Approaches to Increasing GC Speed, Resolution and Response
Overview of Presentation

Fast GC
Ionic liquids for GC
Chiral GC
GC Innovations

• Fast GC – Column dimensions & instrument settings
  • Ionic liquids for GC
  • Chiral GC

Users are.....
• GC and GC-MS analysts

Interested in...
• Decreasing run time
• Maintaining resolution
• Using existing GC systems

Users can expect...
• High speed, high efficiency, adequate resolution GC separations
The Possibilities of Fast GC

Supelco 37-Component FAME (fatty acid methyl ester) Mix

Conventional GC ~40 mins.

10-fold speed improvement, resolution maintained

Fast GC ~4 mins.
Why Use Fast GC?

Fast GC offers four distinct benefits:
1. Reduced analysis time (typically 3- to 10-times faster analyses)
2. Increased sample throughput
3. Faster GC method development
   - Alter analysis conditions and see results faster
4. Improved precision and accuracy
   - Allows more replicates and standards to be analyzed in a shorter time frame

How do we achieve Fast GC?
Foundations: Factors Affecting GC Retention Time

The goal in Fast GC is to reduce the total analysis time without losing resolution. How can this be accomplished?

The following equation defines GC retention time:

\[ t_r = \frac{L (k +1)}{u} \]

There are three options to reduce the retention time (\( t_r \)) of an analyte:

- Reduce column length (L)
- Reduce retention factor (k) by:
  - Changing the stationary phase
  - Increasing temperature
- Increase carrier gas linear velocity (u)

But these factors can reduce efficiency and resolution.
The van Deemter Relationship Visualized

\[ H = A + \frac{B}{u} + Cu \]
\[ H = \frac{L}{N} \]
Foundations: The Golay Equation

The Golay equation is just the classic van Deemter equation minus the A-term, which does not apply to open tubes ($H = \frac{B}{u} + C_u$).

Low values of $H$ are better.

The Golay equation looks complex, but from it a few simple truths relevant to Fast GC are obvious.

1. Smaller column radii give higher efficiency.

2. Thin stationary phase films with high $D_s$ (diffusivity) values give higher efficiency.

3. Carrier gases with high $D_m$ (diffusivity) give higher efficiency. (Use hydrogen.)
How is Fast GC Achieved?

Column and instrument improvements and run conditions that give 3- to 10-times faster analyses, while still giving adequate resolution.

Fast GC column characteristics:
- Short length (< 20 m)
- Narrow I.D. (0.10 mm or 0.18 mm)

Film thickness: any thin film phase can be used
Carrier gas: hydrogen
Programming rates: rapid
What Columns are Used in Fast GC?

Based solely on column internal diameter (I.D.), GC can be grouped into three types:

- **Standard GC**: Columns with I.D. > 0.25 mm
- **Fast GC**: Various I.D. Columns (0.10 and 0.18 mm are typical)
- **Ultra-Fast GC**: Column I.D. < 50 µm

Column lengths are typically < 20 meters

Any thin-film stationary phase can be used
How is Fast GC Achieved?

On standard GC columns (> 0.2 mm I.D.):

- Switch from helium to hydrogen carrier gas.
  - This could reduce runtime by as much as 50%
- Hardware modifications to the GC system that would allow for faster heating and cooling, such as the GC Racer
- Using microwave energy with compatible columns for rapid heating
- Use of resistive heating technology (this requires modified columns and an oven modification as well)

(continued)
How is Fast GC Achieved?

On **short columns with narrow I.D.** (≤0.2 mm)
- High carrier gas linear velocity
- Fast oven temperature programming rates

To offset the losses in efficiency of shorter columns:
- Decrease the column I.D. (reduced “C” term of the Golay)
- Use thinner stationary phase films (less resistance to mass transfer, $C_s$)
- Use hydrogen as the carrier gas (higher $u_{opt}$ than helium or nitrogen)

These parameters must be optimized together! Changing only one may decrease run time, but will likely cause a loss of resolution.
Chromatographer’s Triangle

Optimizing chromatographic parameters requires compromise:

