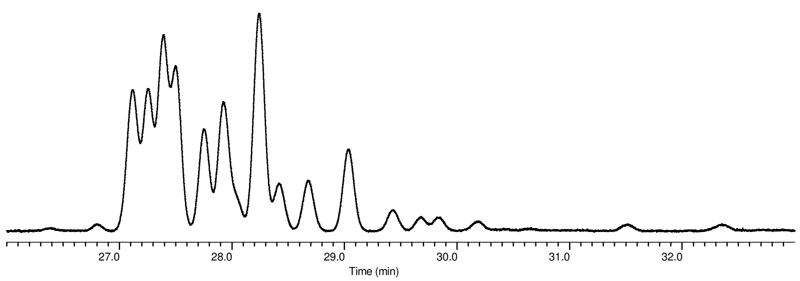
C18:1 cis/trans FAME Isomers in Partially Hydrogenated Vegetable Oil (PHVO) SLB-IL111 vs. SP-2560: 100 m columns



Positional cis/trans FAME Isomers

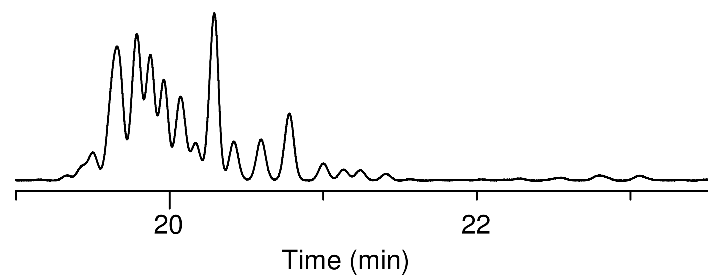
column: SP-2560, 200 m x 0.25 mm I.D., 0.20 µm
oven: 180 °C isothermal
inj.: 250 °C
det.: FID, 250 °C
carrier gas: hydrogen, 1 mL/min.
injection: 1 µL, 100:1 split
liner: 4 mm I.D., split liner with cup (2051001)

PHVO total FAMEs

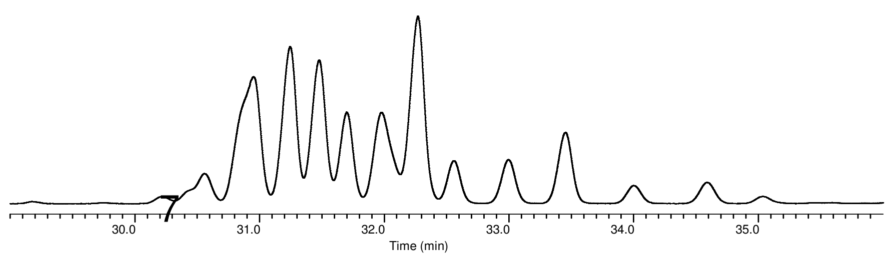


column: SLB-IL111, 200 m x 0.25 mm I.D., 0.20 µm
oven: 168 °C isothermal
inj.: 250 °C
det.: FID, 250 °C
carrier gas: hydrogen, 1 mL/min.
injection: 1 µL, 100:1 split
liner: 4 mm I.D., split liner with cup (2051001)

PHVO total FAMEs



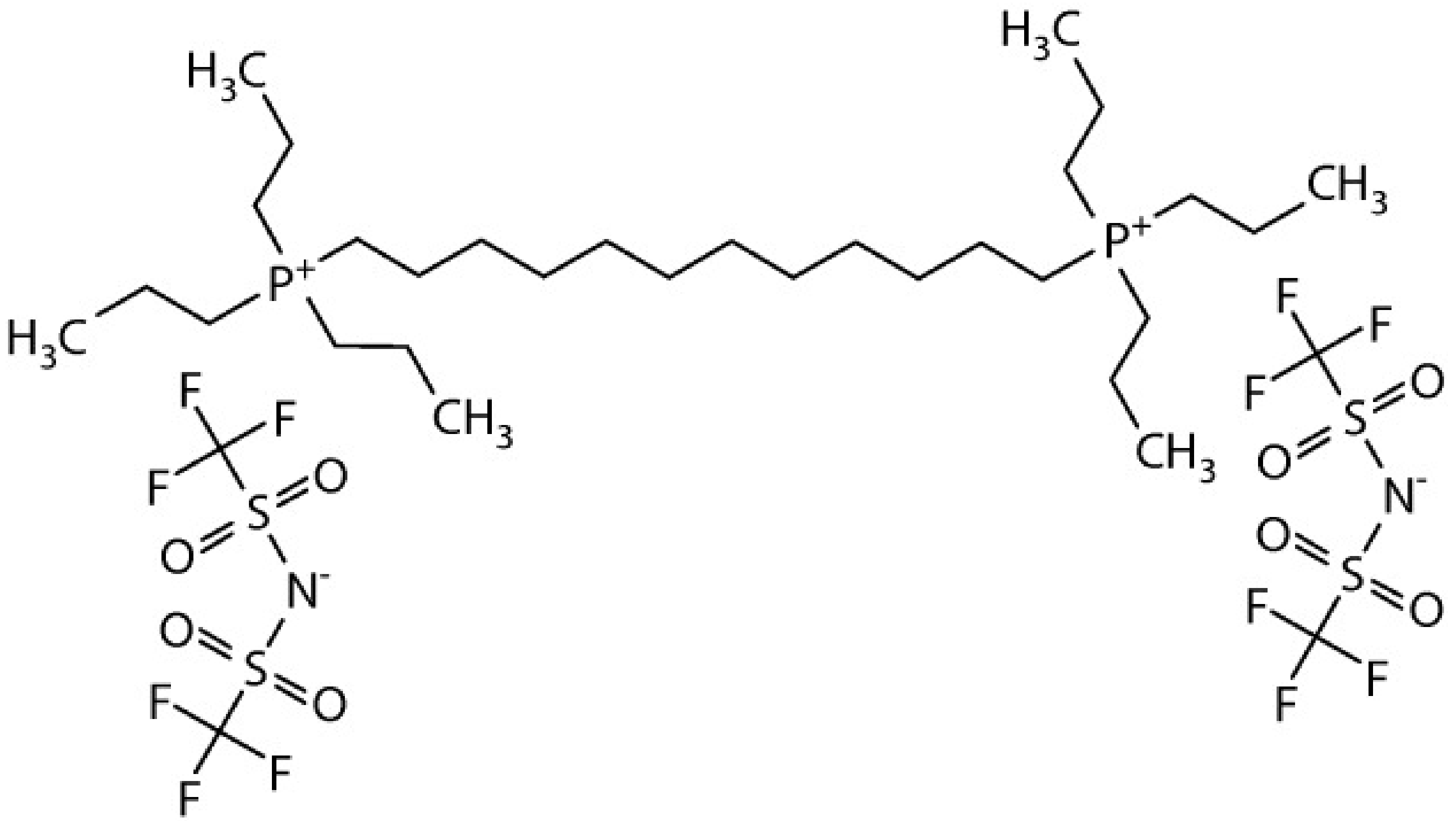
PHVO total FAMEs on SLB-IL111 @ 150 °C isothermal



SLB-IL60

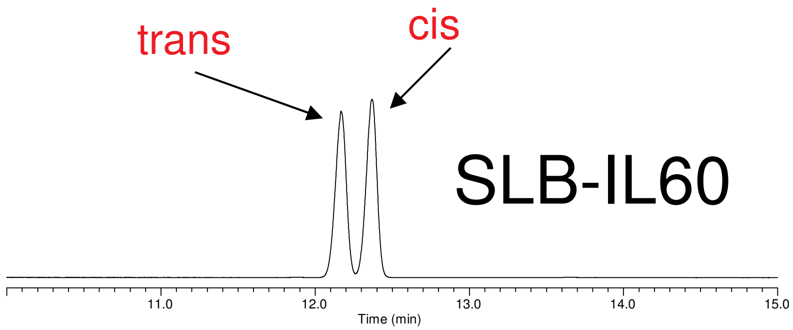
Phase Structure

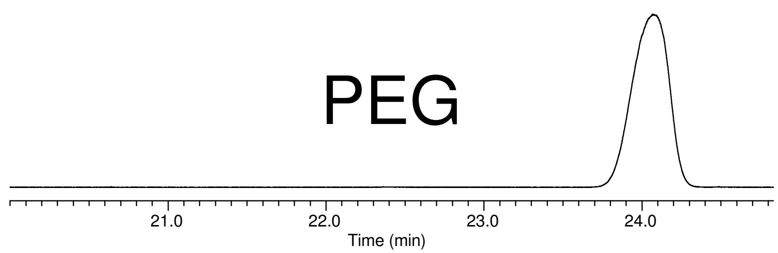
1,12-Di(tripropylphosphonium)dodecane bis(trifluoromethylsulfonyl)imide



