Capillary Flow Technology
-- solves difficult application problems easily & opens up many new (and old) possibilities for GC & GC/MS

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IF We Only Had A Technology That Provided Easy, Reliable Flow Structures In The GC Oven...

It would open up many new (and old) capabilities for GC

- **Column connections** (connect pre-column)
- **Change MSD columns** (without venting)
- **Backflush** (Reverse flow through column)
- **Detector splitter** (effluent split to two or more detectors)
- **Merge flows** (2 columns to 1 MSD)
- **Deans switch** (heart cut select peaks to 2\textsuperscript{nd} column)
- **Comprehensive 2-D GC** (cut all peaks to 2\textsuperscript{nd} column)
- etc.
### Types of Connectors Used In The GC Oven

<table>
<thead>
<tr>
<th>Connector Type</th>
<th>Advantages</th>
<th>Limitations</th>
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<tbody>
<tr>
<td>Metal Fittings</td>
<td>Packed columns, reliable</td>
<td>Not inert, no ferrule for capillary columns</td>
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<tr>
<td>Press Fit Glass</td>
<td>Low dead volume, inert, low cost</td>
<td>Difficult to assemble, comes apart</td>
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<tr>
<td>Graphite</td>
<td>High temperature</td>
<td>Sheds active graphite particles into sample path</td>
</tr>
<tr>
<td>Polyimide</td>
<td>Low initial leakage</td>
<td>Loosens and leaks with oven cycling, solvent tailing</td>
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Challenges For Inside the Oven Devices

- **Inertness** (it is in the sample path)
- **Low dead volume** (it is in the separation path)
- **Leak free** (especially with repeated temp cycling)
- **Fast thermal response** (follow rapid oven ramping)
- **High temp tolerance** (GC oven can go over 350C)
- **Reliable and easy to use**
<table>
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<th>Capillary Flow Technology</th>
<th>Description</th>
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<tr>
<td><strong>5 Key Developments in Capillary Flow Technology</strong></td>
<td></td>
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<tr>
<td><strong>Metal Ferrules</strong></td>
<td>Easy to use, do not loosen or leak with oven cycling to 400°C</td>
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<td><strong>Manifold Plates</strong></td>
<td>Complex flow structures with low thermal mass</td>
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<tr>
<td><strong>Deactivation of Metal</strong></td>
<td>Makes metal surfaces as inert as column</td>
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<td><strong>EPC</strong></td>
<td>Backflushing now possible, change MSD columns without venting, known column outlet pressure</td>
</tr>
<tr>
<td><strong>Calculators</strong></td>
<td>Accurately predict flows and pressures BEFORE installing devices</td>
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Capillary Flow Technology - Design

... a proprietary Agilent Technology

- Photolithographic chemical milling for low dead volume
- Diffusion bond two halves to form a single flow plate
- Small, thin profile provides fast thermal response
- Projection welded connections for leak tight fittings
- Deactivation of all internal surfaces for inertness
The Metal Ferrule

Does not loosen (leak) even with *thousands* of runs to 350C
Does not shed particles

Square cut is not critical
3-Way Splitter With Makeup

Effluent Splitter
(3 Way)

Column in

Aux EPC in

Det3 out

Det2 out

Det1 out

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Comparison of New Fitting with Polyimide Fitting

Polyimide Fitting

Exposure to polyimide and unpurged annular spaces is greatly reduced

New Fitting

Ferrule Ejector Hole
Fitting Design Minimizes Tailing

Pentane test chromatogram

Capillary Flow Technology fittings avoid tailing with small but well swept dead volume
Capillary Flow Technology - Capabilities

- Solvent Bypass
- Heart Cutting (Deans Switch)
- Backflush
- Detector Splitting
- QuickSwap
- Modulation (GCXGC)
Capillary Flow Technology Devices

Ultimate Union

Reliable precolumn connector

Tube Connector

Easy valve to capillary column connector

Tube is 0.25 mm id and is deactivated
Splitters: Unpurged Tee
Simultaneous detection with 2 detectors (but NOT MSD)
Cannot do backflushing