- Speed
- Sensitivity
- Selectivity
Why Column Length Affects Efficiency

Resolution equation:

\[ R_S = \left\{ \frac{k}{1 + k} \right\} \left\{ \frac{(\alpha - 1)/\alpha}{N^{1/2}/4} \right\} \]

Selectivity (\(\alpha\))

Capacity (\(k\))

Efficiency (\(N\))

Relationship between \(L\) and \(N\):

\[ N = \frac{L}{H} \]

Therefore:

- Reducing \(L\), reduces \(N\)
Column Length and Efficiency

Golay plots for long (15 meter) and short (5 meter) capillary columns

- 0.10 mm I.D.
- Hydrogen carrier gas

Short columns give higher efficiency at higher linear velocities
Narrow I.D. Columns: Efficiency Advantages

Typical plate numbers generated by capillary columns of various dimensions.
Also improves s/n ratio.

<table>
<thead>
<tr>
<th>Column ID (mm)</th>
<th>Plate Number ($N_{eff}$)</th>
<th>Plates/meter ($N_{eff}/m$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>219000</td>
<td>7300</td>
</tr>
<tr>
<td>0.18</td>
<td>121500</td>
<td>4050</td>
</tr>
<tr>
<td>0.20</td>
<td>109500</td>
<td>3650</td>
</tr>
<tr>
<td>0.25</td>
<td>87750</td>
<td>2925</td>
</tr>
<tr>
<td>0.32</td>
<td>69000</td>
<td>2300</td>
</tr>
</tbody>
</table>

Theoretical values, calculated at a retention factor of 6.00 and 85% coating efficiency.
Other parameters for Fast GC

Stationary phase & film thickness
- Changing the phase can result in a decrease in analysis time.
- Decreasing the film thickness will decrease the analysis time.

Carrier gas
- Hydrogen is the best choice for Fast GC
  - High diffusivity, high $u_{\text{opt}}$

Carrier gas linear velocity
- Increasing the carrier gas linear velocity will increase the speed of analysis.
- Loss of resolution can occur if the speed is increased much higher than the optimal velocity for the carrier gas.

(continued)
Golay Plots: Comparing Carrier Gases

Using hydrogen and higher linear velocity will improve efficiency. Hydrogen also has a flatter Golay relationship than helium and gives better efficiency at higher linear velocities.
Column I.D. and Optimum Linear Velocity

As this figure shows, columns with narrow I.D. have higher efficiency (lower HETP) and can be operated at higher linear velocities than larger I.D. columns.

From P. Sandra and C. Bicchi, “Capillary Gas Chromatography in Essential Oil Analyses,” Huethig, 1987
Analysis temperature & temperature program rate:

- Increasing the analysis temperature for an isothermal analysis will decrease analysis time.
- It may result in a loss of resolution if the temperature increase is too high.
- A faster temperature program rate will decrease analysis time, but may result in a loss of resolution.
- 0.10 mm I.D. capillary columns typically require a program rate of 1.5 to 2 times faster than a wider bore column to retain their inherent efficiency.
BTEX compounds

0.10 mm I.D. column provides over 10-times faster analysis than the 0.25 mm I.D. column

Columns: Supelcowax 10, 30m x 0.25mm ID, 0.25μm film
or 5m x 0.10mm ID, 0.1μm film
Oven: 60°C
Carrier: 0.25mm ID: helium, 25cm/sec, set at 60°C
0.10mm ID: helium, 50cm/sec, set at 60°C
Det.: FID, 250°C
Inj.: 1μL split, 100:1 (0.25mm ID) or 200:1 (0.10mm ID), 250°C

Narrow column I.D. (0.1 mm) and high linear velocity

Fast GC conditions

0.25 mm I.D. column
He, 25 cm/sec.

1. Benzene
2. Toluene
3. Ethylbenzene
4. p-Xylene
5. m-Xylene
6. o-Xylene

0.10 mm I.D. column
He, 50 cm/sec.
Focus on Fast GC in Food Labs

Challenges in food analysis:
- Labeling requirements
- More detailed analysis
- More samples to analyze
- Resolve positional cis/trans isomers
- Trans fats and the Omega 3 and 6 fatty acids (as FAMEs)
  - AOAC Method 996.06
  - 100 meter capillary column
  - Expensive & time-consuming
Fatty Acid Methyl Esters – 0.25 mm I.D. Column (Conventional GC)

Analysis time on the conventional 0.25 mm I.D. column is nearly one hour.
Fatty Acid Methyl Esters – 0.10 mm I.D. Column (Fast GC)

Analysis time on the 0.10 mm I.D. columns, shown here at two different temperature programs, is as much as 20-times faster than the 0.25 mm I.D. column.
Aroclors: Fast GC Analysis

6 minutes analysis on 0.10 mm I.D. column.