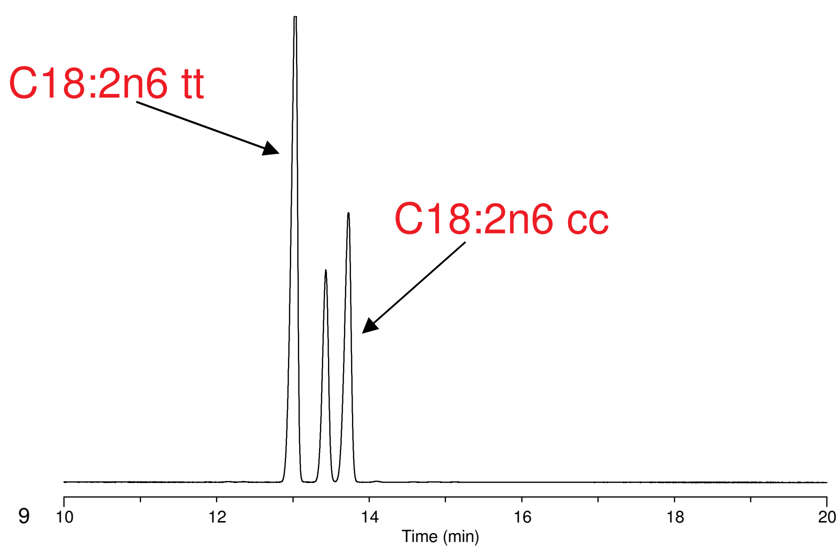
Cis/ trans FAMES on SLB-IL60 vs. PEG Type Phase

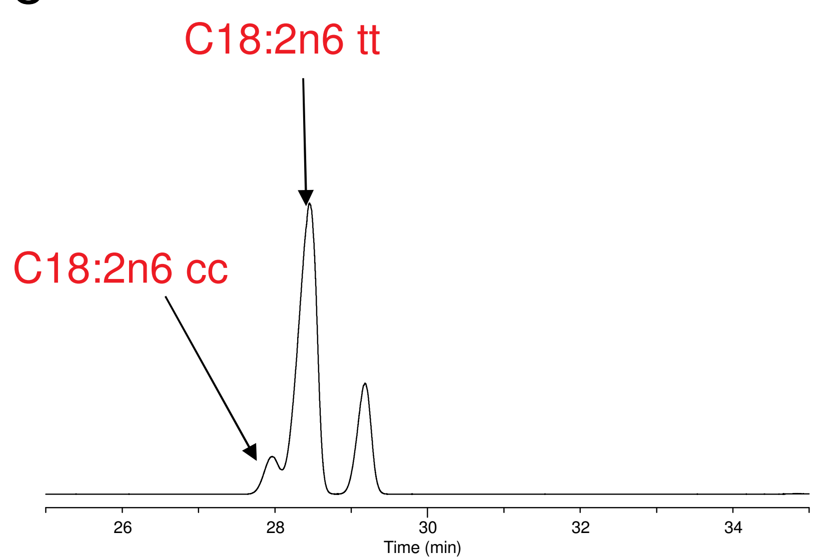
C18:1n9 cis / trans FAMEs @ 180°C





C18:2n6 cis & trans FAME Isomers- 180°C





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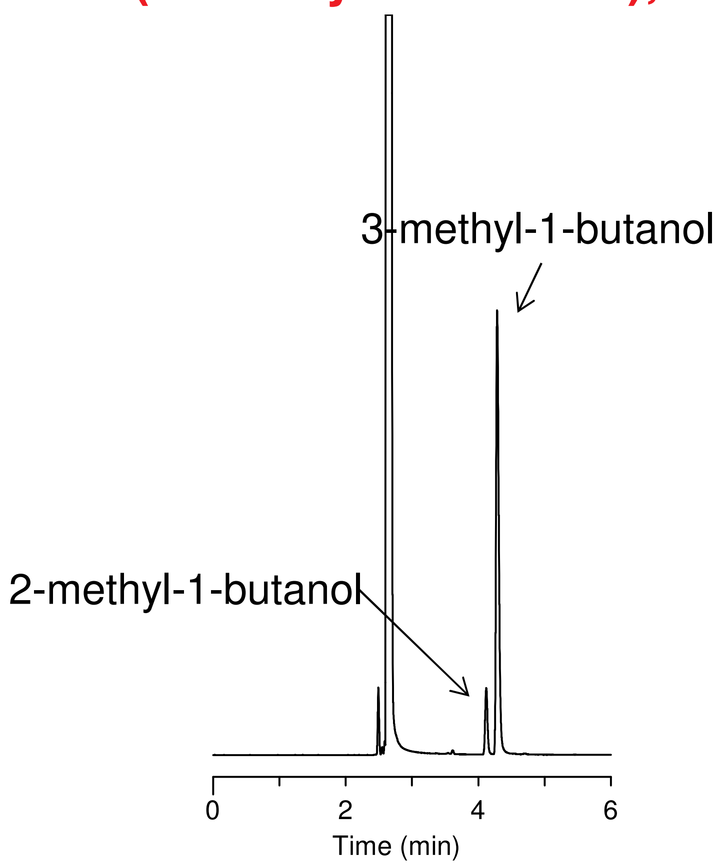
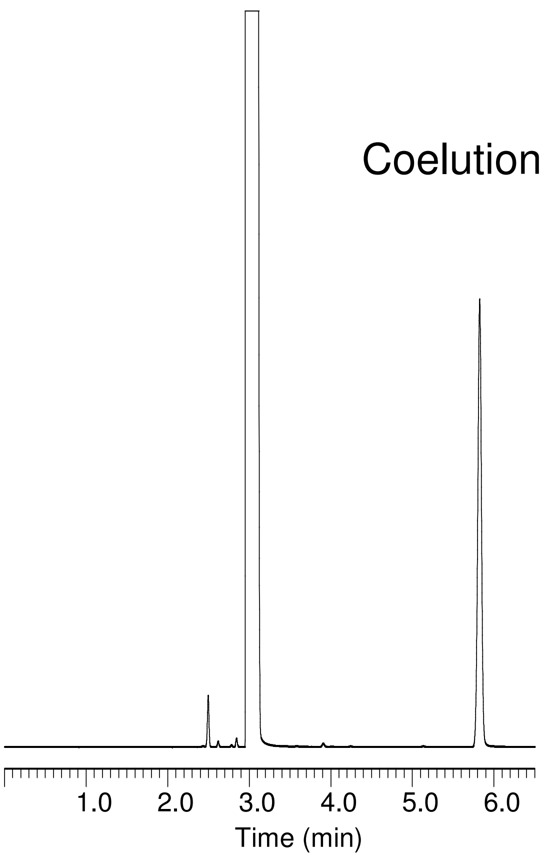
Fusel Oils Separation-

Active amyl alcohol (2-methyl-1-butanol) and Isoamyl alcohol (3-methyl-1-butanol), 90ºC Isothermal



