

# Practical Steps in GC Troubleshooting

Techniques, Tips, and Tricks

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# What went wrong and how to fix it...

Common problems

Troubleshooting tools

Troubleshooting examples

“Everything was just fine and then this happened!”

How do I go about  
TROUBLESHOOTING?”

# “Everything was just fine and then this happened!”

Logic = Something changed (slowly or sudden) =  
Something is different

Track Events – log book

- Changed column, liner, septum, syringe, etc.
- Injected samples, other method, etc.
- Did maintenance, cut column, inlet flush, etc.

# Logical Troubleshooting

- **Troubleshooting Starts with Isolating the problem**
  - There are 5 basic areas from where the problem arises
    - INJECTOR
    - FLOW
    - COLUMN
    - DETECTOR
    - ELECTRONICS
  - But of course it can always be some COMBINATION
- *Knowing what can & can't cause the symptom is the key*

# Typical Problems of Optimized Methods becoming Unoptimized...and the Reason Why.

- Peak Tailing – Flow Path or Activity
- Bonus Peaks – In Sample or Back Flash (Carry Over)
- Split Peaks – Injector Problems, Mixed Solvent
- No Peaks – Wasn't Introduced, Wasn't Detected
- Response Changes – Activity, Injector Discrimination, Detector Problem
- Peak Fronting – Overload or Solubility Mismatch, Injector Problems
- Shifting Retention – Leaks, Column Aging, Contamination or Damage
- Loss of Resolution – Separation Decreasing, Peak Broadening
- Baseline Disturbances – Column Bleed, Contamination, Electronics
- Noisy or Spiking Baseline – Electronics or Contaminated Detector
- Quantitation Problems – Activity, Injector or Detector Problems

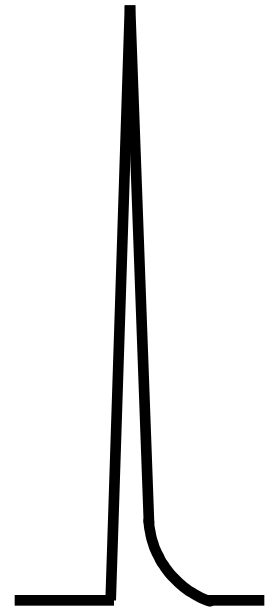
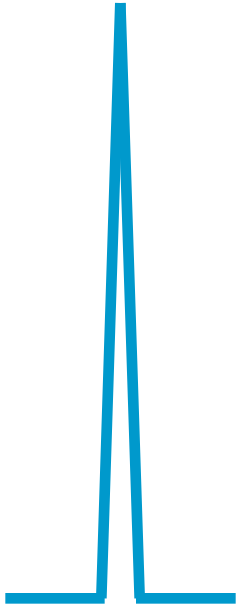
# Peak Tailing

INJECTOR or COLUMN is Active

-Reversible adsorption of active compounds  
(-OH, -NH, -SH)

