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Capillary GC Troubleshooting: a Practical Approach

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Outline

- ✔ Basic Troubleshooting Strategy
- ✔ Preventing Problems
- ✔ Identifying Common Problems
- ✔ Recommended Reading
- ✔ Discussion

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Troubleshooting Strategy

- ✔ Have appropriate equipment and supplies on hand.
- ✔ Establish a systematic approach.
- ✔ Know what to look for first.
- ✔ Record what you did to correct the problem.

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Suggested Equipment to Have on Hand for Troubleshooting:

- ✔ Electronic Leak Detector
- ✔ Flow Meter
- ✔ “Test” Column
- ✔ Replacement Accessories (Syringes, Ferrules, Septa)
- ✔ Replacement Purifiers

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Five Major Sources of Chromatographic Problems:

1. Operator Error
2. The Sample
3. The Column
4. The GC Electrical System
5. The Gas Flow System (both internal and external to the GC)

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Approaching the Problem

- ✔ Check first to see if a “fix” for the problem is already known.
- ✔ Check the Supelco Capillary GC Troubleshooting Guide (Bulletin 853.)
- ✔ Check back in the instrument maintenance record.
- ✔ Talk to others in your lab.

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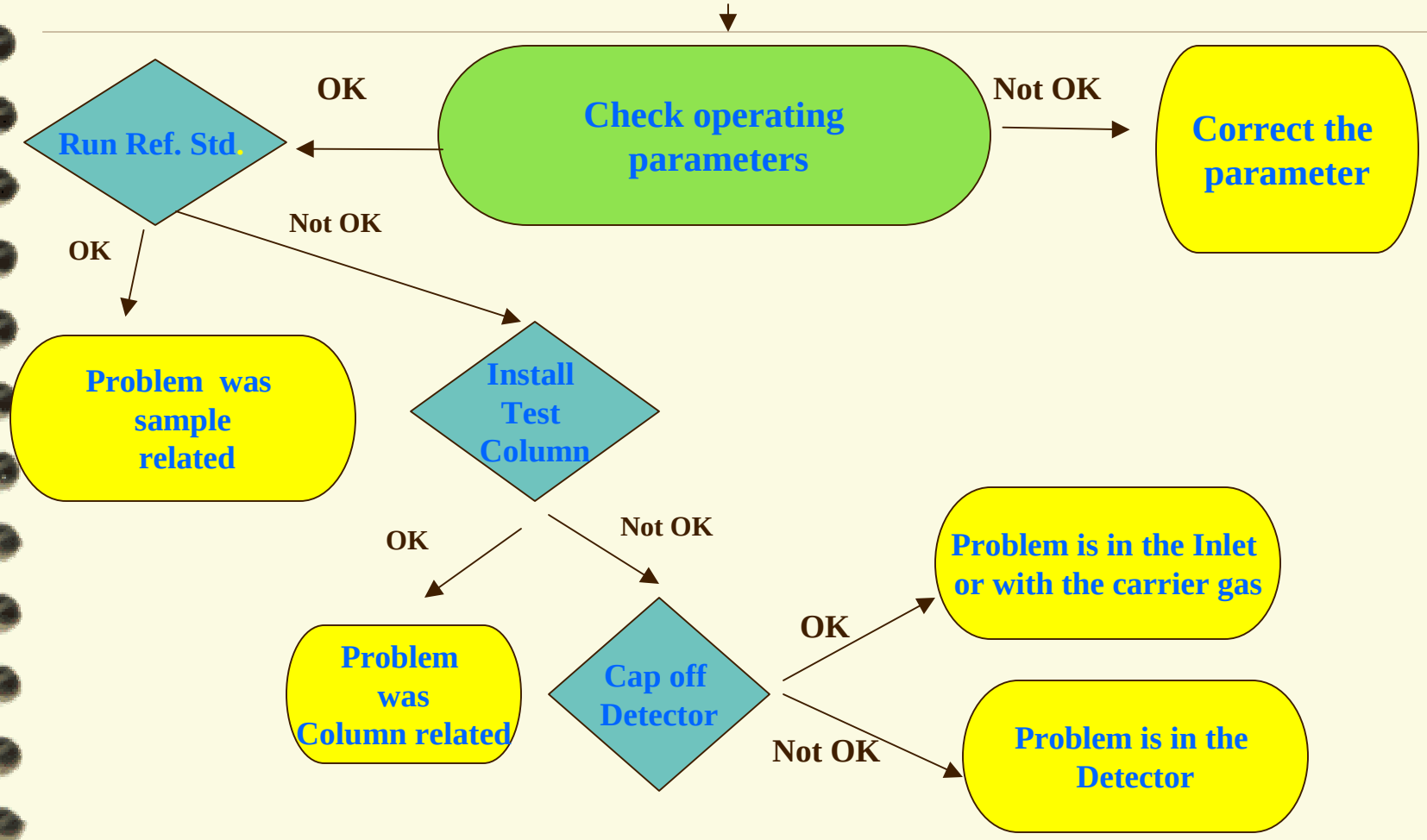
Isolate the Source of the Problem



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When reviewing operating method parameters consider the following:

- Is my starting temp. low enough to allow sufficient sample focusing?
- For splitless injections, is my splitter opening at the appropriate time?
- Is my column flow set to give me maximum efficiency at the most critical point?
- Are heated zones (injectors, detectors, interfaces) set appropriately?

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The Best Way to Solve Problems is to Prevent Them!

- ✔ Install and maintain proper purification for *all* gases in the GC system.
- ✔ Maintain the injector by periodically inspecting and changing the liner, septa, and seal (H/P™.)
- ✔ Use the proper injection technique-this includes using the right liner for the job.
- ✔ When necessary, use a guard column to protect the analytical column.

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Gas Purification

- Carrier Gas
  - At minimum, remove hydrocarbons, water, and oxygen.
- Hydrogen (FID)
  - At minimum, remove hydrocarbons.
- Air (FID)
  - At minimum, remove water and hydrocarbons.
- Nitrogen make-up (FID, ECD)
  - At minimum, remove hydrocarbons.
- P-5 make-up (ECD)
  - At minimum remove hydrocarbons (especially halogen-containing), oxygen.

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Acceptable Purity Levels for Chromatography Grade Gases

Impurity / Maximum Concentration

Gas	O2	H2O	CO2	CO	Total Hydrocarbons
Helium	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm
Nitrogen	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm
Air	20-22%	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm
Hydrogen	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm
Argon/ Methane	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm	<1.0 ppm

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Suggested purifiers

	Hydrocarbons	Water	Oxygen
Carrier	Supelcarb HC Supelpure HC	Mole Sieve 5A	OMI -2
H_2			
Air		Mole Sieve 5A	
N_2 makeup			
P-5	OMI -2		OMI -2

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What are some signs that my purifiers need to be changed?

Hydrocarbon Traps

Noise in the baseline (FID)

Increase in background peaks on tune (MSD)

Higher than normal baseline reading on FID

Extra peaks visible in run

Molecular Sieve 5A

Increase in column bleed

Water visible in MS background

Poor peak shapes for gaseous VOCs (purge and trap)

Extra peaks visible in run

OMI™-2 color change

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Injector Maintenance

- ✔ Change (as needed):
  1. Liner and O-ring*
  2. Seal and washer *

*H/P™ GCs

- ✔ Inspect the inlet periodically
  - -Look for contamination in the liner
  - -Look for residue on the seal

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Using the right liner and injection technique can also prevent problems:

- ✔ Split Injection
  - used for concentrated samples
  - high flow of carrier gas through liner during injection
  - should use liner designed for split injection
- ✔ Splitless Injection
  - used for trace analysis
  - low flow of carrier gas through liner during injection
  - inertness and internal volume of liner used are critical