SLB-IL60

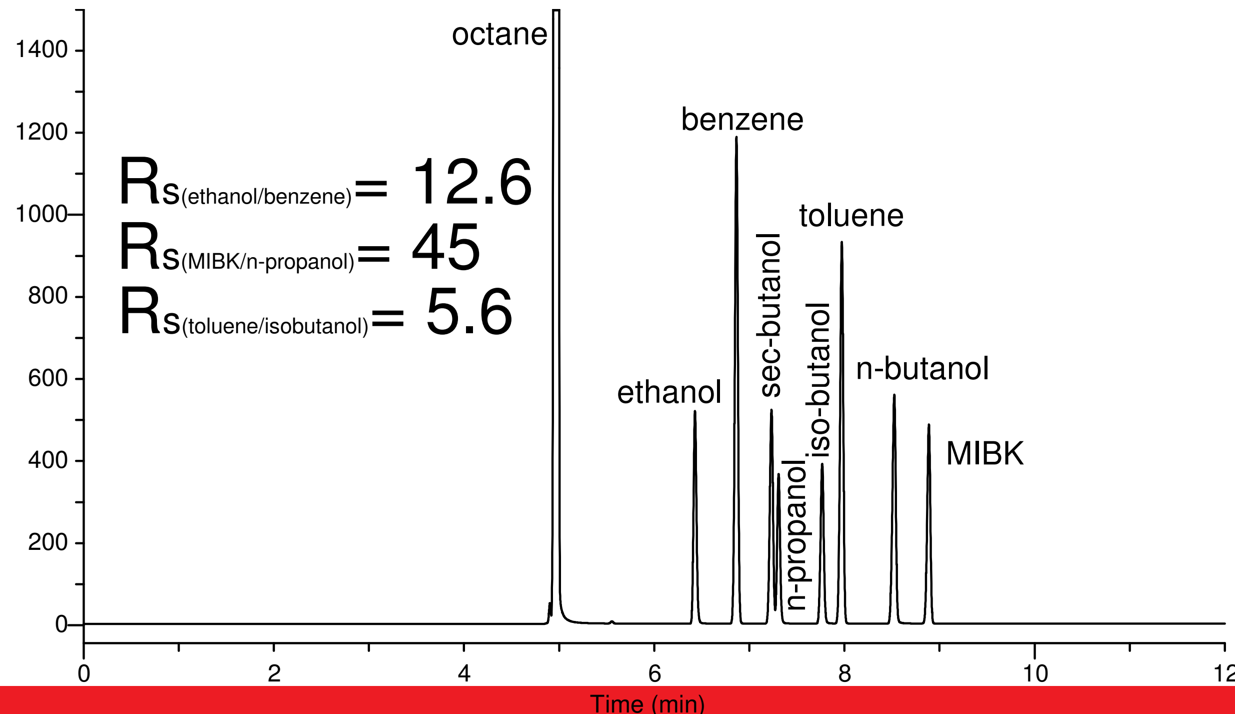


Supelcowax 10

SIGMA-ALDRICH®

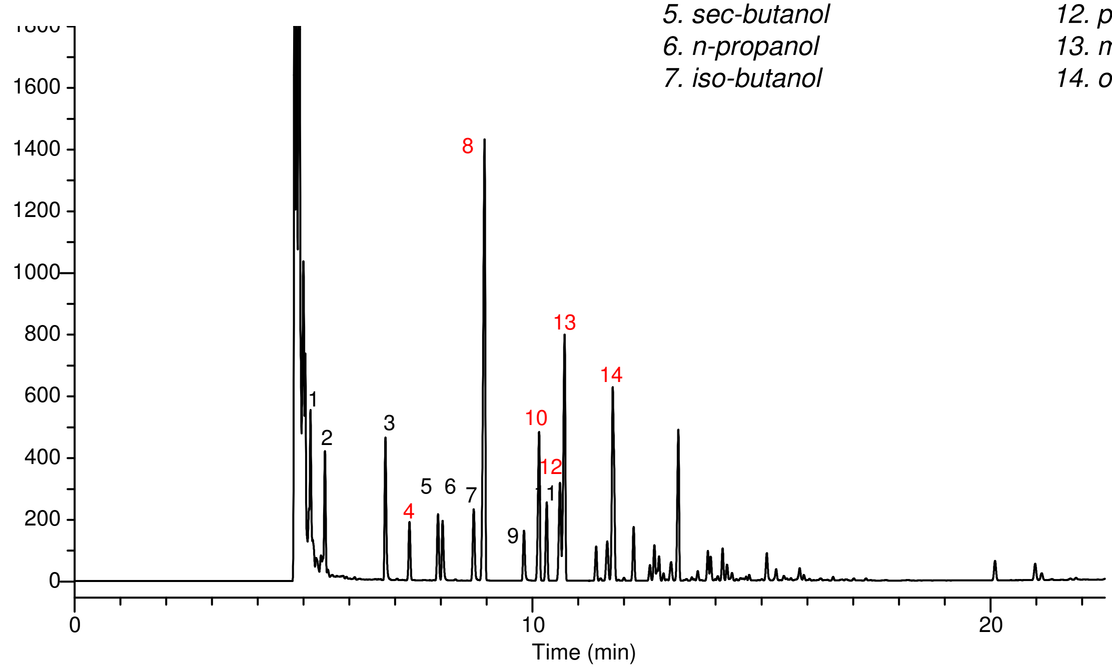
SLB-IL D3606
60m x 0.25 mm ID x 0.20 µm df

- Specially prepared and tested ionic liquid column meets the requirements for resolving benzene and toluene from alcohol interferences (i.e. ethanol, butanol)



Reformulated Gasoline with D3606 Oxygenates
50 ºC (6 min) to 265 ºC (10 min) at 15 ºC/min.

1.Methyl tert-butyl ether (MTBE)
2.tert-Amyl butyl ether (TAME)
3. Ethanol
4. Benzene
5. sec-butanol
6. n-propanol
7. iso-butanol
8. toluene
9. n-butanol
10. ethyl benzene
11. methyl iso-butyl ketone (MIBK
12. p-xylene
13. m-xylene
14. o-xylene.



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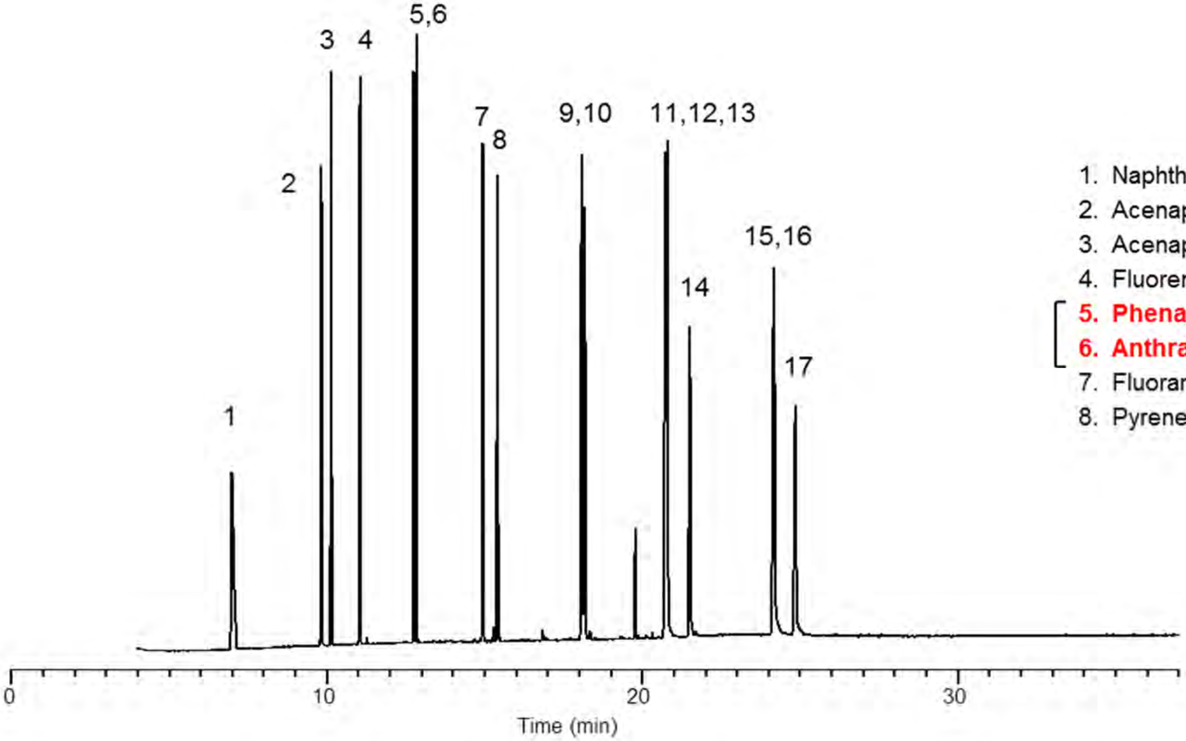
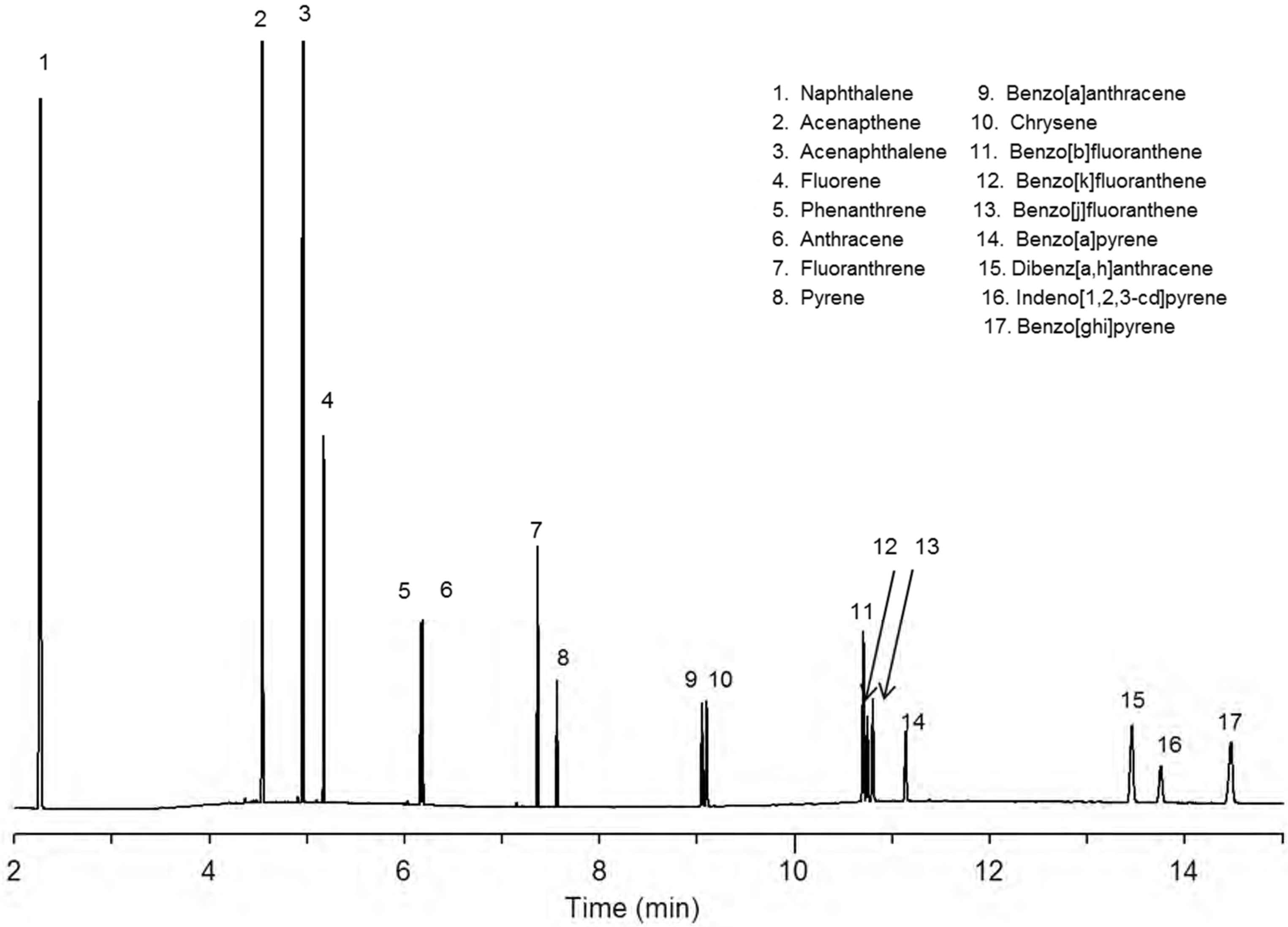
PAHs on a Traditional 5% Silphenylene Phase