Peak ID:
1. 2,4,5,6-Tetrachloro-m-xylene (surr.)
2. Aroclor 1016
3. Aroclor 1260
4. Decachlorobiphenyl (surr.)

Column: Equity-5, 15 m x 0.10 mm I.D., 0.10 µm
Oven: 100°C (0 min), 50°C/min. to 200°C (0 min.), 35°C/min. to 360°C (1 min.)
Inj.: 225°C
Det.: ECD, 360°C
Carrier: Hydrogen, 30 cm/sec. constant
Injection: 2µL, splitless (0.75 min.)
Liner: 4mm I.D. single taper
Sample: 200ppb of Aroclors® 1016 & 1260 with surrogates at 20ppb
Organochlorine Pesticides: 0.10 mm I.D. Column (Fast GC)

6 minutes analysis on 0.10 mm I.D. column.

Column: Equity-5, 15 m x 0.10 mm I.D. x 0.10 µm
Oven: 100 °C, 50 °C/min. to 200 °C, 35 °C/min. to 360 °C (1 min.)
Inj.: 225 °C
Det.: ECD, 360 °C
Carrier: Hydrogen, 30 cm/sec. constant
Injection: 2µL, splitless (0.75 min.)
Liner: 4 mm I.D., single taper
Sample: 50 ppb of a 22 component chlorinated pesticide standard in n-hexane
Instrument Requirements for Fast GC

Fast automatic injection
Split/splitless inlets
Fast ramp rate capability
High-speed detectors
Fast data handling
Method translation software

Agilent’s 6890 and 6850 systems accommodate fast GC separations. Most GC instrument suppliers have Fast GC models.
## Fast GC Dimensions

<table>
<thead>
<tr>
<th>Phase</th>
<th>I.D. (mm)</th>
<th>Length (m)</th>
<th>df (μm)</th>
<th>Beta Value</th>
<th>Cat. No.</th>
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<tr>
<td>SPB-624</td>
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<td>1.0</td>
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<tr>
<td>VOCOL</td>
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<td>20</td>
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<td>45</td>
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<td>SLB-5ms</td>
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<td>0.10/0.18</td>
<td>250/250/250/250</td>
<td>28465-U/28466-U/28564-U/28566-U</td>
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<tr>
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<td>TCEP</td>
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<td>0.18</td>
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<td>SP-2560</td>
<td>0.18</td>
<td>75</td>
<td>0.14</td>
<td>321</td>
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<tr>
<td>SUPELCOWAX 10</td>
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For more information, visit [http://www.sigma-aldrich.com/gc](http://www.sigma-aldrich.com/gc)
GC Innovations

• Fast GC – Special column dimensions
• Ionic liquids for GC – SLB-IL100
• Chiral GC
Ionic Liquids

Ionic liquids - a class of ionic solvents with low melting points. Unique combination of cations and anions that can provide different selectivities when used as stationary phases in GC. Numerous combinations of cations and anions are possible allowing for “tailored” selectivity, application or function.

Featured in August 2009 LC/GC:

Columns
COLUMN WATCH
The Advent and Potential Impact of Ionic Liquid Stationary Phases in GC and GC×GC
Daniel W. Armstrong, Tharanga Payagala, and Leonard M. Sidisky
Ionic liquids appear to have some very good properties for GC stationary phases. Guest columnists explore how they can be tweaked to mimic the popular stationary phases of today.
Desirable Ionic Liquid Properties for GC Use

Several properties make ionic liquids desirable as GC stationary phases:
1. Remain liquid over a wide temperature range (ambient → 350°C)
2. Very low volatility
3. Highly polar nature
4. Broadest range of solvation interactions of any known solvent
5. Good thermal stability for an intermediate to high polarity phase
6. High viscosity
7. Easily tailored to provide different polarities/selectivities
Ionic Liquids Characteristics as GC Phases