FLOW problem

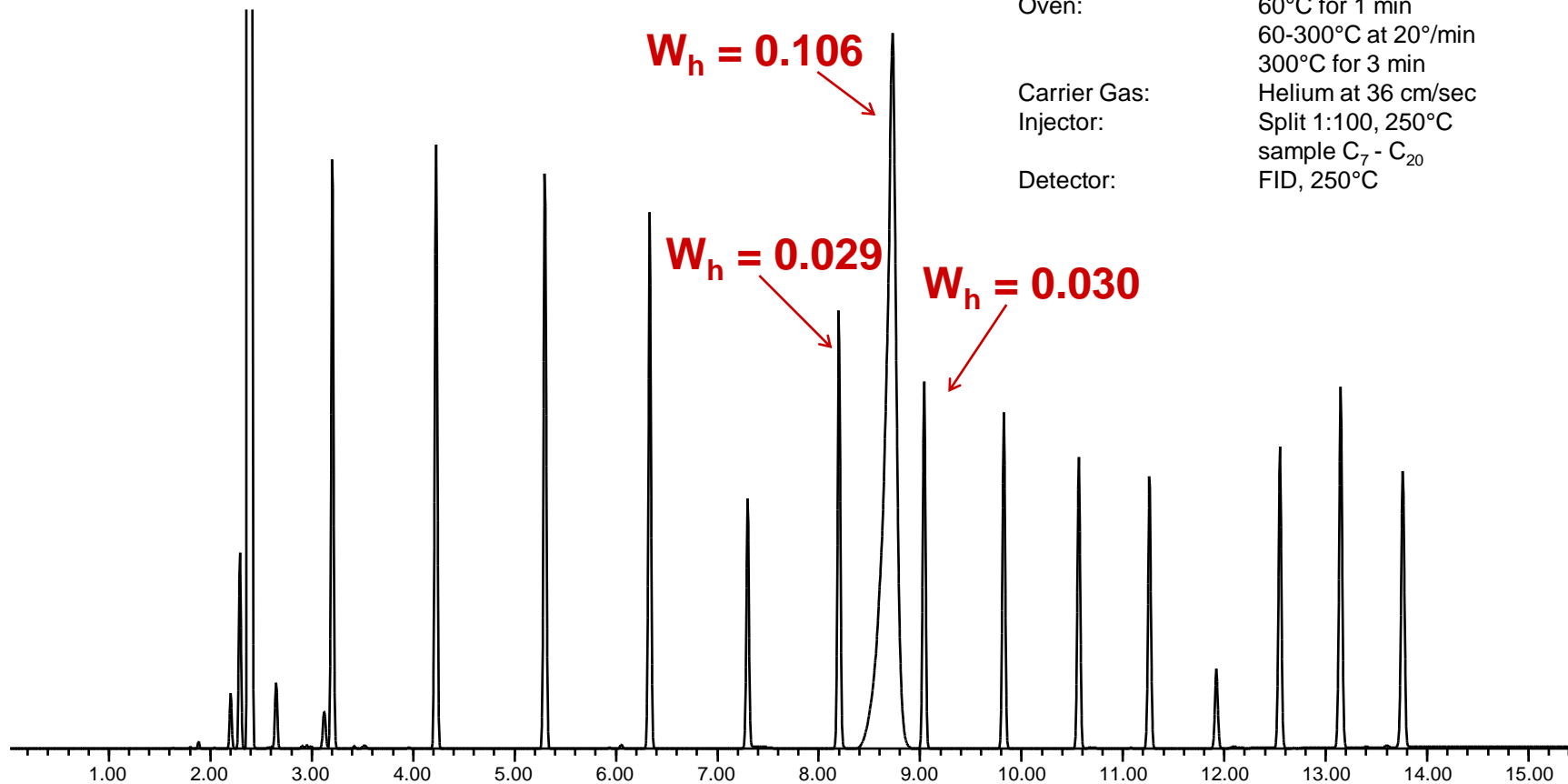
- dead volume, obstruction, poor installation, or  
severe column contamination



Miscellaneous - overloading of PLOT columns, co-elution, polarity mismatch between phase, solute or solvent, and some compounds always tail

\*Tip = Inject a light hydrocarbon, should not tail unless flow path problem.