1. Naphthalene
2. Acenapthene
3. Acenaphthalene
4. Fluorene
5. **Phenanthrene**
6. **Anthracene**
7. Fluoranthrene
8. Pyrene
9. **Benzo[a]anthracene**
10. **Chrysene**
11. **Benzo[b]fluoranthene**
12. **Benzo[k]fluoranthene**
13. ***Benzo[j]fluoranthene***
14. Benzo[a]pyrene
15. **Dibenz[a,h]anthracene**
16. **Indeno[1,2,3-cd]pyrene**
17. Benzo[ghi]pyrene

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm df (28471-U)
oven: 80 ºC, 15 ºC/min to 250 ºC, 8 ºC/min to 325 ºC (15 min)
inj. temp.: 300 ºC
detector: MSD, full scan, 45-500 m/z, 300 ºC interface
carrier gas: helium, 1.2 mL/min constant flow
injection: 0.5 uL, splitless (1 min)
liner: 4 mm I.D. FocusLiner
sample: EPA 610 PAH mix + Benzo[j]fluoranthene, diluted to 100 µg/mL in methylene chloride

PAHs on SLB-ILPAH, 20 m x 0.18 mm I.D., 0.05 µm d_f)



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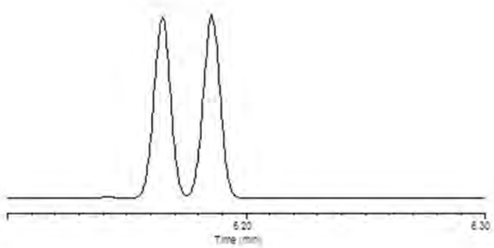
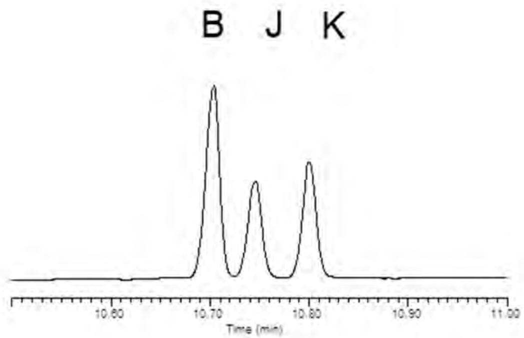
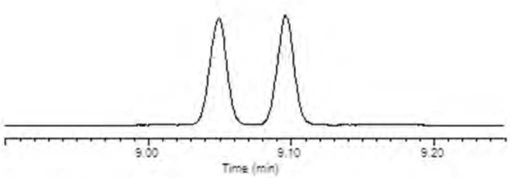
Selected Isomers



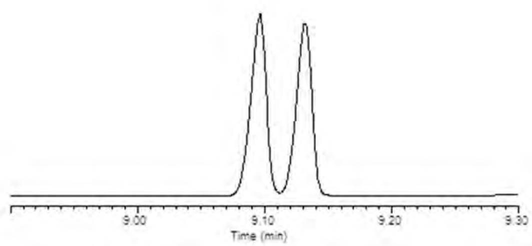
Anthracene/Phenanthrene



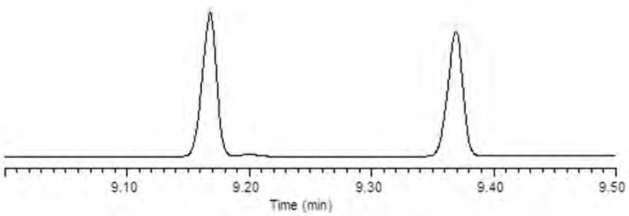
Benzofluoranthenes



Benzo(a)anthracene/Chrysene



Triphenylene/Chrysene



Cyclopenta(cd)pyrene/Chrysene

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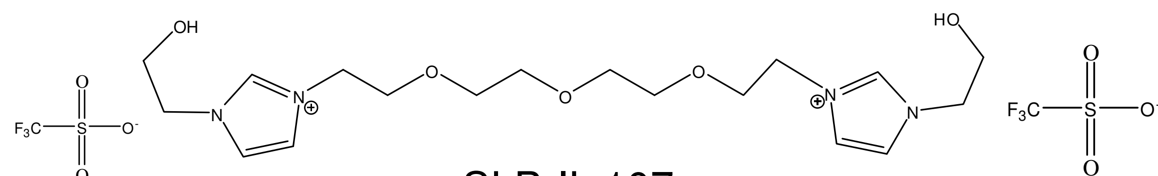
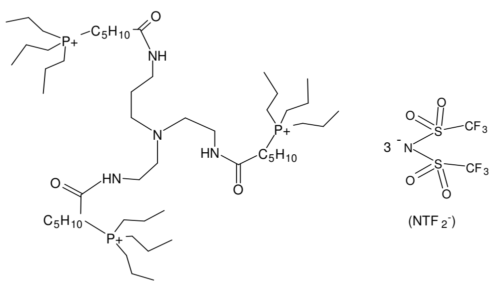
Ionic Liquid Water Separations

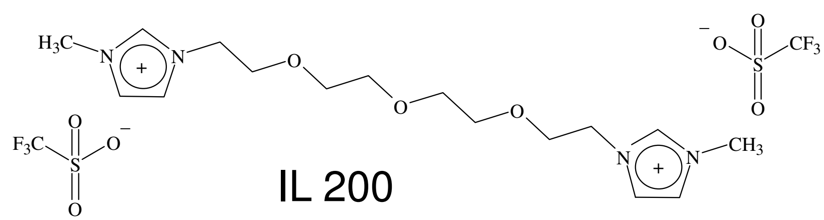
Column: SLB-IL 94, SLB-IL 107, IL 200 30m x 0.25mm x 0.20umd_f
Oven: 35˚C, 4˚C/min to 125˚C, 125˚(2min)
Det: TCD, 300˚C
Flow Rate: 25cm/sec constant pressure He
Inj: 250˚C,1uL, split, 100:1
Liner: 4mm ID cup design split liner
Samples: IL Solvent Test Mix: MeOH, EtOH, Acetone, IPA, n-propanol, 1-butanol, 1,4-Dioxin in water