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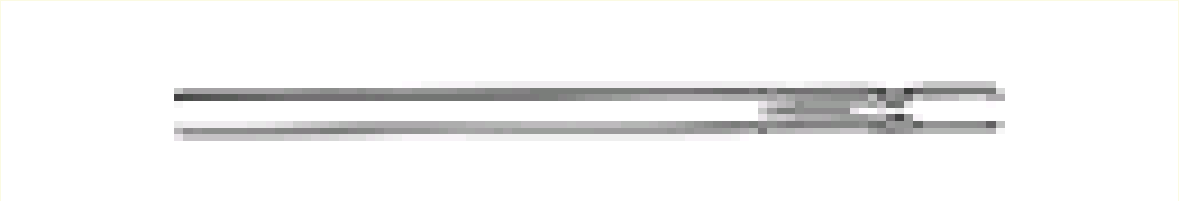
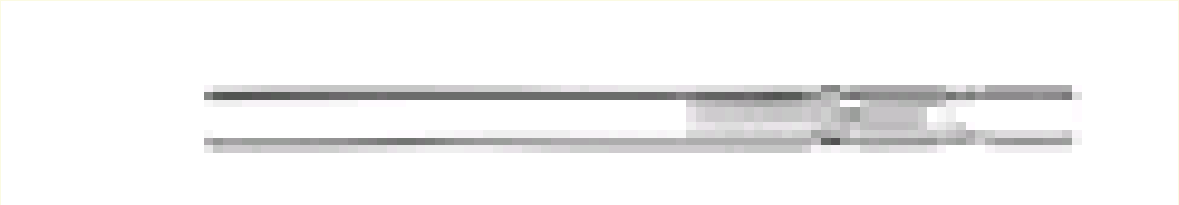
Some liners used for split injection

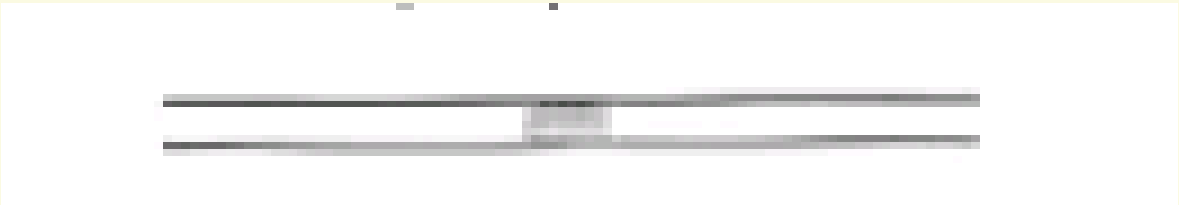
Cup (unpacked)



Cup (wool packed)



Split/splitless- wool packed



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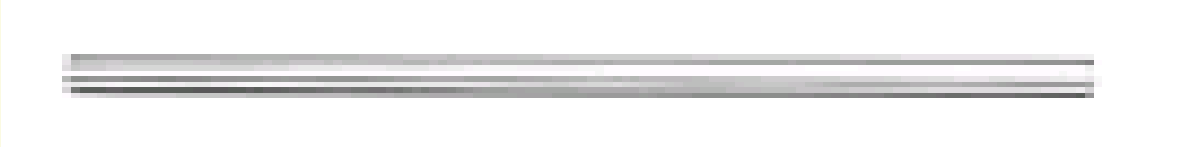
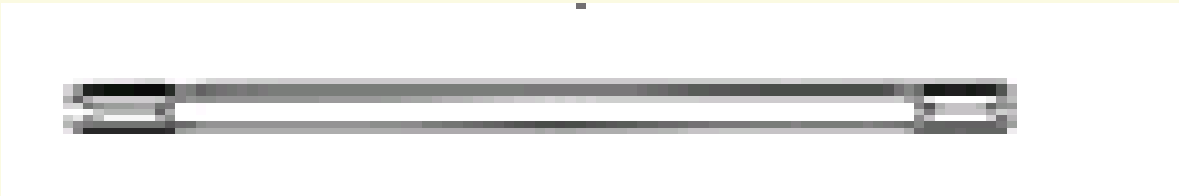
Some liners used for splitless injection

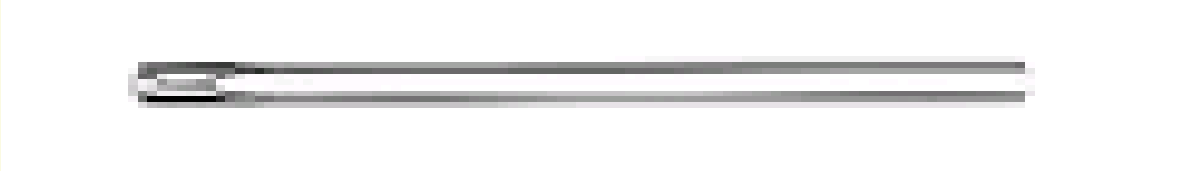
2 mm ID, straight



Dual-tapered



Single-tapered



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If you *must* clean a liner….

- ✔ Handle liners with gloves or forceps.
- ✔ Use *clean* compressed gas and/or a fine brush to remove particles.
- ✔ Rinse liner in an appropriate solvent and dry with *clean* compressed gas.
- ✔ Use mineral acid and/or detergent only if absolutely necessary.

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Using a Guard Column

- ✔ Choose a guard column which has been deactivated.
- ✔ Usually, the ID of the guard matches the analytical column.
- ✔ A 5-10 meter length is normally used.
- ✔ Connect with either a GlasSeal™ or butt connector.

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Common Problems

1. Poor Peak Shapes (either tailing, fronting, or just generally ugly.)
2. Nonlinearity
3. Baseline Noise and /or Drift
4. Ghost Peaks
5. Missing Peaks / Poor Response
6. Insufficient Resolution

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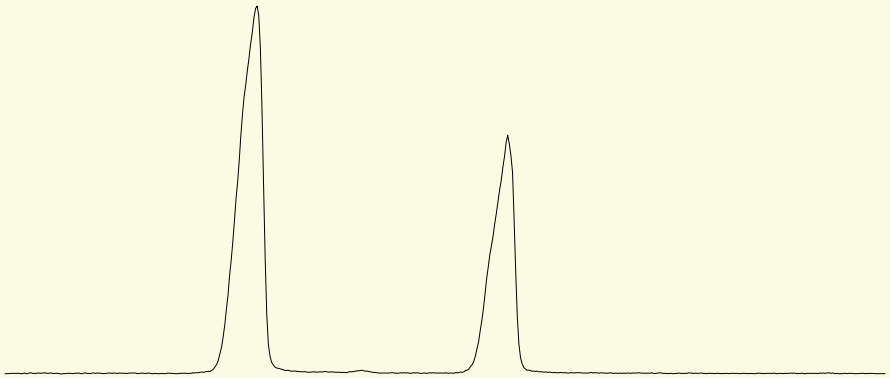
1. Poor Peak Shapes

- ✔ Fronting can indicate column overload.



- ✔ Tailing can indicate activity in the system or improper column installation.

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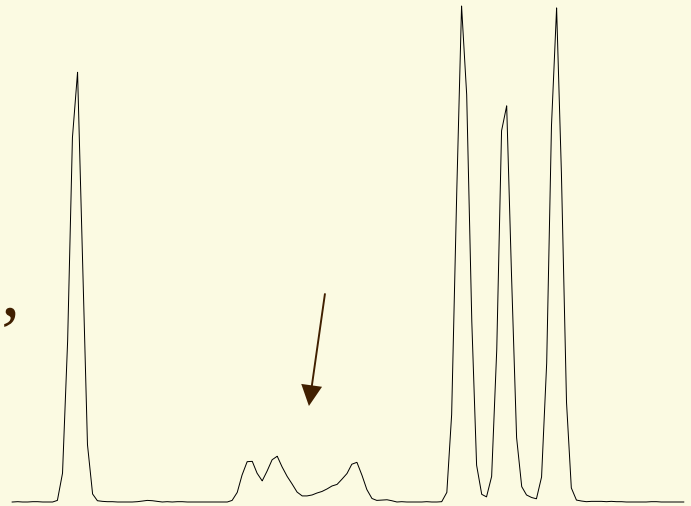
1. Poor Peak Shapes (cont.)

- Generally ugly peaks, such as a,a-dimethylphenethylamine, can be caused by a variety of problems.