# Bonus Peaks



**Column:** DB-5  
30 m x 0.53 mm I.D., 1.5  $\mu$ m

**J&W P/N:** 125-5032

**Oven:** 60°C for 1 min  
60-300°C at 20°/min  
300°C for 3 min

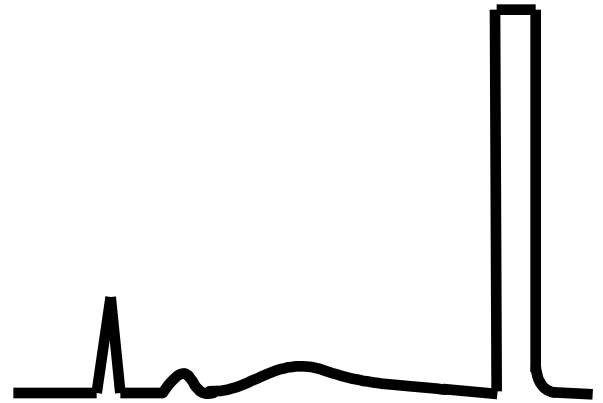
**Carrier Gas:** Helium at 36 cm/sec

**Injector:** Split 1:100, 250°C  
sample C<sub>7</sub> - C<sub>20</sub>

**Detector:** FID, 250°C



# Bonus Peaks or Ghost Peaks

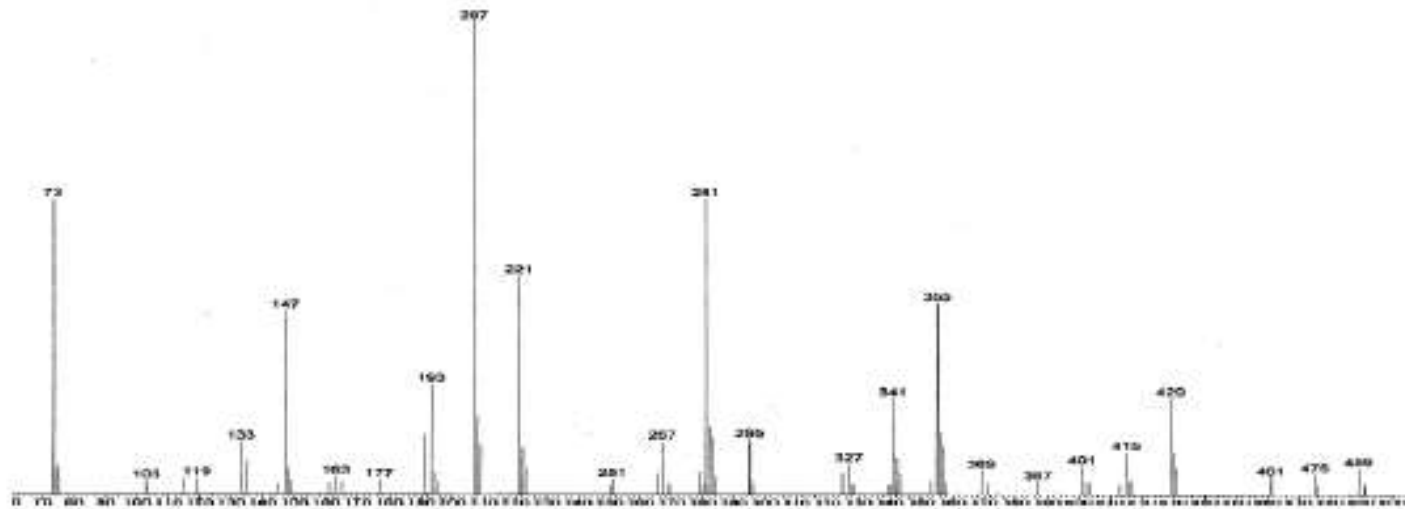
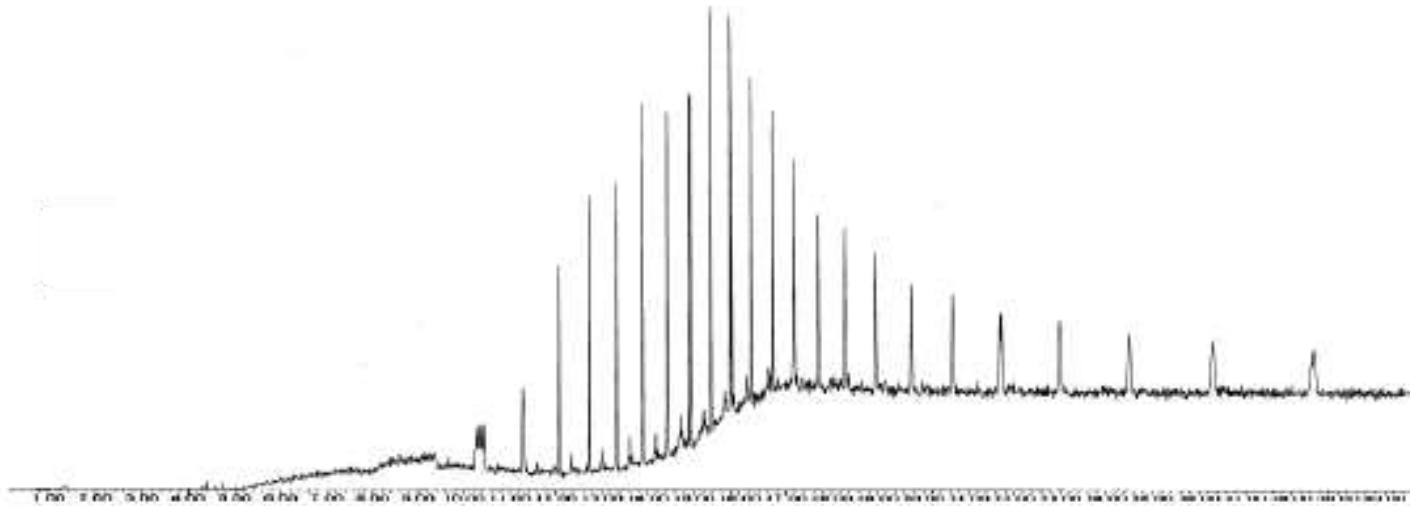