Compared to conventional GC phases:

• Selectivity differences:
  – Intermediate to extremely polar
  – Multiple solvation interactions yields unique selectivity

• Stability & operating range:
  – Higher thermal stability than traditional phases of similar polarity
  – Lower column bleed
  – Simple m/z bleed fragmentation patterns (smaller m/z fragments)
  – Stable retention times
  – Long column life
  – Lower minimum operating temperature
  – Resistant to damage from moisture/oxygen in carrier gas/samples
  – Less prone to damage from acidic/basic compounds in samples
GC column polarity scale:

0 = squalane (considered the least polar GC stationary phase)
100 = TCEP (considered the most polar GC stationary phase)

Siloxane-based

<table>
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<th>Temperature</th>
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<tr>
<td>360°C</td>
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<td>Non-Polar</td>
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<tr>
<td>310°C</td>
<td>20</td>
<td>Intermediate Polar</td>
</tr>
<tr>
<td></td>
<td>1701</td>
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<tr>
<td></td>
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</tr>
<tr>
<td></td>
<td>50</td>
<td></td>
</tr>
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Wax

280°C Wax (PEG)

Cyanosilicones

<table>
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<th>Value</th>
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</thead>
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<td>2331</td>
</tr>
<tr>
<td>250°C</td>
<td>2560</td>
</tr>
<tr>
<td>140°C</td>
<td>TCEP</td>
</tr>
</tbody>
</table>

Range of alternative polarities possible from ionic liquid GC
Ionic Liquid Stationary Phases for GC

ILs for GC possess:
- Cation (imidazolium shown)
- Anion (nTF$_2^-$ shown)
- Linker or connecting group
- Functional groups on the cation

SLB-IL100 – Geminal dicationic ionic liquid
1,9-di(3-vinyl-imidazolium) nonane bis(trifluoromethyl) sulfonyl imidate

imidazolium cation

C9 linkage

R-group (vinyl)

bis(trifluoromethyl) sulfonyl imidate (nTF$_2^-$) anion counterion
SP-2560: 22 Isomers, AOCS Conditions
180°C, H₂ at 25 cm/s in ~ 32 min.

Column: SP-2560, 100 m x 0.25 mm I.D. 0.20mm film; Inj. vol.: 1µL; Sample: C18:1 isomer mix + Linolenic acid methyl ester isomer mix (Supelco Cat. No. 47792) + Linoleic acid methyl ester mix, cis/trans (Supelco Cat. No. 47791) in dichloromethane. Split ratio: 1:20 (220 °C); Temp. progr.: Isothermal at 180°C; Press. progr.: 161.6 kPa at linear velocity constant; Carrier gas: H₂; u: 25.0 cm/s; Detector: FID (220 °C) H₂: 50 mL/min., Air: 400 mL/min, Make-up: 50 mL/min kPa (N₂); Sampling rate: 80 msec; Filter Time Constant: 200 msec
SLB-IL100: Ionic Liquid GC Phase
150°C, H₂ at 30 cm/s in ~ 19 min.

Column: SLB-IL100 30 m x 0.25 mm I.D., 0.20mm film; Inj. vol.: 2 µL; Sample: C18:1 Isomer mix + Linolenic acid methyl ester isomer mix (Supelco Cat. No. 47792) + Linoleic acid methyl ester mix, cis/trans (Supelco Cat. No. 47791) in dichloromethane. Split ratio: 1:20 (240 °C); Temp. progr.: Isothermal at 150°C; Press. progr.: 52.8 kPa at linear velocity constant; Carrier gas: H₂; u: 30.0 cm/s; Detector: FID (240 °C) H₂: 50 mL/min, Air: 400 mL/min, Make-up: 50 mL/min kPa (N₂); Sampling Rate: 80 msec; Filter Time Constant: 200 msec
TCEP Mix on TCEP Column 110 °C

1. n-Tridecane
2. Toluene
3. Ethylbenzene
4. p-Xylene
5. Isopropylbenzene (Cumene)
6. 1,2,4-Trimethylbenzene
7. 1,2,4,5-Tetramethylbenzene (Durene)
8. Cyclohexanone
TCEP Mix 110 °C Isothermal