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2. Nonlinearity

The most common causes are:

- Column overload
- Detector overload
- Standards preparation
- Poor peak shape resulting in improper integration

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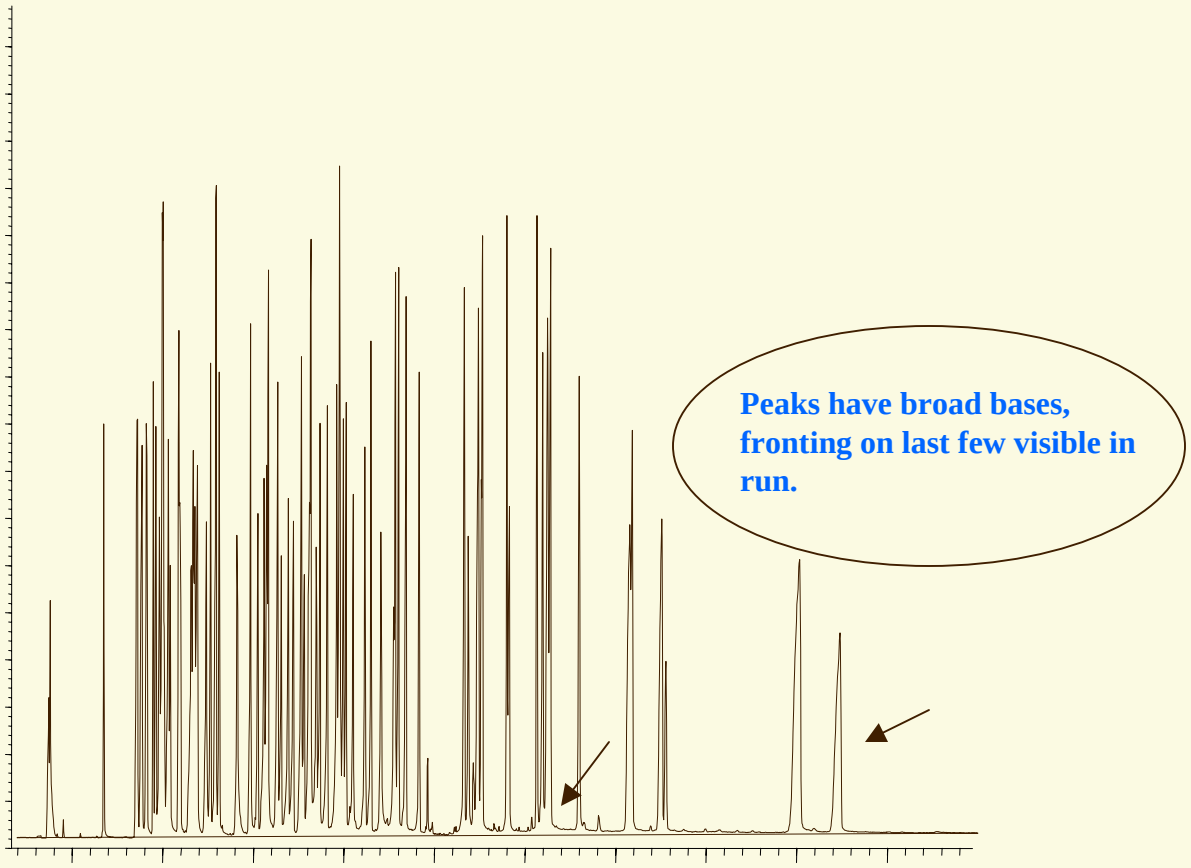
An Example of Overload:



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Preventing column overload for splitless injections:

- ✔ Inject a smaller amount / use a 1 ul syringe.
- ✔ Use a thicker film column.
- ✔ Use a column with a wider ID.
- ✔ Decrease upper limit of calibration range.
- ✔ Use a column of slightly different polarity.

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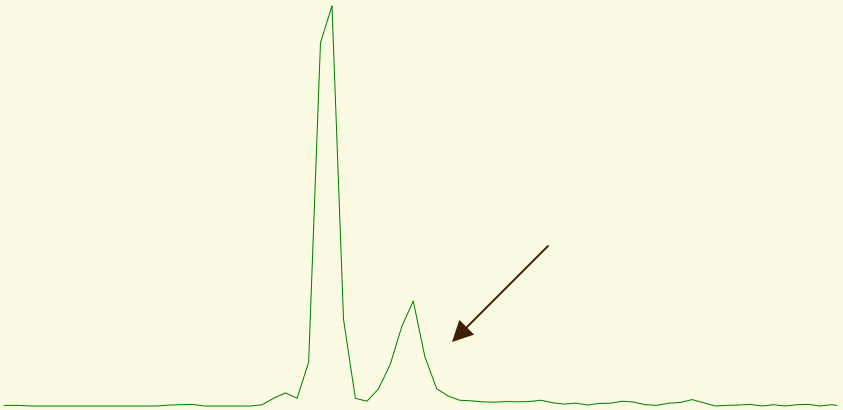
An example of poor peak shape affecting linearity:

- Benzoic acid is typically of poor shape when doing splitless injections.



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3. Common causes of baseline noise / drift.

- ✔ Column bleed
- ✔ Dirty detector
- ✔ Contaminants in carrier gas / carrier gas purity

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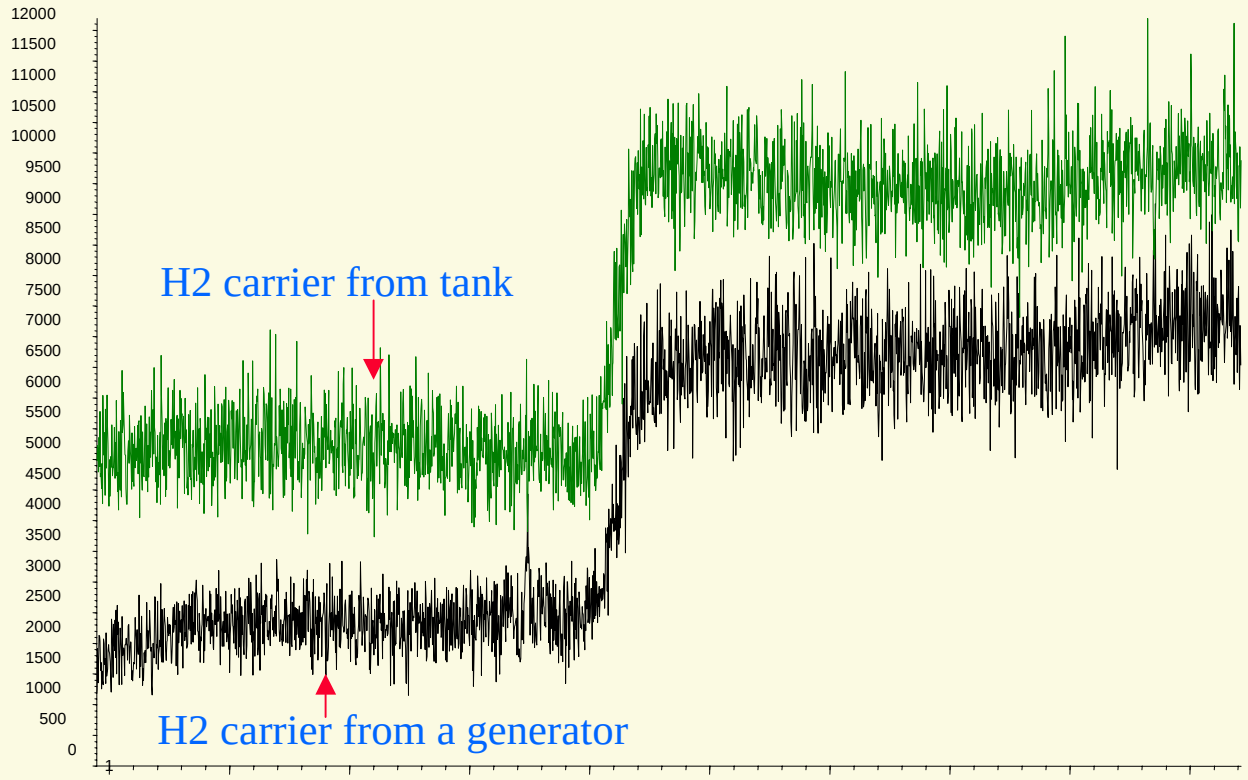
Effect of carrier gas purity on baseline noise:



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Column bleed results from the normal degradation of the stationary phase.

- ✔ All columns bleed to some extent.
- ✔ Bleed increases with temperature.
- ✔ The amount of bleed will increase in the presence of oxygen.

