Multidimensional Gas Chromatography

- The use of two or more columns to resolve a sample (or using a single column in more than one direction.
- Current column technology is very near the theoretical limit.
- However, it's still not possible to resolve all components in a complex mixture.

Examples of Complex Samples

Tobacco smoke

Over 1000 peaks identified - each actually can contain two or more components.

PCBs

207 species but only ~180 resolved

Coffee

Over 600 components identified.

Multidimensional GC

Elemental specific and mass detectors are helpful but can't always solve the problem.

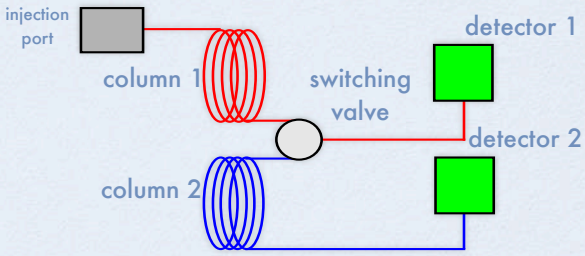
Example

MS can't tell o-, m-, -p substitution or cis from trans.

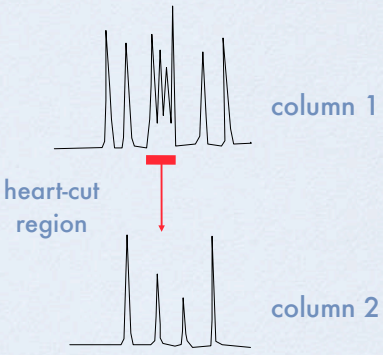
Multidimensional GC can assist in some cases and relies on relatively inexpensive technology.

Multidimensional GC

Method relies on passing a portion of a columns effluent to a second column using flow switching.



Multidimensional GC



Why Bother?

Basic assumption is that no single column can resolve all components of interest.

Possible choices

- Best - if single column/analysis can do the job.
- Second best - use two separate assays with different conditions or columns.
- Last resort - multidimensional GC.

WHY BOTHER?

Sample is limited

- You need all of your data from a single run

Time is limited

- While a multidimensional run can be longer than a single assay, its still shorter than two separate runs

Equipment is limited

- One GC setup can do the entire assay

APPROACHES

Enrichment

- Used to increase amounts of trace components

Heart-cutting

- Grabs an unresolved portion of a sample for improved separation

Backflushing

- Reverse column flow to drive off highly retained components

ENRICHMENT

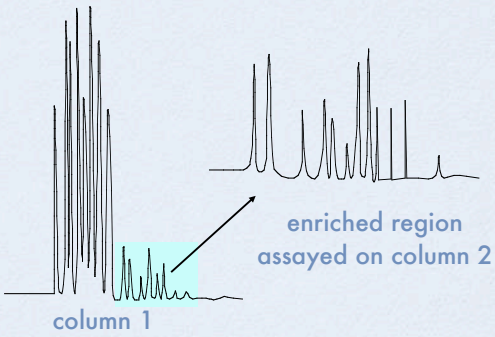
Pre-concentration of trace components initially on a packed column.

More sample can be placed on a packed column than a capillary.

Only the trace components of interest are passed to the capillary column.

Results in more sample being introduced.

ENRICHMENT EXAMPLE



HEARTCUTTING

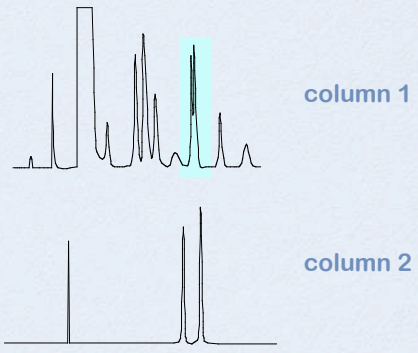
Also called "cut and transfer."

No single column can resolve all components of interest or a very large peak masks other components.