Ionic Liquid 6

Ionic Liquid 10

1. n-Tridecane
2. Toluene
3. Ethylbenzene
4. p-Xylene
5. Isopropylbenzene (Cumene)
6. 1,2,4-Trimethylbenzene
7. 1,2,4,5-Tetramethylbenzene (Durene)
8. Cyclohexanone
Rapeseed Oil – SP-2560

Column: SP-2560, 15 m x 0.10 mm I.D. x 0.08 µm
Oven: 125 °C (0 min.), 25 °C/min. to 245 °C (1 min.)
Inj.: 220 °C
Det.: 260 °C (FID)
Carrier: hydrogen, 45 cm/sec., constant flow
Injection: 0.1 µL, split 300:1
Liner: 4 mm I.D. cup split
Sample: rapeseed oil reference mix (methylated)
(Cat. No. O7756-1AMP), diluted to 10 mg/mL in methylene chloride

Peak List
1. Myristic (C14:0)
2. Palmitic (C16:0)
3. Stearic (C18:0)
4. Oleic (C18:1n9c)
5. Linoleic (C18:2)
6. Linolenic (C18:3)
7. Arachidic (C20:0)
8. cis-11-Eicosenoic (C20:1)
9. Behenic (C22:0)
10. Erucic (C22:1)
11. Lignoceric (C24:0)
Rapeseed Oil FAMEs
180 °C Isothermal

SLB-IL100

6. Linolenic (C18:3)
7. Arachidic (C20:0)
8. cis-11-Eicosenoic (C20:1)

Ionic Liquid 6

Ionic Liquid 10
Rapeseed Oil FAMEs on Ionic Liquid 15
180 °C Isothermal

5. Linoleic (C18:2)
6. Linolenic (C18:3)
7. Arachidic (C20:0)
8. cis-11-Eicosenoic (C20:1)
9. Behenic (C22:0)
1. Undecane
2. Benzene
3. Tridecane
4. Toluene
5. Ethyl benzene
6. m-Xylene
7. p-Xylene
8. o-Xylene

Elution of toluene (C7) after tridecane (C13) highlights the great selectivity for aromatics, allowing the separation of aliphatic and aromatic analytes in separate fractions.

Shorter analysis time compared to non-ionic liquid columns.

column: SLB-IL100, 30 m x 0.25 mm I.D., 0.20 µm (28884-U)
oven: 110 °C
inj.: 250 °C
det.: FID, 250 °C
carrier gas: helium, 26 cm/sec @ 110 °C
injection: 0.1 µL, 300:1 split
liner: 4 mm I.D., split, cup
sample: NEAT mixture containing varying percentages of each component
PCB Congener Standard (Biphenyl through Deca) on SP-2331
PCB Congener Standard (Biphenyl through Deca) on the SLB-IL100
Run Conditions for PCB Congeners

Columns: SP-2331, 30 m x 0.25 mm I.D., 0.20 µm d<sub>f</sub>
        SLB-IL100, 30 m x 0.25 mm I.D., 0.20 µm d<sub>f</sub>
Oven:  60 °C (1 min.), 8 °C/min. to 230 °C
Inj.:  250 °C
MSD interface: 220 °C
Carrier Gas: helium, 1.5 mL/min. constant flow
Injection: 1 µL, splitless (splitter open at 1 min.)
Liner:  4 mm I.D. single taper
Sample: PCB congener mix, 2.5 ppm each cpd. in n-hexane
PCB Congener ID’s

#0: Biphenyl
#1: 2-monochlorobiphenyl
#3: 4-monochlorobiphenyl
#10: 2,6-dichlorobiphenyl
#15: 4,4’-dichlorobiphenyl
#30: 2,4,6-trichlorobiphenyl
#37: 3,4,4’-trichlorobiphenyl
#54: 2,2’,6,6’-tetrachlorobiphenyl
#77: 3,3’,4,4’-tetrachlorobiphenyl
#104: 2,2’,4,6,6’-pentachlorobiphenyl
#126: 3,3’,4,4’,5-pentachlorobiphenyl