1:1 split FPD:μECD

7890A GC
30 m X 0.25 mm id X 0.25 μm HP-5MS

Column

Effluent Splitter WITHOUT Makeup

Auto-sampler

FPD P

μECD

0.3 m X 0.25 mm id

Det 1 OUT

Column IN

Det 2 OUT
QuickSwap

Change MSD columns without venting
Backflush heavy components out split vent

Auto-sampler

AUX EPC
4 psig

Column

7890A GC

MSD Transferline

171 mm X 0.121 mm id restrictor

5975C Inert MSD
QuickSwap MSD Interface

Remove column w/o venting
  – Air & $\text{H}_2\text{O}$ blocked

Safe disconnection of column from inlet for inlet maintenance
  – Reversed flow through column during inlet maintenance

Backflushing
  – Removes heavies from column

Maintain constant flow to MSD

*(flow rates exceeding 2 mL/min require an MSD with Performance Turbo)*
Backflush with QuickSwap

**During GC Run**
- S/S Inlet
- Split Vent Trap
- Column (25 psi)
- QuickSwap
- Aux EPC
- MSD

**After GC Run**
- S/S Inlet
- Split Vent Trap
- Column (1 psi)
- QuickSwap
- Aux EPC
- MSD (4 psi)
- MSD (45 psi)
Benefits of Backflushing

– More samples/day/instrument
– Better quality data
– Lower operating costs
– Less frequent and faster GC & MSD maintenance
– Longer column life
– Less chemical background
Three Other Devices Provide Backflush Capability

- 2-Way Splitter with Makeup
- 3-Way Splitter with Makeup
- Deans Switch
Pesticides: Three Way Splitter with Makeup

1X method with 1:1:0.1 split FPD:MSD:ECD

Auto-sampler

Phosphorus FPD

AUX EPC 3.8 psig

uECD

3-Way Splitter with Makeup

Column

7890 GC

30 m X 0.25 mm id X 0.25 um HP-5MS

5975C MSD
Milk Extract (1 injection)

Full scan TIC

SIM

μECD

FPD(P)
Run stopped at 42 min and backflushed at 280° C for 7 mins.

Blank run after backflushing showing the column was clean.

It took additional 33 mins and column to 320° C to remove these high boilers.
Dean Switch

Heartcutting 2-D GC provides extremely high chromatographic resolution
2-D Separation of Sulfur Compound in Diesel Fuel

Compound is completely resolved and can be analyzed with FID

- Diesel Fuel
- Heart Cut to Column 2
- Hydrocarbon Matrix
- Trace Sulfur Compound (4,6-DMDBT)

Column 1 - FID 1

Column 2 – FID 2
Agilent’s flow modulator design: **Differential Flow**

Flow modulator eliminates the need for cryo. Sample compression controlled by flow ratios occurs in the collection loop and is quickly injected into the second column, resulting in very narrow and tall peaks.

**Differential flow concept is designed by John V. Seeley, Oakland University**
Flow modulation:  
(GC x GC) of diesel fuel: 7890A

GC x GC Chromatogram:
- Showing the normal B.P. distribution (1\textsuperscript{st} dimension)
- Also shows hydrocarbon classes in clusters
- Consistent RT for alkanes in 1\textsuperscript{st} dimension showing precise modulation
- Comparable peak in 2\textsuperscript{nd} dimension band shows minimum peak broadening with flow modulation
Agilent Flow Modulation GC x GC

- **Reliable Setup:** Based on capillary-flow technology, easy to setup, high performance chromatography, and reliable.
- **No Cryogen Required:** Flow modulation means no tanks of Liquid N₂ or CO₂
- **7890A Enabled GC x GC:** Capillary-flow-technology ready, synchronized periodic events ensure precise modulation, control from a modified TCD board
- **Comparable resolution without N₂:** Cap Flow Technology allows low dead volume and precise flow control, resulting in minimum peak broadening even without cryo-focusing. Peak widths on the second column are typically 70 to 100 ms at half maximum.
- **Sensitivity:** Approaches that obtained by thermally modulated systems
Summary

Capillary Flow Technology solves difficult application problems easily. It opens up many new (and old) possibilities for GC and GC/MS systems.