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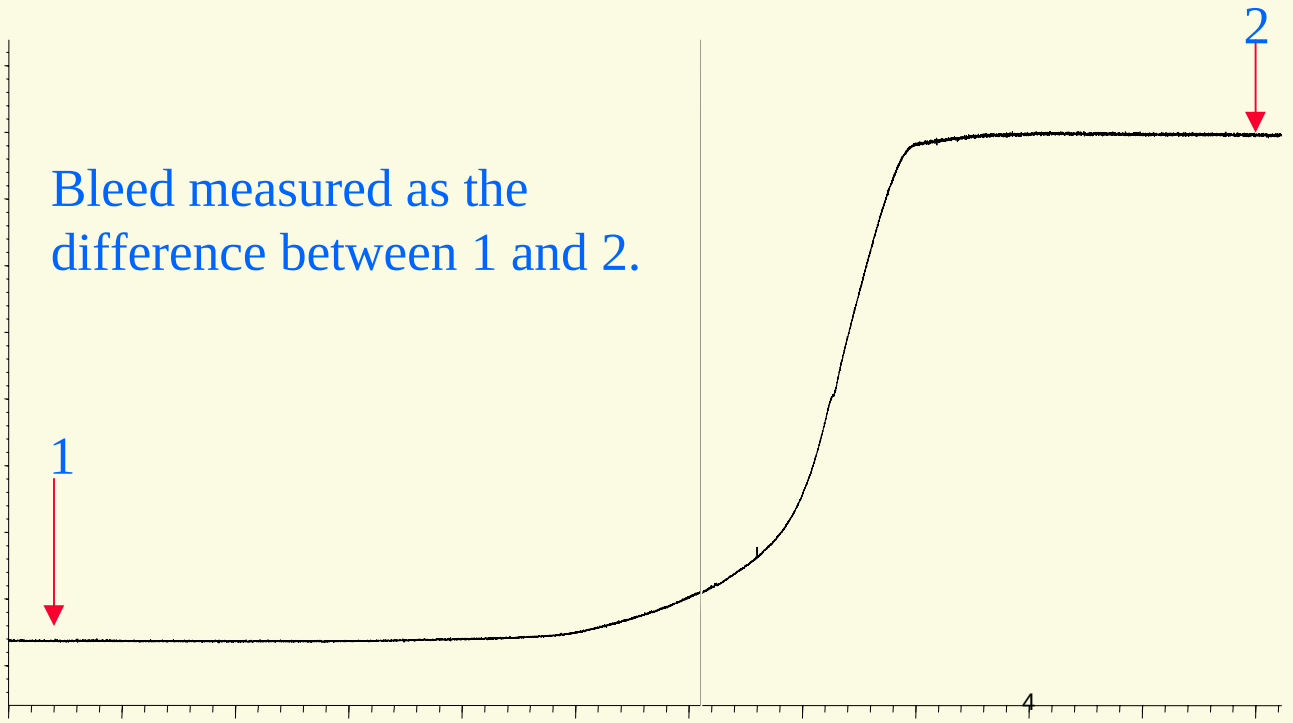
A Typical Bleed Profile:



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Column Bleed on an MSD

- ✔ Visible as baseline rise in the TIC.
- ✔ Check spectra for key bleed ions:
  - PTE™-5: 207, 281
  - SPB™-1: 73, 207, 281
  - SPB™-624: 207, 269
- ✔ Make sure interface temp. is < column max. temp.

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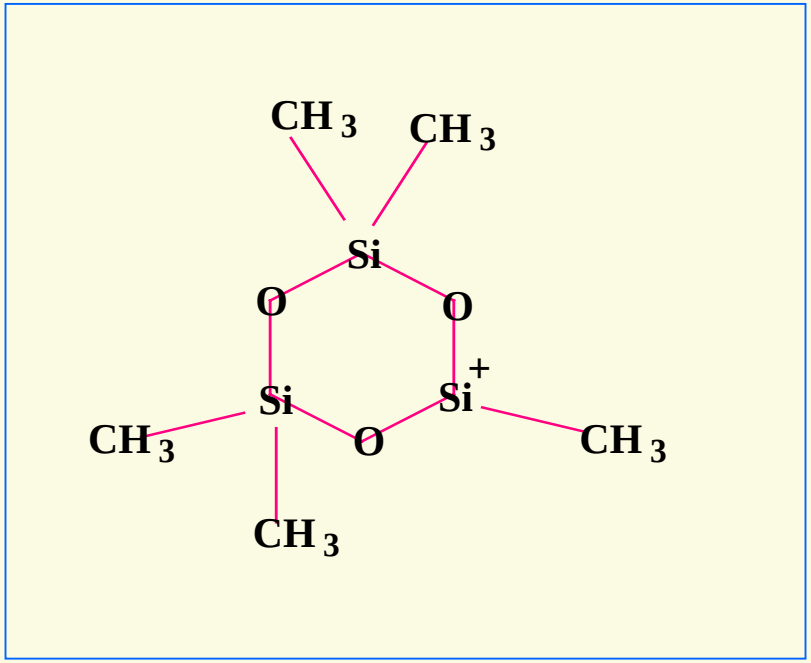
Ion 207 corresponds to a fragment known as D3:



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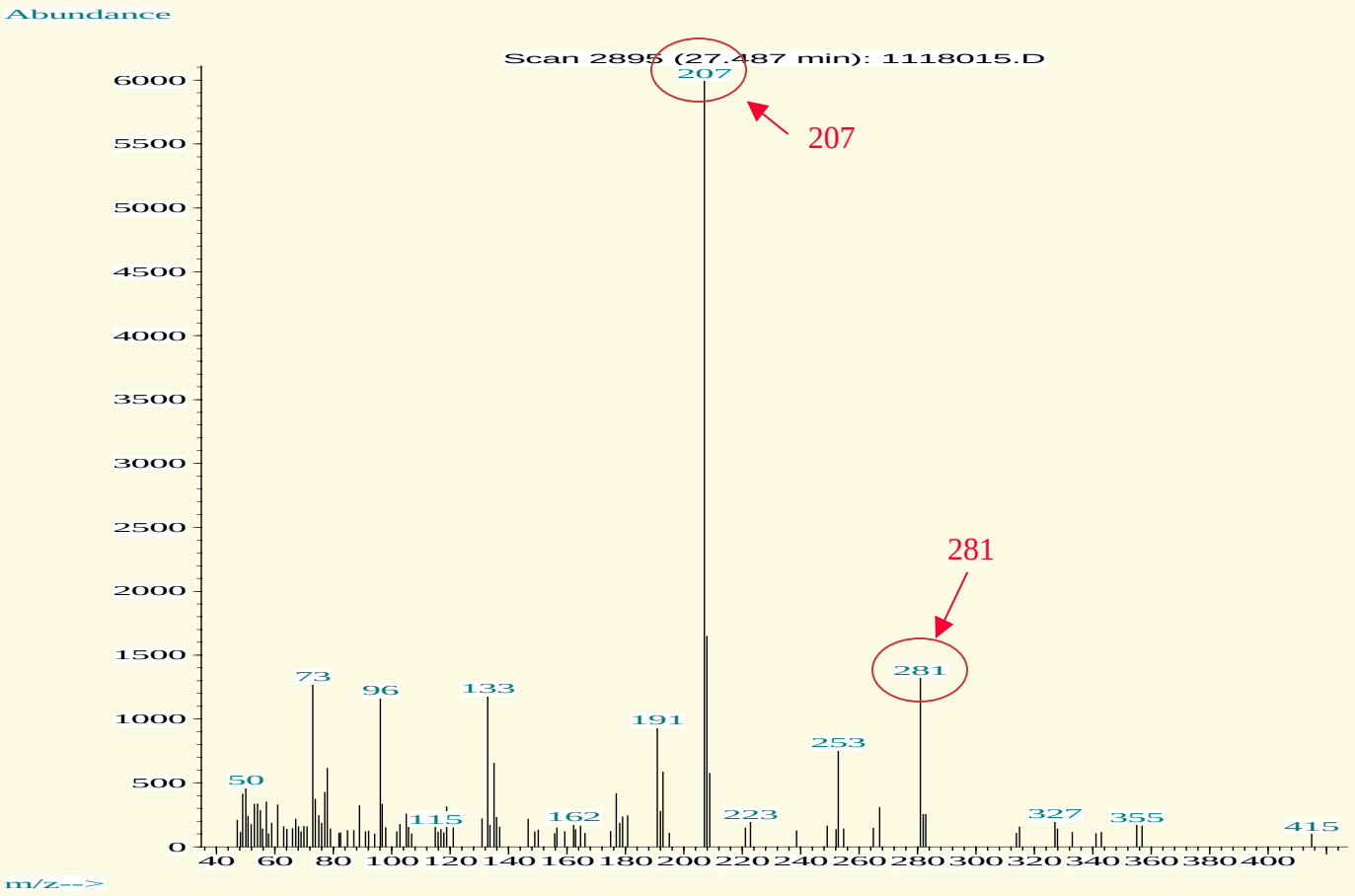
MS spectra of bleed from a PTE™-5 Column