#155: 2,2’,4,4’,6,6’-hexachlorobiphenyl
#169: 3,3’,4,4’,5,5’-hexachlorobiphenyl
#188: 2,2’,3,4’,5,6,6’-heptachlorobiphenyl
#189: 2,3,3’,4,4’,5,5’-heptachlorobiphenyl
#202: 2,2’,3,3’,5,5’,6,6’-octachlorobiphenyl
#194: 2,2’,3,3’,4,4’,5,5’-octachlorobiphenyl
#208: 2,2’,3,3’,4,5,5’,6,6’-nonachlorobiphenyl
#206: 2,2’,3,3’,4,4’,5,5’,6-nonachlorobiphenyl
#209: Decachlorobiphenyl
MS spectrum of baseline at 230 °C

Simpler (smaller) m/z bleed fragments (compared to predominant m/z 207 and m/z 281 typically formed by polysiloxane polymers) results in easier mass spectral identifications due to less interference.

column: SLB-IL100, 30 m x 0.25 mm I.D., 0.20 µm (28884-U)
oven: 60 °C (1 min.), 8 °C/min. to 230 °C (5 min.)
inj.: 250 °C
MSD interface: 220 °C
scan range: m/z = 35-500
carrier gas: helium, 1.5 mL/min. constant
injection: 1 µL, splitless (1.0 min.)
liner: 4 mm I.D., single taper
Ionic Liquid GC Phases

Higher maximum temperature
Lower column bleed
Smaller m/z bleed fragments
Stable retention times
Long column life
Lower minimum temperature
Resistant to damage from moisture/oxygen in carrier gas/samples
Less prone to damage from acidic/basic compounds in samples
Multiple solvation interactions

Supelco SLB-IL100:
- 15 m x 0.10 mm I.D., 0.08 µm df (28882-U)
- 20 m x 0.18 mm I.D., 0.14 µm df (28883-U)
- 30 m x 0.25 mm I.D., 0.20 µm df (28884-U)
- 60 m x 0.25 mm I.D., 0.20 µm df (28886-U)
- 30 m x 0.32 mm I.D., 0.26 µm df (28887-U)
- 60 m x 0.32 mm I.D., 0.26 µm df (28888-U)

IL36:
- 15 m x 0.10 mm I.D., 0.08 µm df (28880-U)
- 30 m x 0.25 mm I.D., 0.20 µm df (28891-U)

http://www.sigma-aldrich.com/gc
Ionic Liquid GC Phases

Currently the project is aimed at identifying research partners to develop novel uses for ionic liquid GC phases

More information and training will follow in the upcoming months
GC Innovations

• Fast GC – Special column dimensions
• Ionic liquids for GC – SLB IL100
• Chiral GC – CHIRALDEX and Supelco DEX

Users are...
• GC and GC-MS analysts
• Performing chiral GC separations

Interested in...
• High enantioselectivity
• Using existing GC systems

Users can expect...
• High enantioresolution
• Choices in selectivities
Enantiomers

- Stereoisomers that differ in the direction they rotate a plane of polarized light are called optically active, or chiral, and their isomers are called enantiomers.
- Non-superimposable mirror images
- Biological systems recognize chirality → analysis

Two chiral molecules (enantiomers). The asymmetric carbon is the purple atom in the middle.
The Cyclodextrin Molecule

http://www.lsbu.ac.uk/water/images/cyclodex.gif
# Chiral GC Product offering: CD Derivatives

<table>
<thead>
<tr>
<th>Column</th>
<th>Type of CD</th>
<th>Derivative type</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHIRALDEX B-PM</td>
<td>β</td>
<td>Permethy 1</td>
</tr>
<tr>
<td>α, β, γ-DEX 110/120</td>
<td>α, β or γ</td>
<td>Permethy 1</td>
</tr>
<tr>
<td>CHIRALDEX B-, G-DM</td>
<td>β or γ</td>
<td>Dimethyl</td>
</tr>
<tr>
<td>α, β, γ-DEX 325</td>
<td>α, β or γ</td>
<td>Dimethyl</td>
</tr>
<tr>
<td>CHIRALDEX A-, B-, G-DA</td>
<td>α, β or γ</td>
<td>Dialkyl</td>
</tr>
</tbody>
</table>

