New Developments in Chromatography at Supelco

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

SLB-IL111: Increased retention of cis relative to trans

complimentary selectivity
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 on SLB-IL111 @ 150 °C isothermal
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

SLB-IL60

PEG

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

C18:2n6 tt

C18:2n6 cc

C18:2n6 tt

C18:2n6 cc
Fusel Oils Separation -
Active amyl alcohol (2-methyl-1-butanol) and Isoamyl alcohol (3-methyl-1-butanol), 90°C Isothermal

SLB-IL60

Supelcowax 10
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)

\[ R_s(\text{ethanol/benzene}) = 12.6 \]
\[ R_s(\text{MIBK/n-propanol}) = 45 \]
\[ R_s(\text{toluene/isobutanol}) = 5.6 \]
Reformulated Gasoline with D3606 Oxygenates
50 °C (6 min) to 265 °C (10 min) at 15 °C/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.
PAHs on a Traditional 5% Silphenylene Phase

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)

1. Naphthalene
2. Acenaphthene
3. Acenaphthylene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
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. Dibenzo[a,h]anthracene
16. Indeno[1,2,3-cd]pyrene
17. Benzo[ghi]pyrene
Selected Isomers

- Anthracene/Phenanthrene
- Benzofluoranthene
- Benzo(a)anthracene/Chrysene
- Triphenylene/Chrysene
- Cyclopenta(cd)pyrene/Chrysene
Ionic Liquid Water Separations

Column: SLB-IL 94, SLB-IL 107, IL 200 30m x 0.25mm x 0.20um
Oven: 35°C, 4˚C/min to 125˚C, 125˚C (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
Figure 9. Solvent test standard programmed separation on SLB-IL 94; 1) MeOH, 2) MeCl₂, 3) acetone, 4) ethanol, 5) IPA, 6) n-Propanol, 7) 1,4-dioxane, 8) butanol, 9) water.
Figure 8. Solvent test standard programmed separation on SLB-IL 107; 1) MeCl₂, 2) acetone, 3) IPA, 4) ethanol, 5)methanol, 6) n-Propanol, 7) 1,4dioxane 8) butanol, 9) water
Figure 1. Temperature programmed run for SPME Fiber Test Standard on SLB-IL 107. 1μL injection of standard with varied concentrations (10-200ppm) at 100:1 split. Standard is prepared in water.

1. Acetone
2. IPA
3. EtOH/MeOH
4. N-propanol/1,4-Dioxane
5. Butanol
6. H2O
C1-C6 Alcohol Mix

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.
Figure 4. Temperature programmed run for Grappa Bassano on SLB-IL 107. SPME Carboxen extraction. Selected peaks with high confidence of identification.
Figure 5. Temperature programmed run for Grappino on SLB-IL 107. SPME Carboxen extraction. Selected peaks with high confidence of identification.
Tito’s Vodka

Figure 6. Temperature programmed run for Tito’s Vodka on SLB-IL 107 SPME Carboxen extraction. Selected peaks with high confidence of identification.
**Figure 9.** Temperature programmed run for Ouzo on SLB-IL 107. SPME Carboxen extraction. Selected peaks with high confidence of identification.
Over-coated PDMS/ DVB SPME Fibers
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.

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