Contamination in INJECTOR, COLUMN or FLOW (carrier gas)

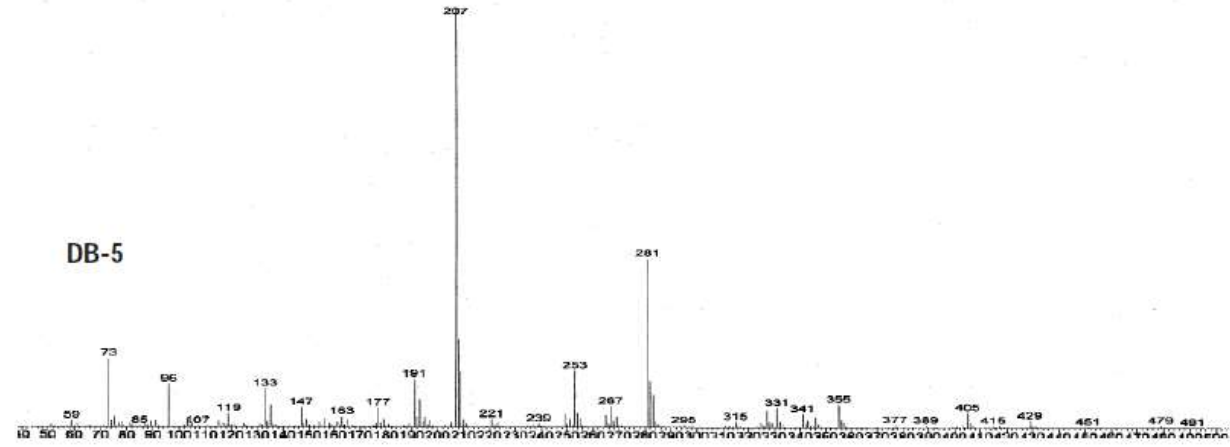
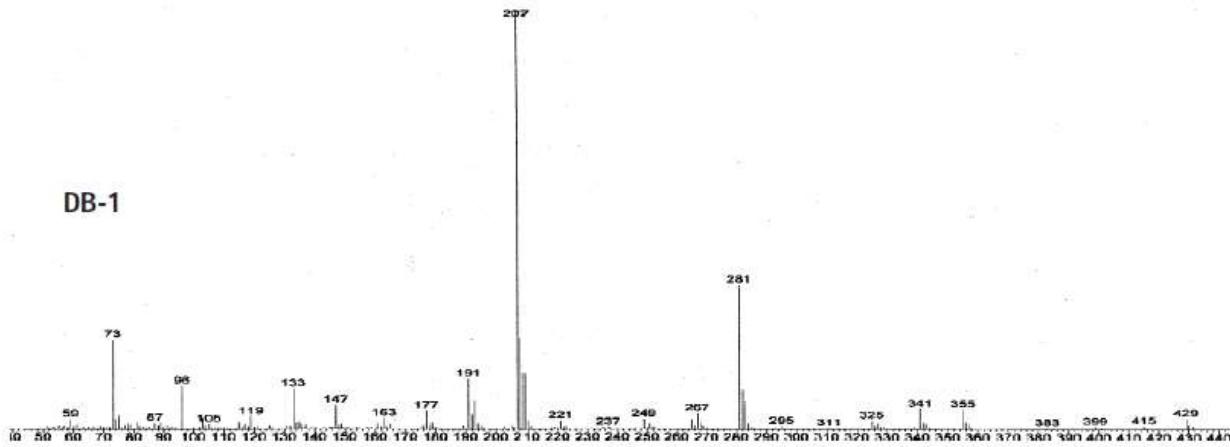
- Carry-over from a backflash or previous sample
- Bad tank of gas or traps have expired
- Septum bleed