### Non-polar chiral GC columns

<table>
<thead>
<tr>
<th>Column</th>
<th>Type of CD</th>
<th>Derivative type</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHIRALDEX A-, B-, G-TA</td>
<td>α, β or γ</td>
<td>Trifluoroacetyl</td>
</tr>
<tr>
<td>α, β, γ-DEX 225</td>
<td>α, β or γ</td>
<td>Diacetyl</td>
</tr>
<tr>
<td>CHIRALDEX B-, G-DP</td>
<td>β or γ</td>
<td>Dipropionyl</td>
</tr>
<tr>
<td>CHIRALDEX G-PN</td>
<td>γ</td>
<td>Propionyl</td>
</tr>
<tr>
<td>CHIRALDEX G-BP</td>
<td>γ</td>
<td>Butyryl</td>
</tr>
<tr>
<td>CHIRALDEX B-PH</td>
<td>β</td>
<td>S-Hydroxypropyl</td>
</tr>
</tbody>
</table>

### Polar chiral GC columns
Orthogonality of Chiral GC Phases

oven: 60 °C (0 min. hold) program @ 2 °C/min. to 160 °C (hold 20 min.)
inj.: 250 °C
det.: FID, 250 °C
carrier gas: helium, 25 to 30 cm/sec. set @ 75 °C
injection: 1 μL, split 100:1

1. methyl lactate
2. β-butyrolactone
3. 1-phenylethanol δ(+)
4. l(-)- carvone
5. α-ionone
6. S(+), R(-) methyl mandelate
7. 1-octanolactone
Examples of Chiral GC Separations

Separation of 2,5-Dimethoxytetrahydrofuran

- $t_1 = 5.98$
- $t_2 = 7.33$
- $t_3 = 8.85$ (meso)

Separation of Chiral Amines

<table>
<thead>
<tr>
<th>Rt</th>
<th>Area</th>
<th>Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.12</td>
<td>765562</td>
<td>R-Amphetamine</td>
</tr>
<tr>
<td>10.86</td>
<td>766171</td>
<td>S-Amphetamine</td>
</tr>
<tr>
<td>11.41</td>
<td>900479</td>
<td>S-Methamphetamine</td>
</tr>
<tr>
<td>11.92</td>
<td>902574</td>
<td>R-Methamphetamine</td>
</tr>
<tr>
<td>13.06</td>
<td>668337</td>
<td>d-Pseudoephedrine</td>
</tr>
<tr>
<td>13.93</td>
<td>736383</td>
<td>l-Pseudoephedrine</td>
</tr>
</tbody>
</table>

CHIRALDEX G-BP (20m)
- 60°C, Isothermal
- Nitrogen @ 6 psi
- Split Ratio 100/1

CHIRALDEX G-PN (30m)
- 130°C
- Helium @ 35 psi
- Split Ratio 100/1
The CHIRALDEX powerhouse trio

These three CHIRALDEX columns will accomplish most Chiral GC separations

- **CHIRALDEX G-TA**
  - γ cyclodextrin derivatized with trifluoroacetate functional groups
  - Separates the greatest number and widest variety of compounds

- **CHIRALDEX B-DM**
  - ß cyclodextrin derivatized with methyl functional groups
  - A second generation dimethylated column, ideal for underivatized acids and bases

- **CHIRALDEX B-DA**
  - ß cyclodextrin derivatized with dialkyl functional groups
  - Size selective, so it can separate analytes based on structural differences

[http://www.sigma-aldrich.com/chiral](http://www.sigma-aldrich.com/chiral)
Supelco Chiral Services

Chiral column screening (HPLC & GC)
Method optimization
Small-scale purification (<10 grams of each enantiomer)
Larger scale through our SAFC partners

http://www.sigma-aldrich.com/chiral
Summary

Fast GC – Dimensions and phases
Ionic liquids for GC – SLB-IL100 and IL36
Chiral GC – CHIRALDEX and Supelco-DEX
Acknowledgements/Collaborators

Prof. Daniel Armstrong, U. Texas Arlington
Prof. Luigi Mondello, U. Messina, Messina, Italy
Supelco and Fluka R&D Teams

For more information on the subjects presented here, please contact techservice@sial.com or your regional sales team.