SLB-IL 107



SLB-IL 94



IL 200

SIGMA-ALDRICH®

IL Solvent Mix on SLB-IL 94 30m x 0.25mm x 0.20umd$_f$

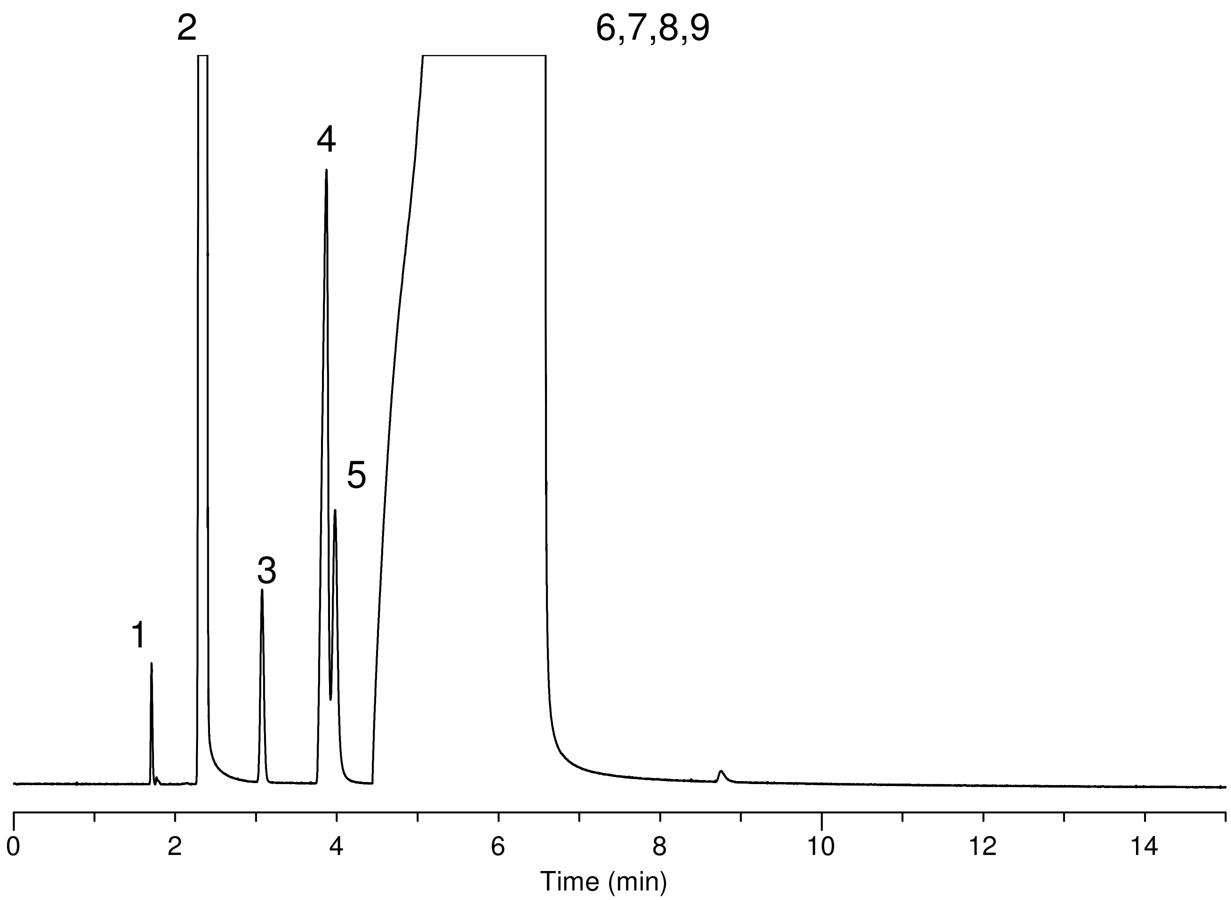


Figure 9. Solvent test standard programmed separation on SLB-IL 94; 1) MeOH, 2) $MeCl_2$ 3) acetone, 4) ethanol, 5) IPA, 6) n-Propanol, 7) 1,4dioxane, 8) butanol 9) water

SIGMA-ALDRICH®

IL Solvent Mix on SLB-IL 107 30m x 0.25mm x 0.20umd_f

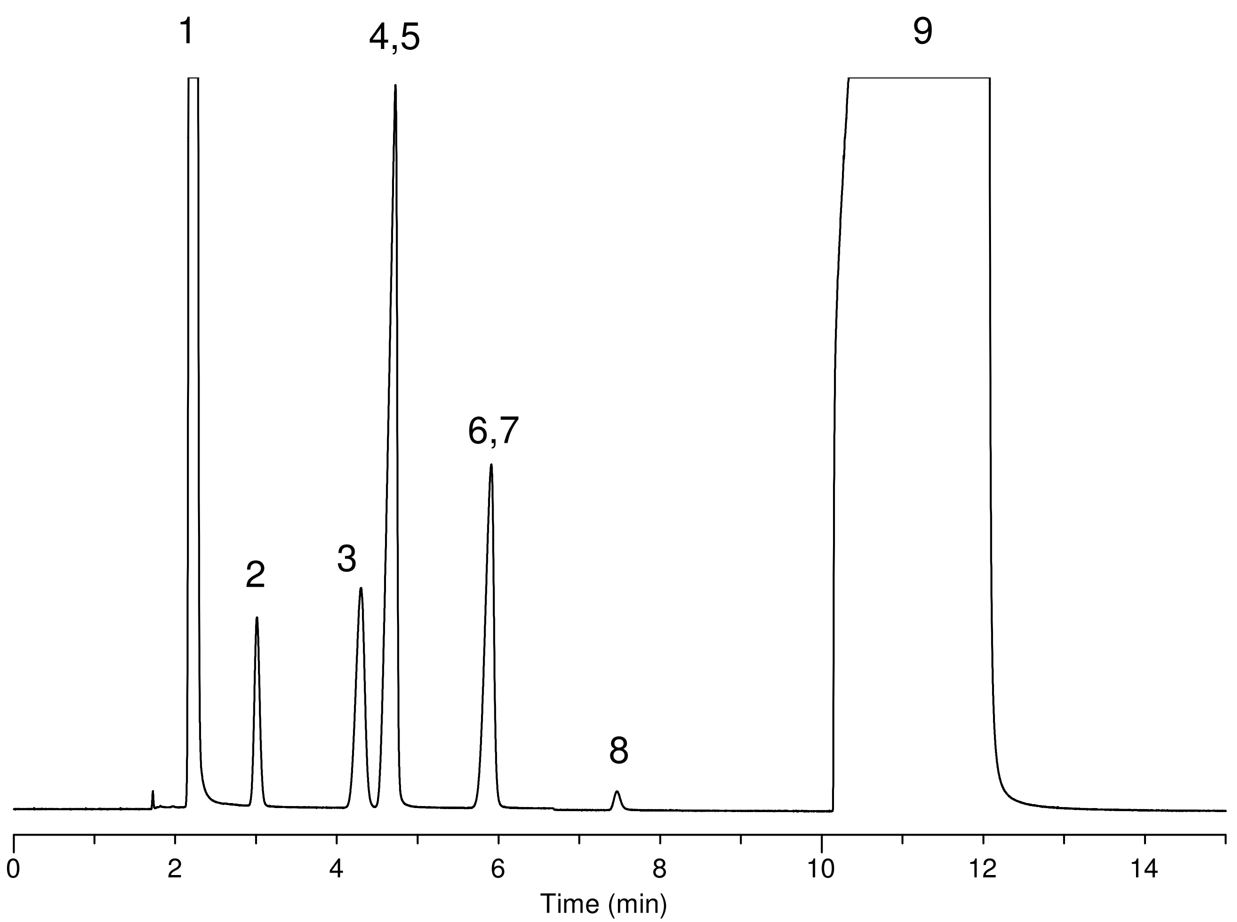


Figure 8. Solvent test standard programmed separation on SLB-IL 107; 1) $MeCl_2$, 2) acetone, 3) IPA, 4) ethanol, 5)methanol, 6) n-Propanol, 7) 1,4dioxane 8) butanol, 9) water