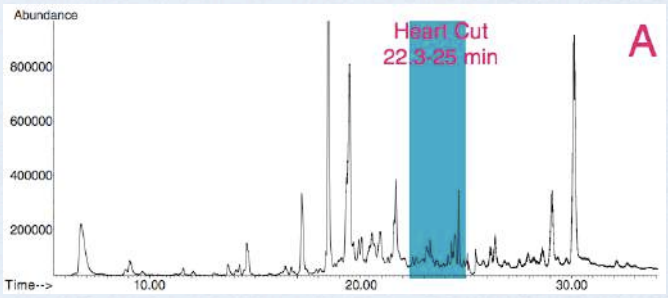
Passing the unresolved area to a second column can be used to fix the problem.

The second column can also be a different polarity.

HEART-CUTTING EXAMPLE



LATEX BALLOON VOLATILES



LATEX BALLOON VOLATILES

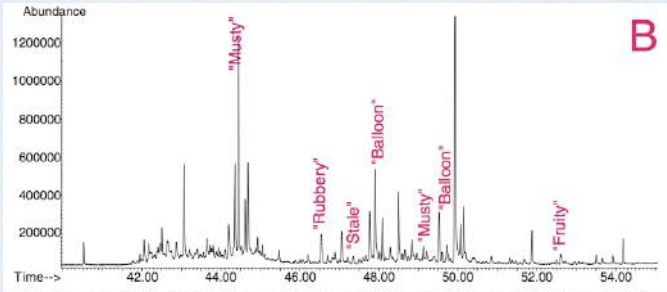


Figure 7. (A) FID trace from GC-GC precolumn separation of latex balloon volatiles (B) TIC from GC-GC main column separation of 22.3-25 min heart cut from precolumn.

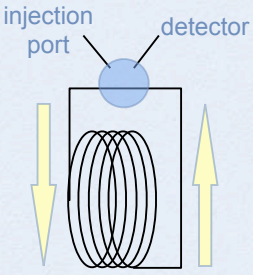
BACKFLUSHING

Used when you have a sample that contains both volatile and relatively non-volatile species.

Total analysis in one direction would take forever.

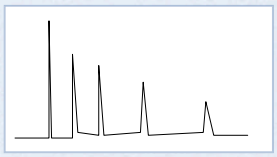
Only a single column is needed.

BACKFLUSHING - NORMAL MODE

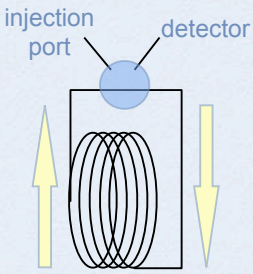


In normal operation, flow occurs as one would expect.

After all faster eluting species have evolved, the valve is switch, reversing the flow.



BACKFLUSHING - REVERSE MODE



Now the higher MW species will evolve.

In effect, we are only using the first portions of the column to do our separation.

BACKFLUSHING EXAMPLE

- Method uses single GC ad column
- Forward analysis using a TCD detector to assay nonflammable gases.
- Heartcut part of the sample to a 2nd TCD.
- Backflushing to an FID to assay flammable gases.

