7890A – An Essential Partner for the Trace Analysis of Complex Matrices

• Backflush – An Essential Tool for Complex Matrices
  – QuickSwap option for MS transferline
  – Purged Ultimate Union Backflush option for the GC
  – Backflush App Notes
Backflush: Many Advantages for GC/MS Analysis of Complex Samples (‘Dirty Matrices’)

• Provides more consistent GC retention times
• Provides better, more consistent MS spectra through a sample sequence
  – Reduces chemical noise that may increase during a sequence of samples due to small carryover of matrix from sample to sample
  – Higher quality quantitation (no increase in interfering ions during analysis sequence)
• Reduces contamination (and cleaning frequency) for the source
• Reduces analysis time (more samples per day)
• Increases lifetime of analytical column
Analytical Reality of GC/MS/MS Methods

• With the selectivity of MS/MS, the user cannot see the matrix
  – Hundreds and even thousands of matrix peaks are “invisible” in MS/MS mode
• MS/MS users want the LOWEST detection limits, so they inject even more sample into the GC/MS/MS system with PTV in LVI mode
  – “Invisible” matrix peaks are even MORE intense
• Many late eluting peaks are not “chromatographically ideal” and leave a residue throughout the column
  – This residue increases with each subsequent injection
  – Matrix carryover increases as more samples are injected
• Heavy matrix contaminates the source faster and sensitivity is LOST!
GC/MS/MS needs backflush as much or more than GC/MS to avoid “invisible” problems:

- source contamination
- loss of sensitivity

And to avoid the visible problems:

- changing retention times
Without Backflush: A Serious Problem

After only 3 samples, the background is significantly higher (increase chemical noise is every spectrum).

Overlay of two chromatograms of a blank extract injected BEFORE (A) and AFTER (B) three injections without backflush.

Data provided by MSD user in Almeria, Spain.
Without Backflush: Changes in Retention Time

Highly retained matrix is altering the column selectivity and changing the retention time.

Overlay of two chromatograms of a blank extract injected BEFORE (A) and AFTER (B) three injections without backflush.

Data provided by MSD user in Almeria, Spain.
Without Backflush: Increased Background

Overlay of two chromatograms of a blank extract injected BEFORE (A) and AFTER (B) three injections without backflush

Increased background due to carryover of concentrated matrix

Data provided by MSD user in Almeria, Spain
With Backflush: No Increased Background (Less Spectral Noise) and Consistent Retention Times

Overlay of three chromatograms of lettuce extract run with 2 min of back flush
Agilent’s 7890A CFT Advantage for Backflush

• Multiple ways to setup the backflush using Capillary Flow Technologies
  – QuickSwap
  – Deans Switch
  – 2-Way Splitter with Makeup
  – 3-Way Splitter with Makeup
  – The new Purged Ultimate Union
New Purged Ultimate Union: Analysis Mode

Column 1 and 2 could be different phases and different dimensions

Available Oct'08
Purged Ultimate Union: Backflush Mode

- Decrease inlet pressure during backflush
- Capillary Flow Technology Device
- 15-m HP-5ms (0.25mm id x0.25um)
- 5975C MSD EI mode

Agilent Technologies
Backflush (BF) with the Purged Ultimate Union

Sample - no backflush

Sample - with backflush

Solvent blank - no backflush

a = begin backflush (last peak exits column 1)
b = increase flow 4 mL/min
## Comparison of Backflush Configurations

<table>
<thead>
<tr>
<th>Backflush (BF) Pressure (psi)</th>
<th>QuickSwap (QS) 30 m column; restrictor (0.092mm*)</th>
<th>Purged Ultimate Union Two 15 m columns</th>
<th>Time savings (N x)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>BF flow (mL/min)</td>
<td>1 column vol (min)</td>
<td>MDS flow (mL/min)</td>
</tr>
<tr>
<td>10.0</td>
<td>0.3</td>
<td>3.82</td>
<td>1.5</td>
</tr>
<tr>
<td>20.0</td>
<td>0.7</td>
<td>1.89</td>
<td>2.9</td>
</tr>
<tr>
<td>30.0</td>
<td>1.3</td>
<td>1.28</td>
<td>4.8</td>
</tr>
<tr>
<td>40.0</td>
<td>2.1</td>
<td>0.98</td>
<td>7.2</td>
</tr>
</tbody>
</table>

* smallest restrictor => best QS case

4x faster & lower flow!

T=300°C; head pressure =0.5psi
Sensitivity Without and With Purged Ultimate Union

- Because there is no flow dilution (or less), there is “no” sensitivity lost ...

**No PCT**
- \( S \text{ (ht)} = 12600 \ (\pm 3.4\%) \)
- \( S \text{ (area)} = 8600 \ (\pm 3.6\%) \)
- \( S/N = 1540 \ (\pm 10\%) \)

**With PCT**
- \( S \text{ (ht)} = 12700 \ (\pm 4.4\%) \)
- \( S \text{ (area)} = 8900 \ (\pm 1.5\%) \)
- \( S/N = 1180 \ (\pm 10\%) \)

Signal is maintained (Detectability) but Noise increases (from the additional EPC)
Purged Ultimate Union Example: Lipid Peroxidation Products in Blood

4-hydroxy-2,3-nonenal (HNE), an indicator of oxidative stress, and its metabolite, 1,4-dihydroxynonene (DHN)

PCI with NH₃

Note late eluting peaks
Loss of Response without Backflush Lipid Peroxidation Products in Blood
Purged Ultimate Union for GC/MS

New, simple CFT technology for backflush

- Vent-less injector and column maintenance (first column)
- Improved backflush
  - 1/4 the time and/or lower pressure
  - Compatible with diffusion pump MSD
- Less loss of sensitivity (less flow added = less dilution)
- Option of different phase/dimension for each column
- Less complex
  - No restrictor to choose as with QuickSwap
  - Simple GC configuration (existing supplies)
  - Setpoints calculated by GC
  - Pressure pulsed injections OK
  - Constant flow mode easy!!
Newest References for Backflush  (Pg 1 of 2 )

• **5989-8664EN** Capillary Flow Technology for GC/MS: a Simple Tee Configuration for Analysis at trace Concentrations with Rapid Backflushing for Matrix Elimination (June 08, Prest)

• **5989-9359EN** Capillary Flow Technology for GC/MS: Efficacy of the Simple Tee Configuration for Robust Analysis Using Rapid Backflushing for Matrix Elimination (Aug 08, Prest, Foucault, and Aubut)
References for Backflush

- **5989-6460EN** Analysis of Suspected Flavor and Fragrance Allergens in Cosmetics Using the 7890A GC and Capillary Column Backflush (March 2007)
- **5989-6066EN** Rapid Forensic Toxicology Screening Using an Agilent 7890A/NPD/5975C/DRS GC/MSD System (Jan 2007)
- **5989-6095EN** Direct Injection of Fish Oil for the GC-ECD Analysis of PCBs: Results Using a Dean Switch with Backflushing (Jan 2007)
- **5989-6018EN** Improving Productivity and Extending Column Life with Backflush (Dec 2006)
- **5989-5111EN** Simplified Backflush Using Agilent 6890 GC Post Run Command (June 2006)
- **5989-1716EN** New Tools for Rapid Pesticide Analysis in High Matrix Samples (October 2004)