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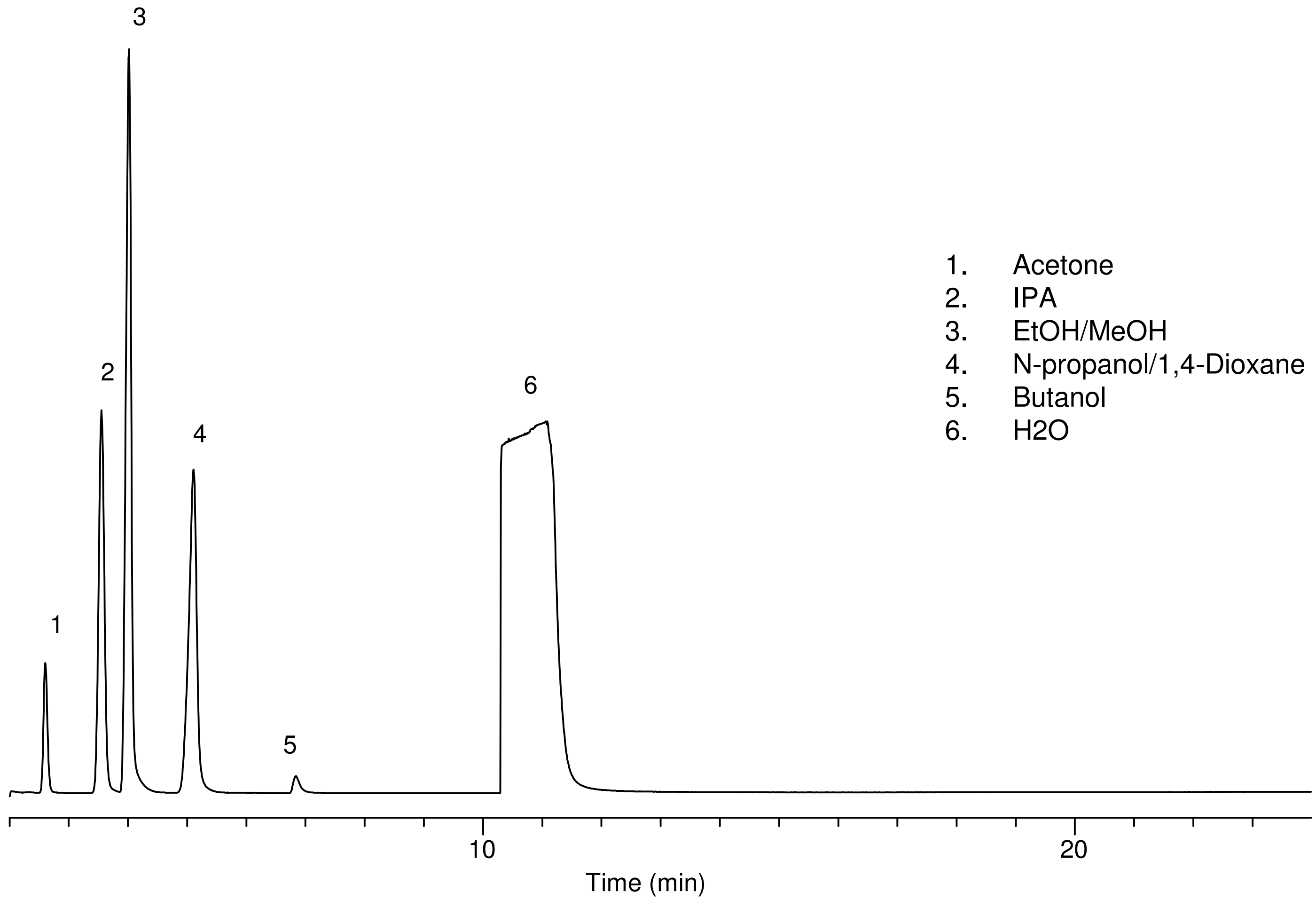
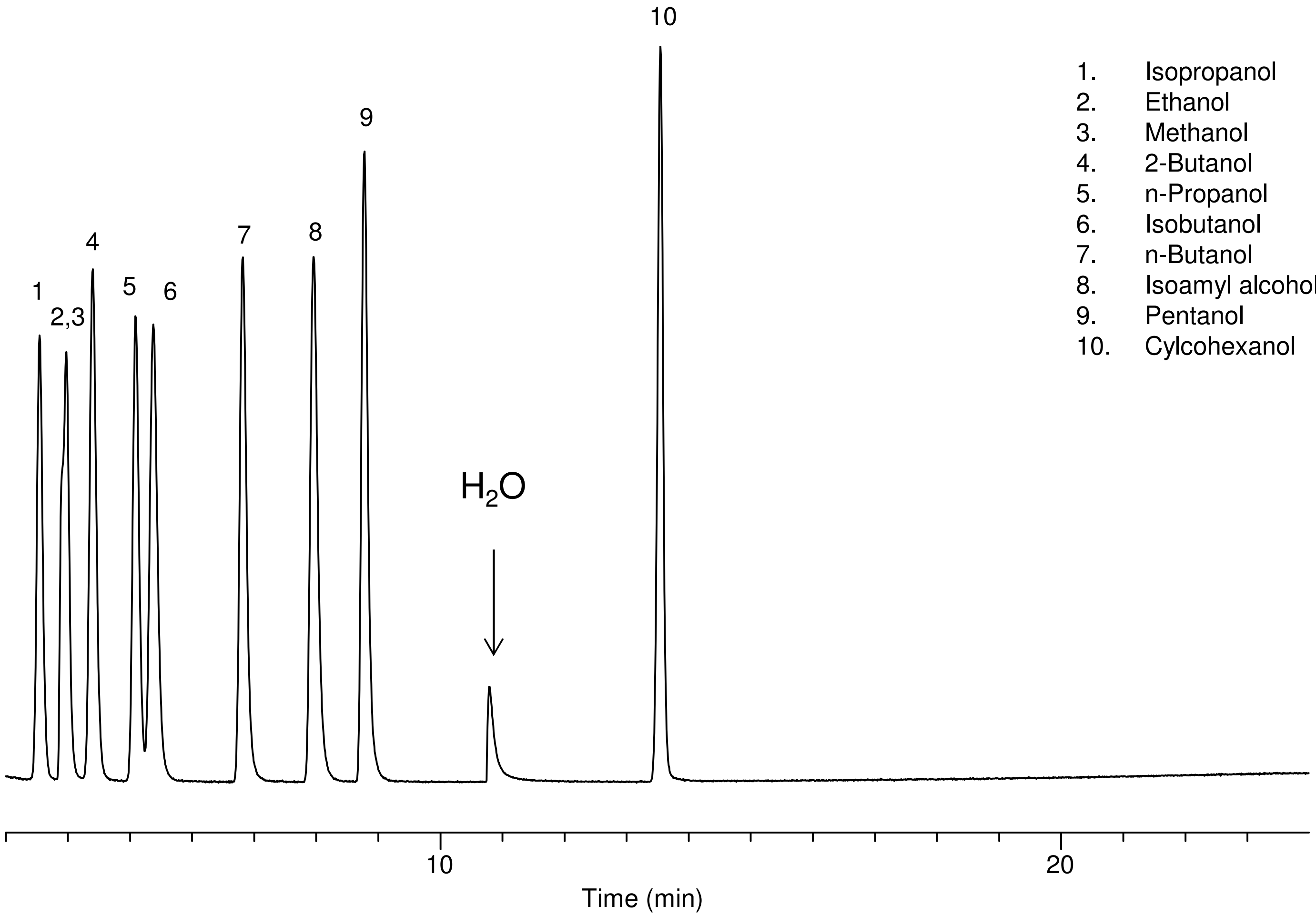
SLB-IL 107 SPME Fiber Test STD



1. Acetone
2. IPA
3. EtOH/MeOH
4. N-propanol/1,4-Dioxane
5. Butanol
6. H2O

Figure 1. Temperature programmed run for SPME Fiber Test Standard on SLB-IL 107. 1uL injection of standard with varied concentrations (10-200ppm) at 100:1 split. Standard is prepared in water.

C1-C6 Alcohol Mix



1. Isopropanol
2. Ethanol
3. Methanol
4. 2-Butanol
5. n-Propanol
6. Isobutanol
7. n-Butanol
8. Isoamyl alcohol
9. Pentanol
10. Cylcohexanol

Figure 2. Temperature programmed run for light alcohol mix on SLB-IL 107. 1uL injection of a 500ug/mL sample at 100:1 split. Note the sample has adsorbed some water in storage.

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Grappa Bassano

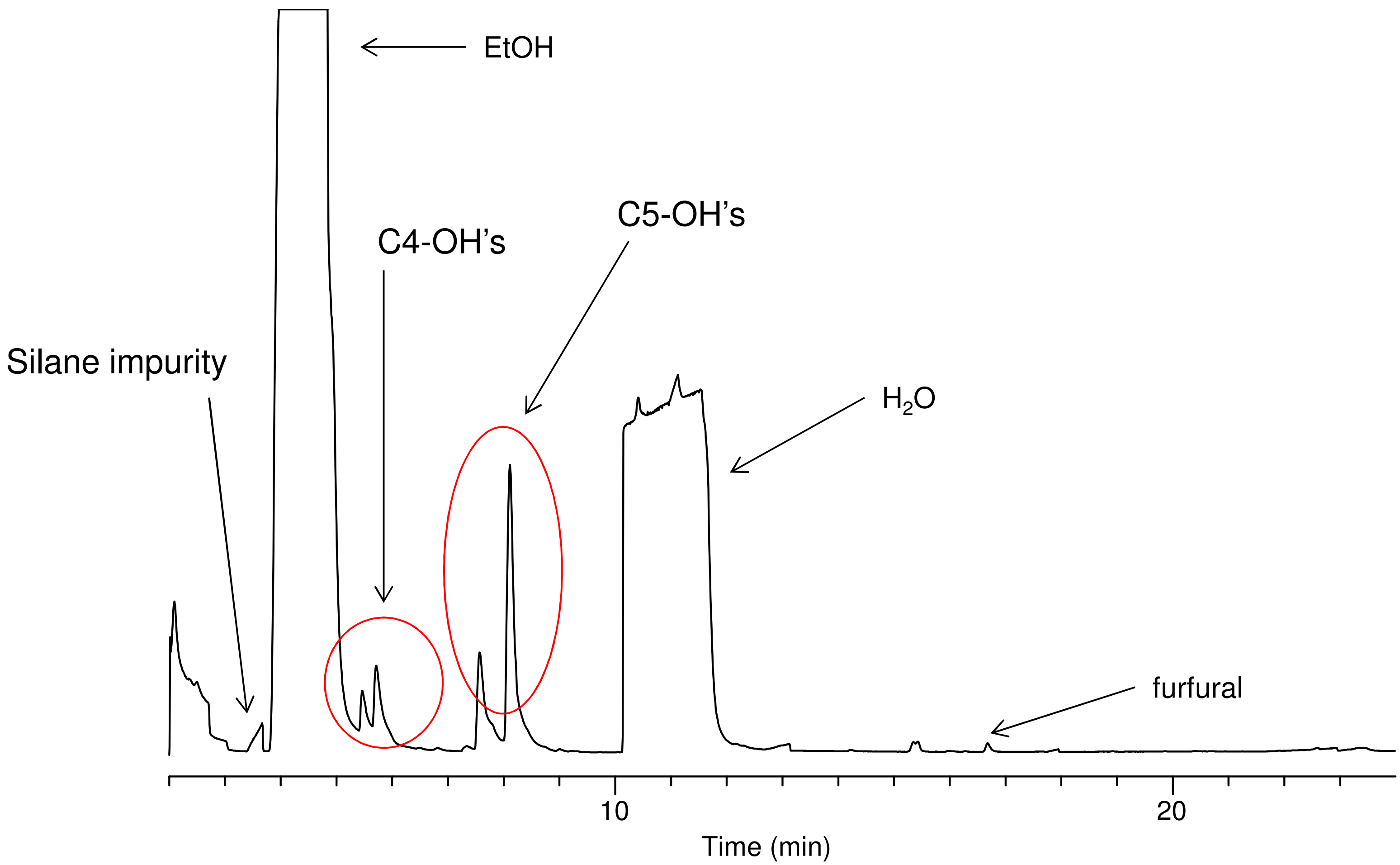

Figure 4. Temperature programmed run for Grappa Bassano on SLB-IL 107. . SPME Carboxen extraction. Selected peaks with high confidence of identification.