**\*TIP = Run a blank run...it should be blank!**

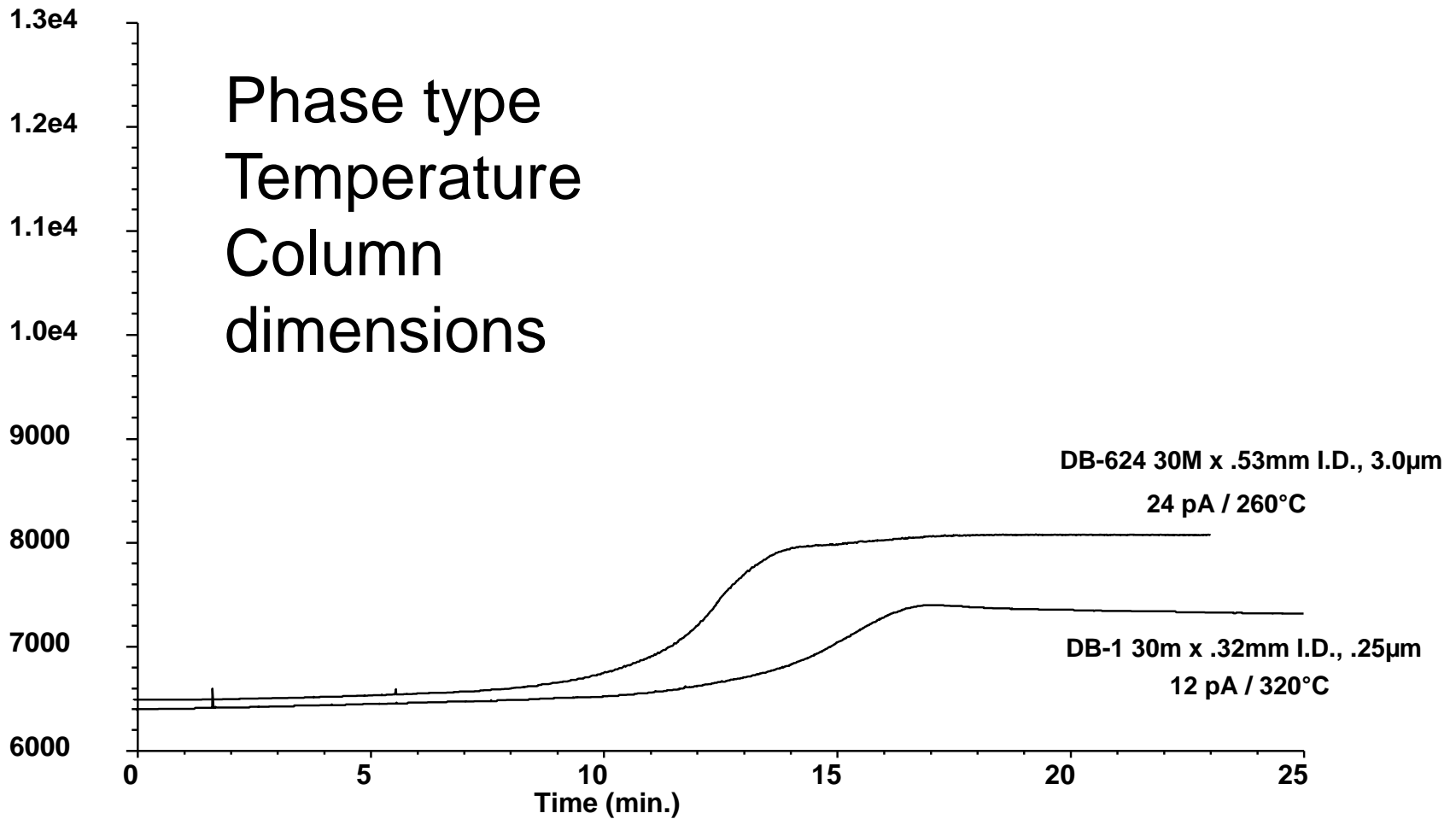
# Bonus 'Siloxane' Peaks



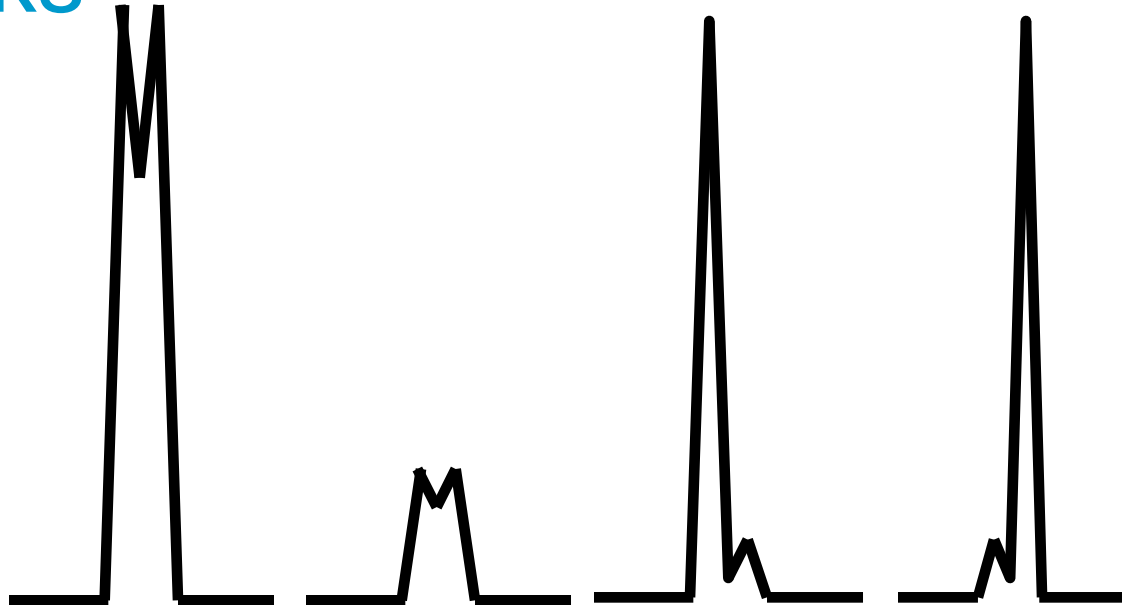
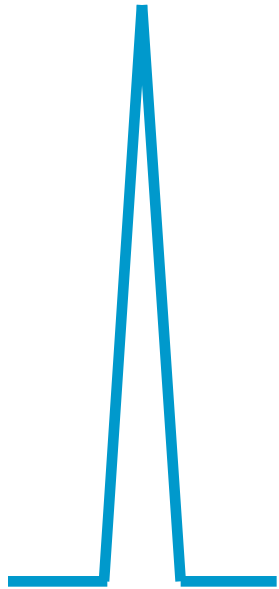
# GC Column Bleed Ions



# Column Bleed is Influenced by:



# Split Peaks



**INJECTOR** (poor sample introduction)

- Injecting the sample twice (some how?)
- Mixed sample solvent (polarity difference)
- Sample in syringe needle (manual inject)