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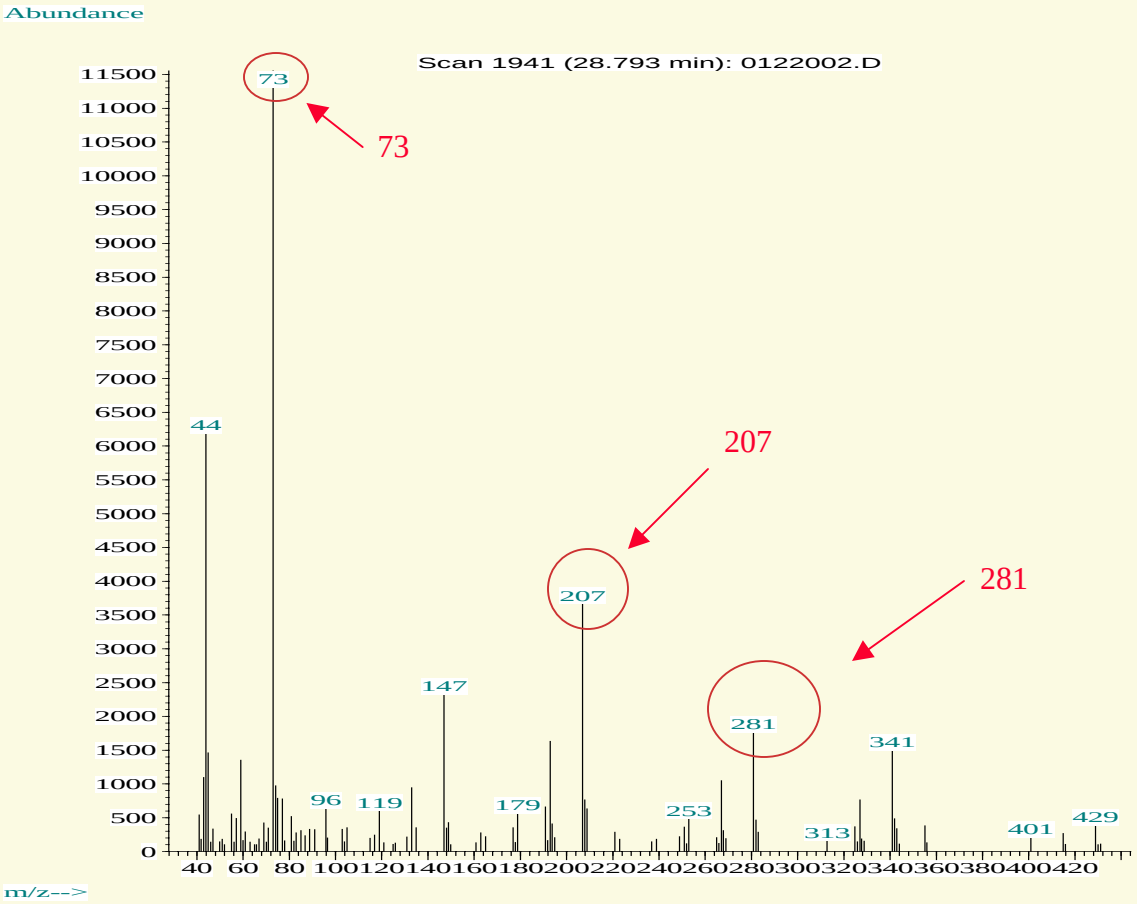
MS spectra of bleed from an SPB™-1 Column



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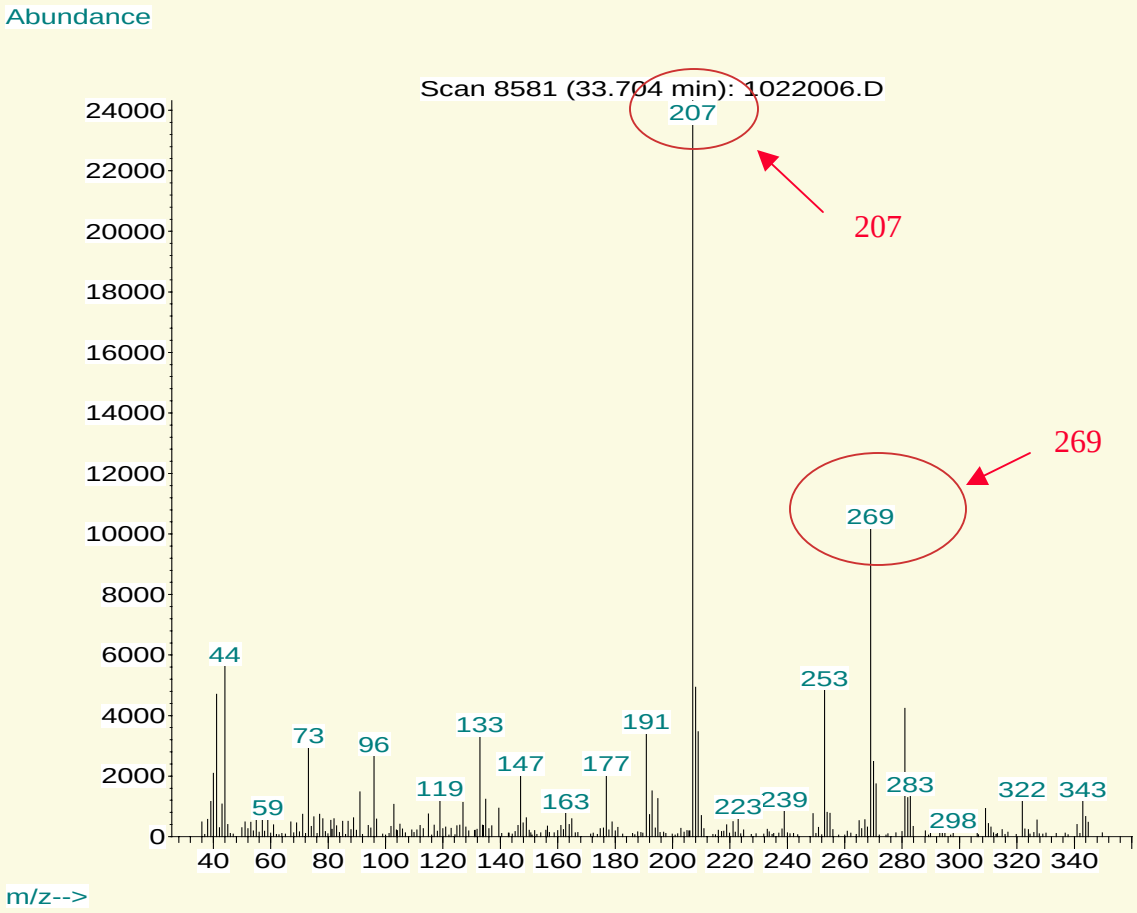
MS spectra of bleed from an SPB™-624 Column