Grappino

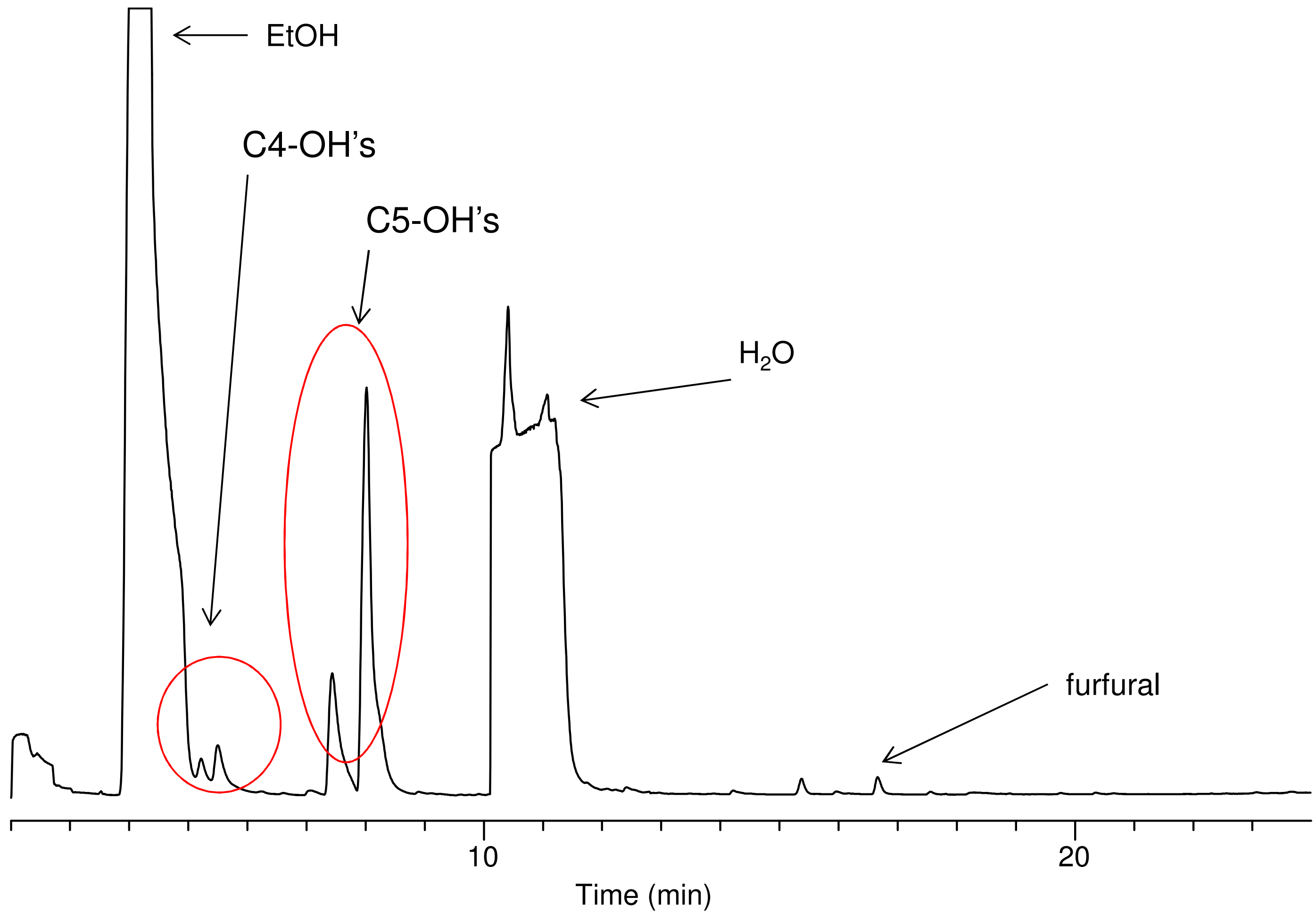

Figure5. Temperature programmed run for Grappino on SLB-IL 107. . SPME Carboxen extraction. Selected peaks with high confidence of identification.

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Tito's Vodka

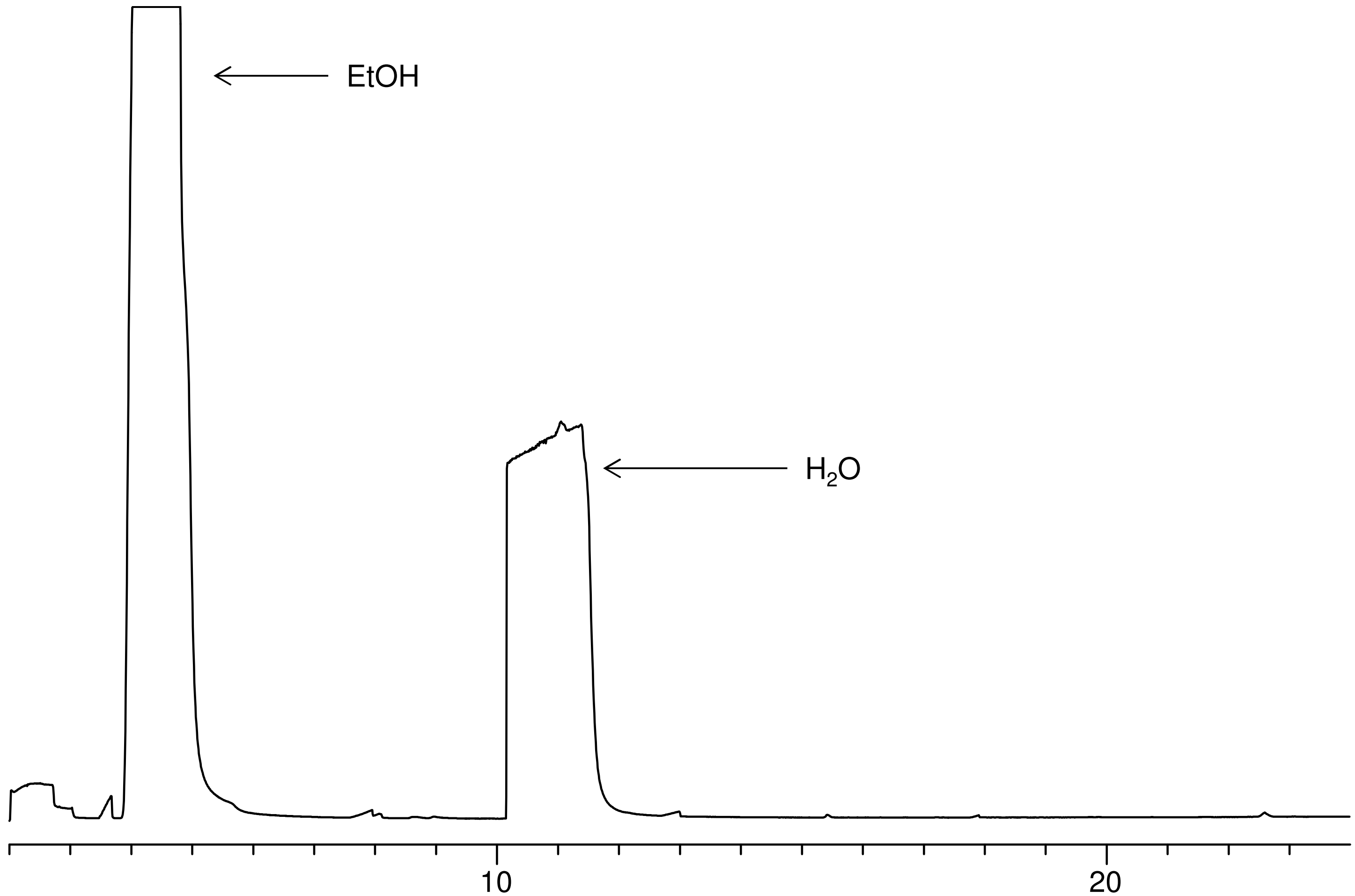

Figure6. Temperature programmed run for Tito's Vodka on SLB-IL 107. . SPME Carboxen extraction. Selected peaks with high confidence of identification.