**INJECTOR** (activity)

- Breakdown (not really a split peak, 2 peaks)
- Sample degradation in injector

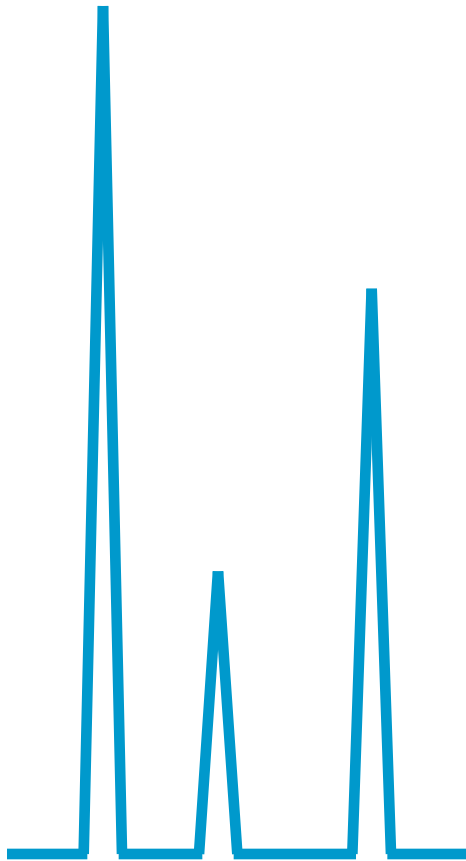
**VOLATILITY**

High boilers dropping out on Cold Spots

-Transfer line temps

-Unions or fittings not tracking column temp

# No Peaks



**DETECTOR** (not on or not operational)

**INJECTOR** (not working)

-Plugged syringe/plunger not moving

-Wrong injector (or detector)