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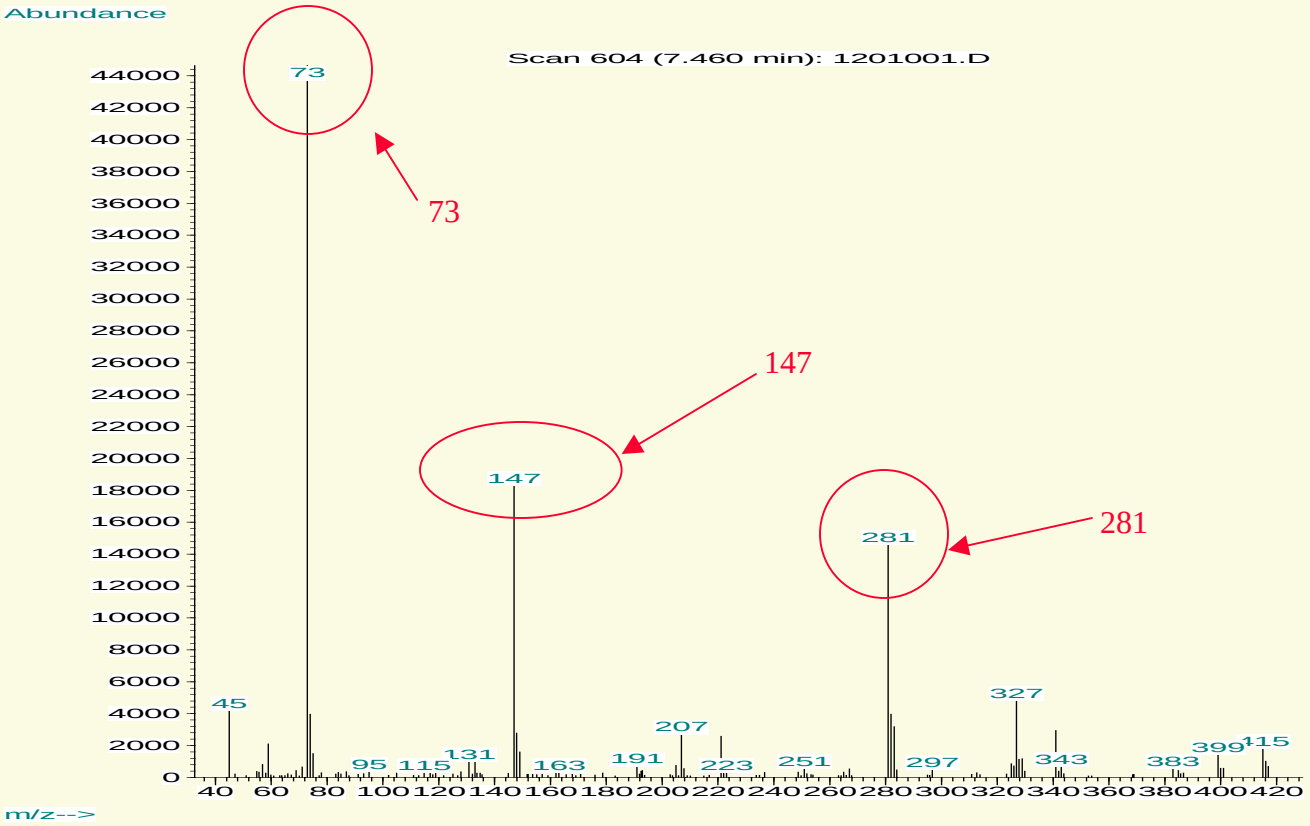
MS Spectra of Septa Bleed



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Prevent column bleed!

- ✔ Sufficiently purge column with carrier before ramping it up in temperature.
- ✔ Make sure carrier gas is filtered for water and oxygen.
- ✔ Check integrity of all fittings leading to the column.
- ✔ Do not heat the column above its maximum temp.
- ✔ Precondition the column prior to use.

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4. Ghost Peaks

- ✔ Residue in the inlet liner and at the head of the column
- ✔ Contaminated syringe / and or wash solutions on an autosampler.
- ✔ Sample carryover
- ✔ Contaminated carrier gas

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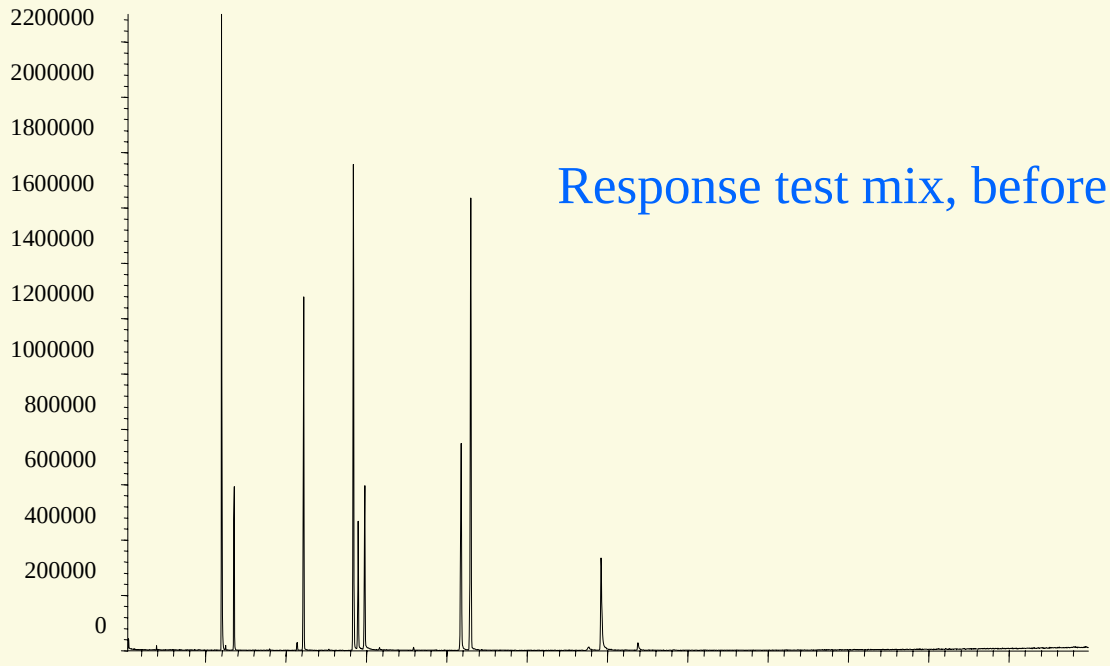
If pieces of septa get into an inlet liner...



Response test mix, before

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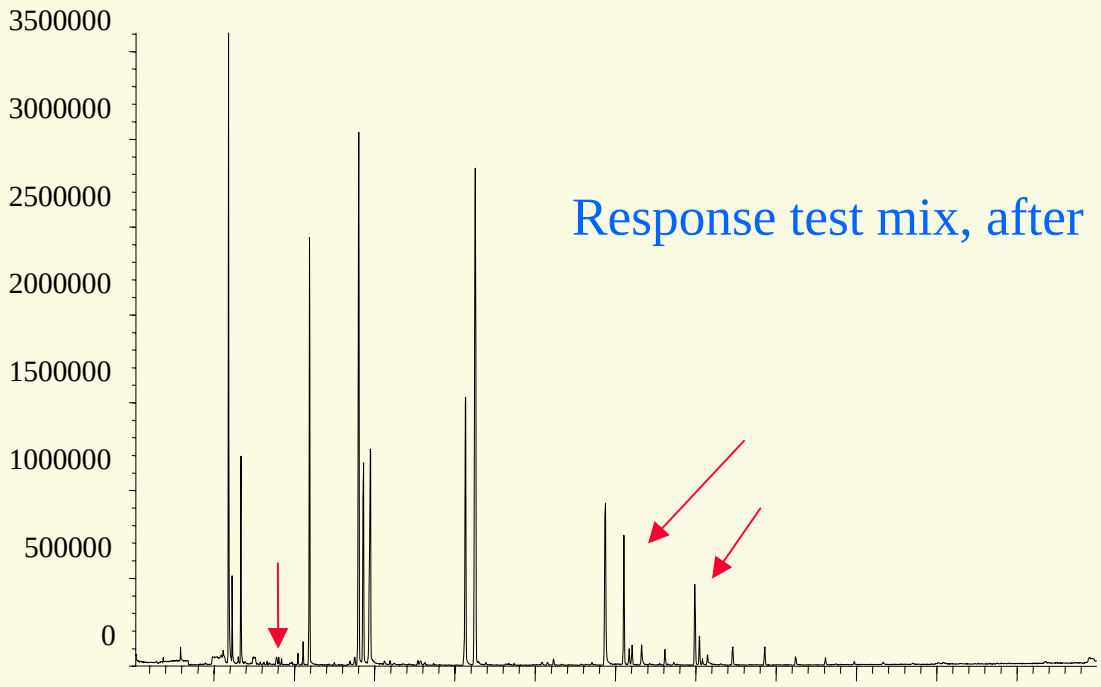
…even a simple analysis can be ruined.