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Ouzo

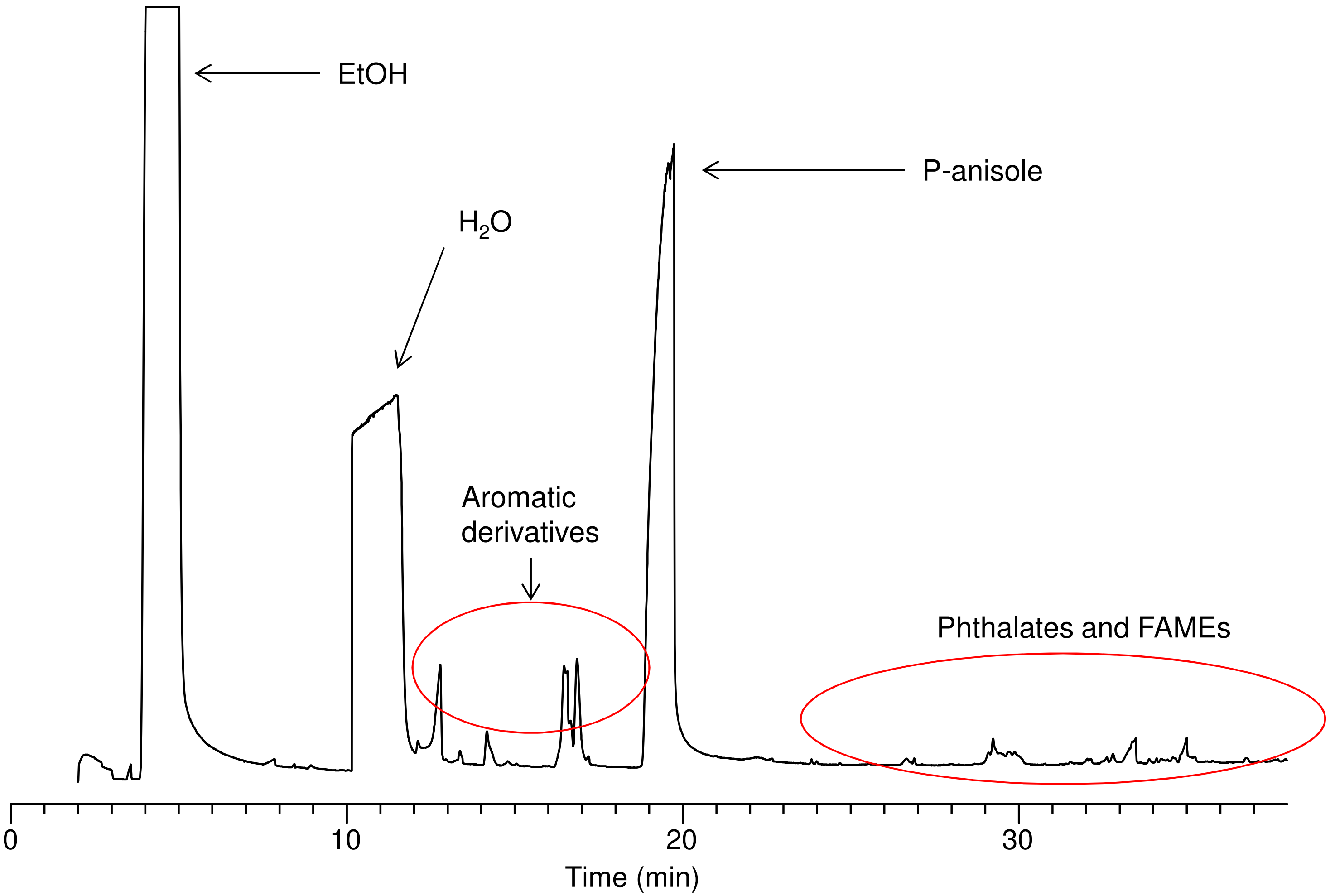


Figure9. Temperature programmed run for Ouzo on SLB-IL 107. . SPME Carboxen extraction. Selected peaks with high confidence of identification.

SIGMA-ALDRICH®

Over-coated PDMS/ DVB SPME Fibers

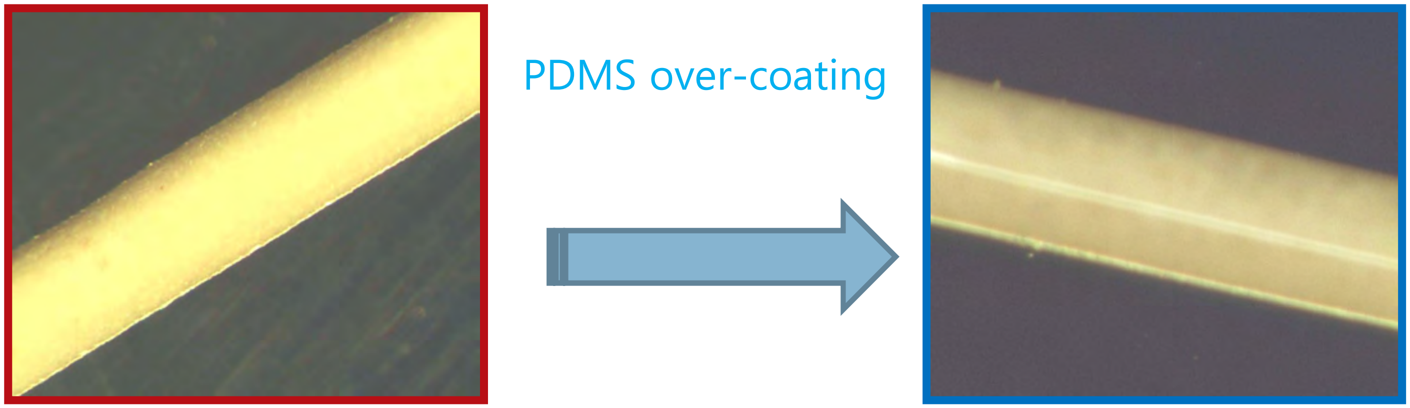
SIGMA-ALDRICH®

Purpose for Over-coating Adsorbent Fibers

1. PDMS over-coating is intended to extend fiber life when fibers are immersed in the matrix solution.
2. Matrix components such as sugars tend to stick to adsorbent coating coatings that reduces fiber life.
3. PDMS coating serves as a barrier to the matrix. The matrix components tend not to stick to PDMS.
4. Analytes tend to migrate through the PDMS coating onto the adsorbent surface or into the pores where they are more tightly retained.
5. Over-coating application seals the ends of the fiber so that matrix does not wick into solution.
6. Fibers are more durable.
7. Less background in chromatograms
8. Reduces matrix competition with analytes

Coating Modification Optimization

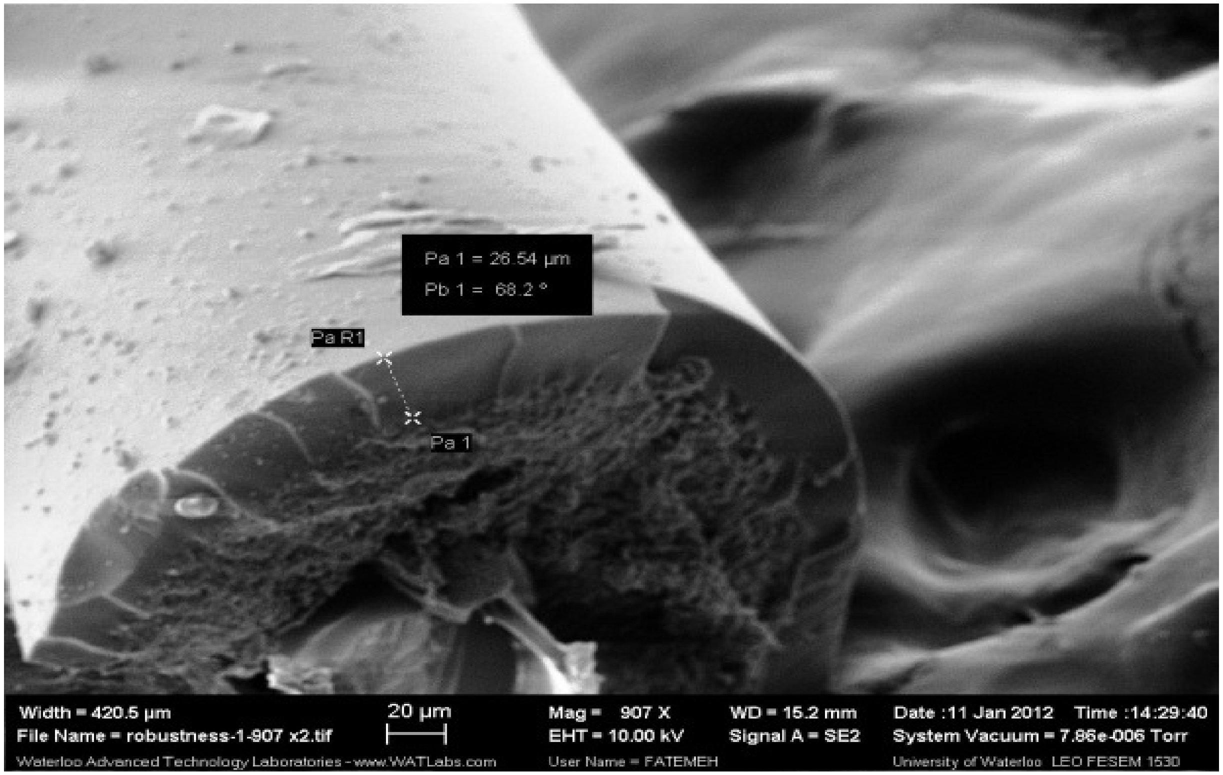
✓ Over-coating standard PDMS/DVB with a PDMS



Microphotographs of a standard PDMS/DVB fiber and the same fiber coated with an external PDMS layer.

E. A. Souza-Silva, J. Pawliszyn, *Anal. Chem.* 84 (2012), 6933-6938.

SEM of Cross Section PDMS-DVB Fiber with 30µm PDMS Overcoat



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