BACKFLUSHING EXAMPLE

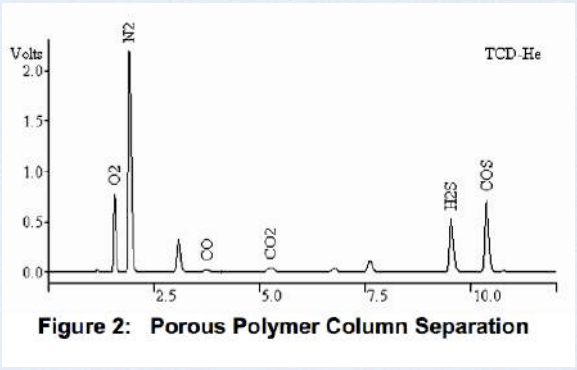


Figure 2: Porous Polymer Column Separation

BACKFLUSHING EXAMPLE

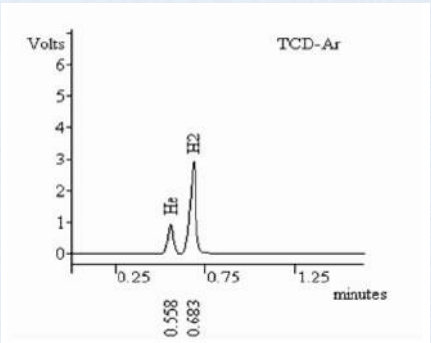


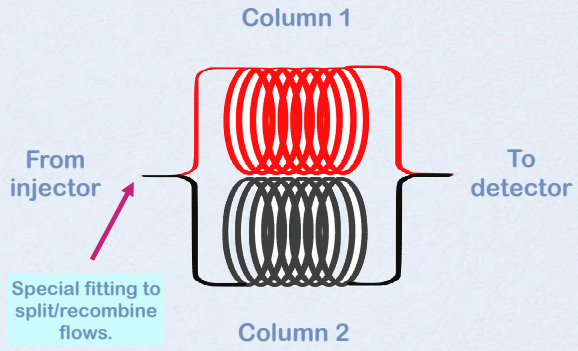
Figure 3: Separation for Helium/Hydrogen

DUAL COLUMN EXAMPLE

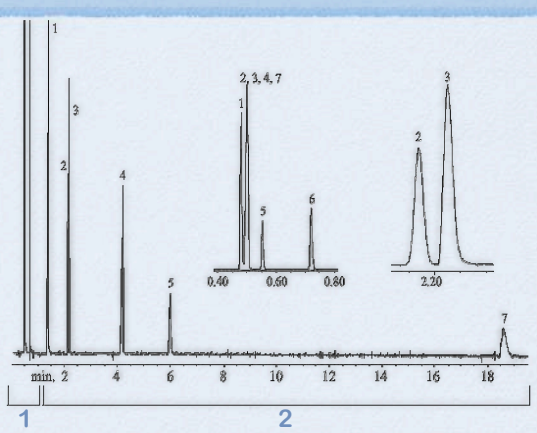
Restek describes the use to two capillary columns used in parallel to separate gases and volatile components.

- 5A molecular sieve column for gases
- (Rt-Msieve 5A PLOT column, 30m 0.32 mm ID)
- A bonded porous layer polymer for volatiles.
- (Rt-QPLOT, 30m, 0.53 mm ID)

DUAL COLUMN EXAMPLE



DUAL COLUMN EXAMPLE

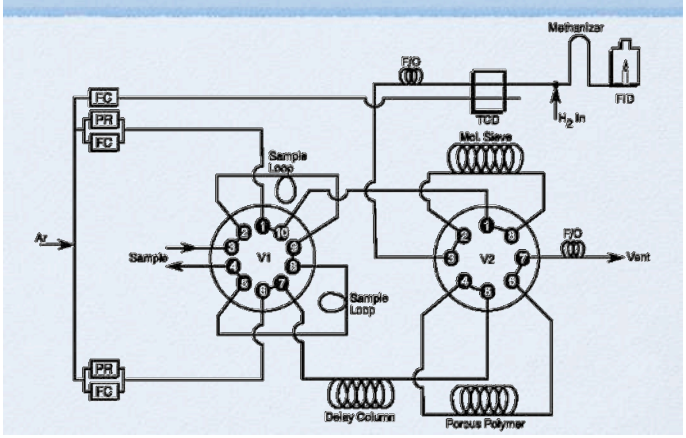


1 - He
2 - Ar
3 - O_2
4 - N_2
5 - CH_4
6 - CO_2
7 - CO

BACKFLUSHING EXAMPLE

- ✓ Determination of dissolved gases in transformer fluid - ASTM methods D3613, D2945, D3612
- ✓ Carbon oxides are catalytically converted to methane for detection as methane using a FID.
- ✓ Elemental gases are detected using a TCD.
- ✓ Backflushing is used to remove transformer fluid from one column while results are being obtained from the other.

THE PLUMBING



EXAMPLE