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5. Missing Peaks / Poor Response

- ✔ Sample decomposition
  - Activity in the inlet or column
  - Injection port temperature too high
  - Sample not stable enough for GC
  - Standards not stable
- ✔ Coelution
- ✔ Insufficient run time / final temperature
- ✔ Sample not volatile enough for GC
- ✔ Improper column installation

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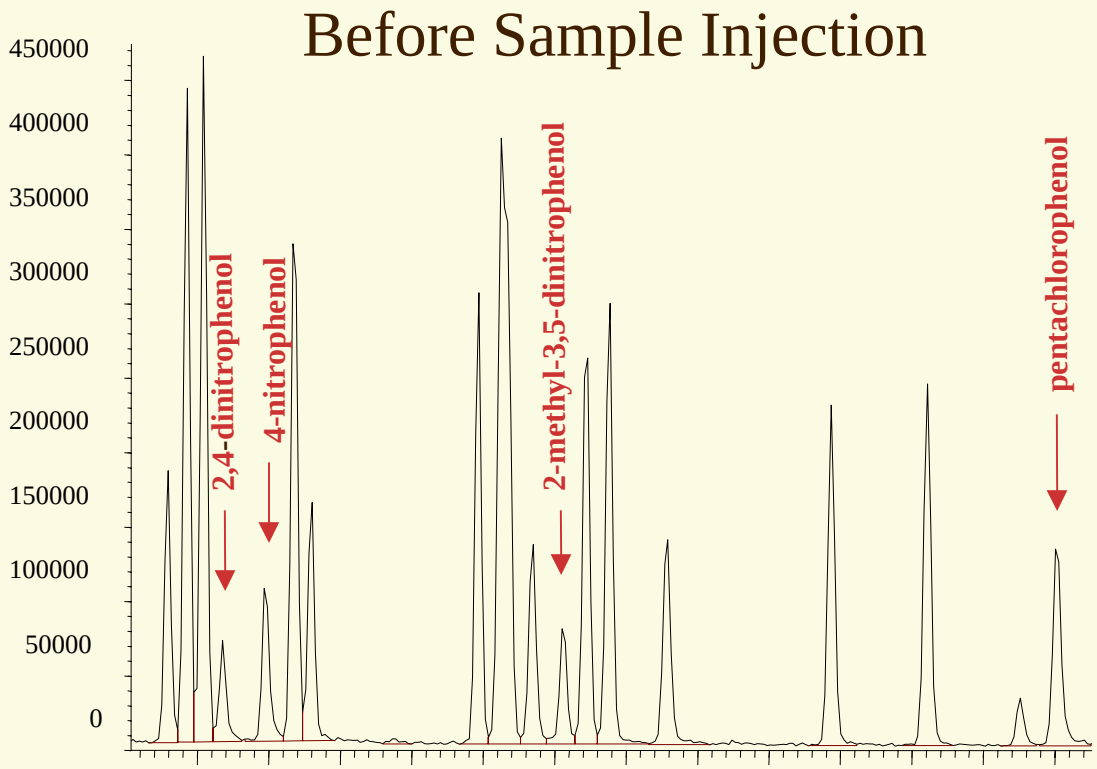
Nasty samples can damage a column by creating active sites:



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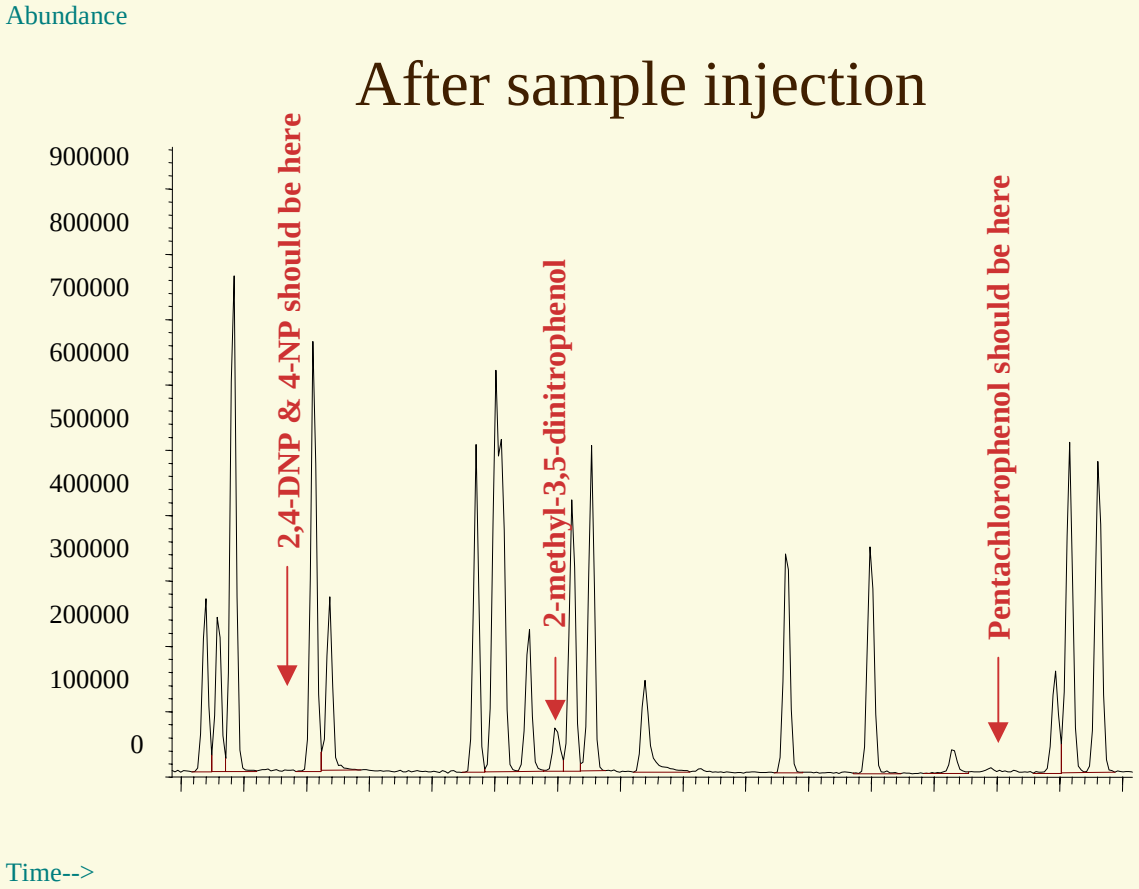
Here, the responses of some acids were affected:



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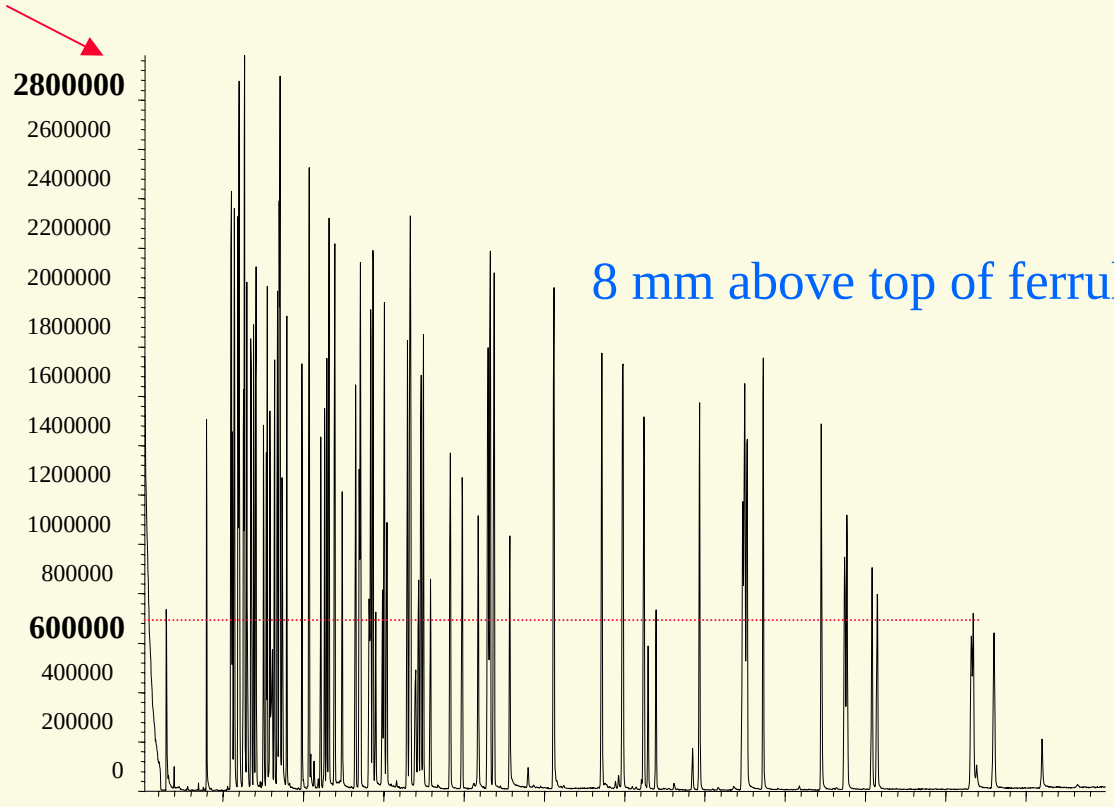
Response can also be affected by the position of the column in the inlet:



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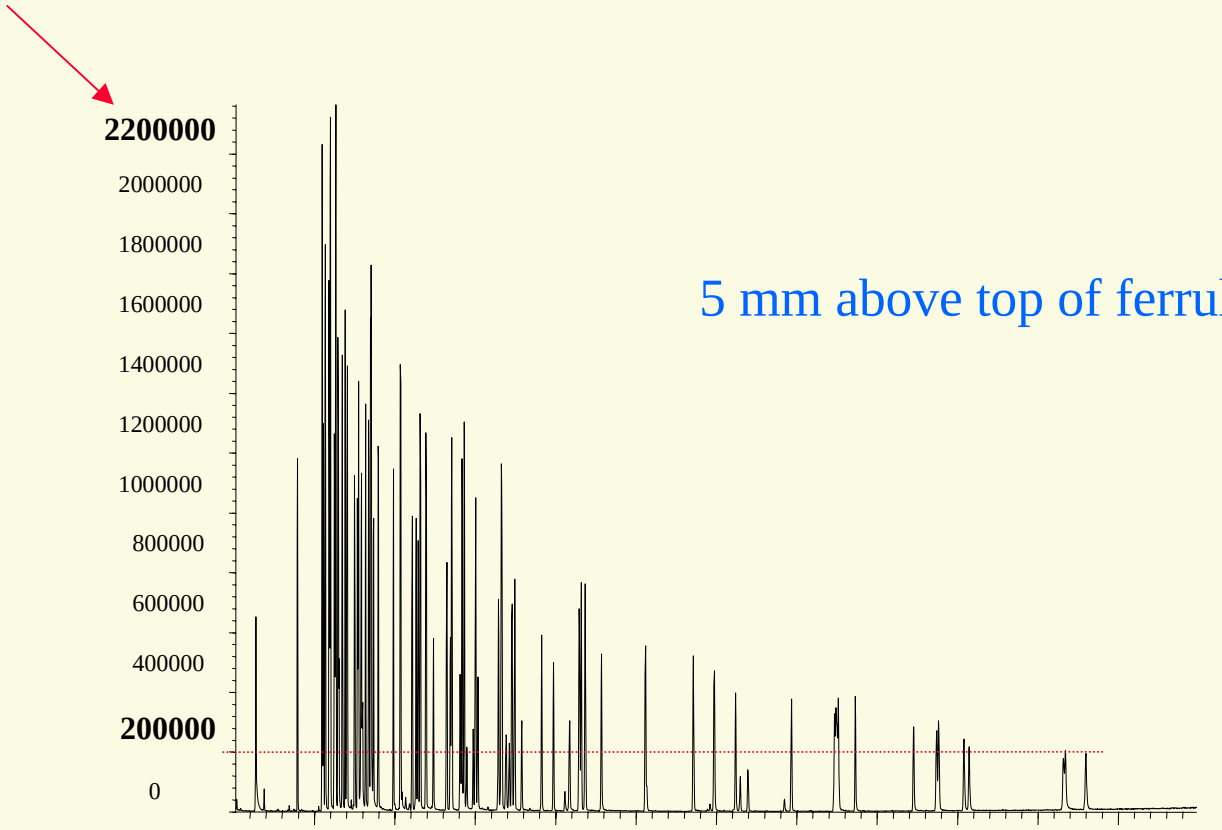
Here, the column was not inserted far enough:



5 mm above top of ferrule

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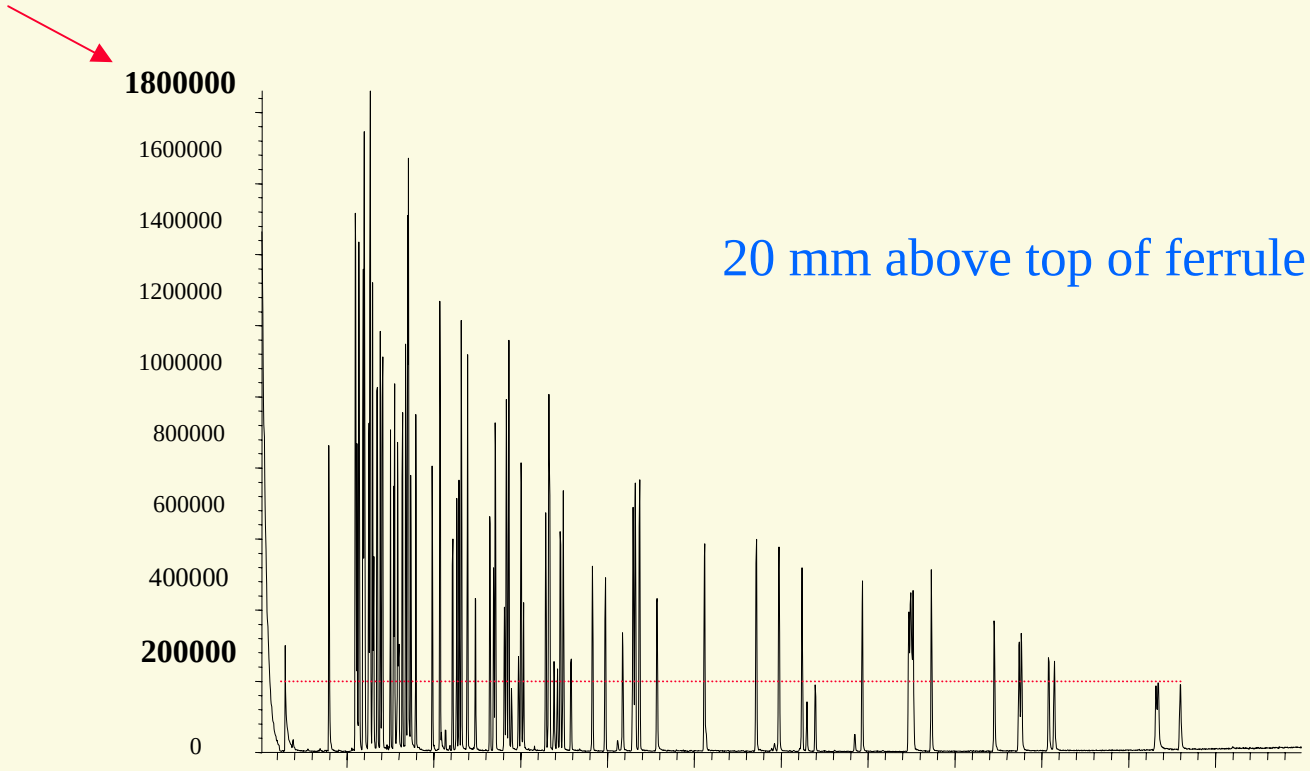
Here, the column was inserted too far:



20 mm above top of ferrule

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6. Insufficient Resolution

- ✔ Wrong column
  - Longer columns increase resolution.
  - Smaller ID columns increase resolution.
  - A different phase altogether may be needed.
- ✔ Wrong Conditions
  - Carrier gas flow too fast or slow .
  - Oven ramp rate too fast.

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Recommended Reading
Supelco Bulletins

1. 741: The Supelco Guide to Leak-Free Connections
2. 783: Cleaning Flame Ionization Detectors
3. 853: Capillary Troubleshooting Guide
4. 875: Supelco Capillary GC Selection Guide
5. 895: Installation and Maintenance Instructions for .25 mm and .32 mm ID Fused Silica Capillary Columns
6. 897: Installation and Maintenance Instructions for .53 mm ID Fused Silica Capillary Columns
7. 898: Gas Management Systems for GC
8. 899: Capillary GC Inlet Sleeve Selection Guide
9. 916: Purge and Trap System Guide
10. 918: Selecting Purifiers for Gas Chromatography

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Help is just a phone call or mouse click away!

✔**Supelco Technical Service**

phone: 1-800-359-3041

email: techservice@supelco.sial.com

✔**Supelco Customer Service**

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