-Huge leak (older systems)

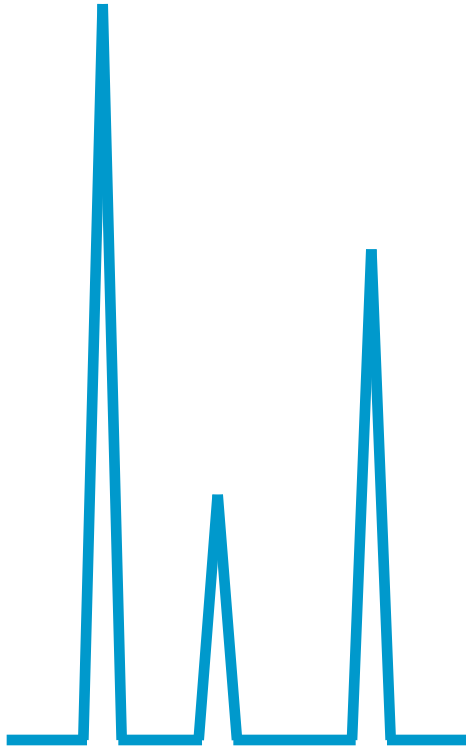
-No carrier gas flow

NOT the COLUMN Unless...

-Broken column or No column

# Peak Response

All Change in Size



## INJECTOR

- Leaky syringe
- Split ratio set incorrectly
- Wrong purge activation time
- Septum purge flow too high
- Injector temperature too low\*

## DETECTOR (response problem)

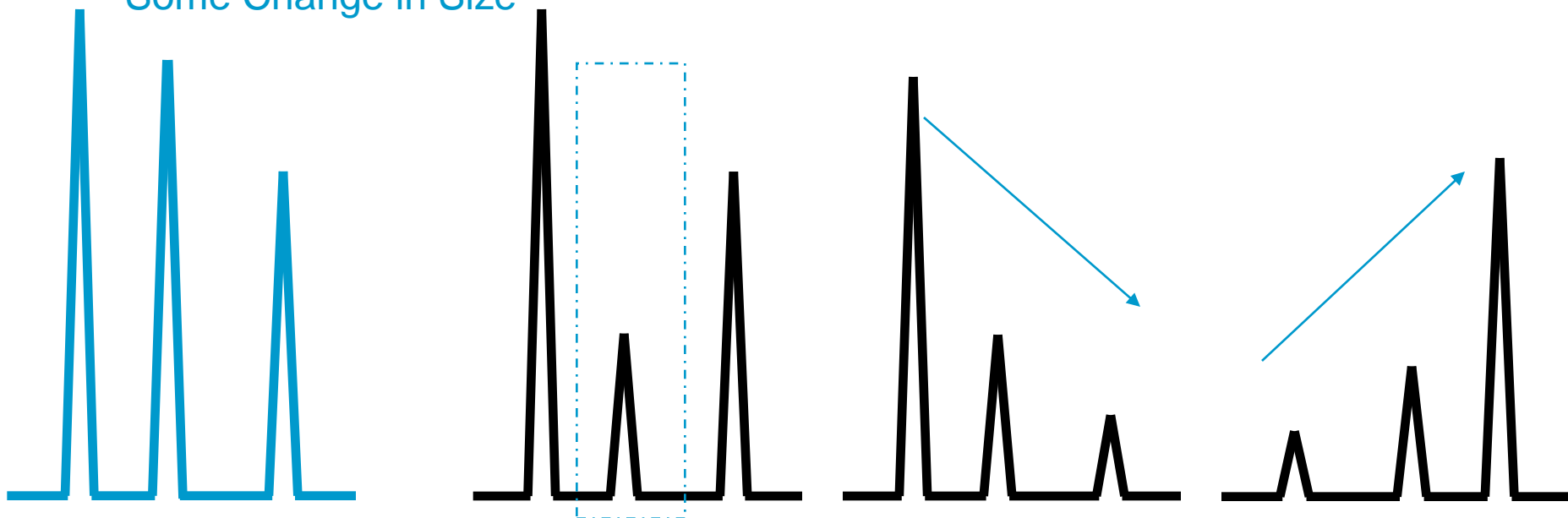
- Settings or flows changed
- Electronics failing



\*Tip = Ask is it all of them or some of them, if all then injector or detector

# Peak Response

Some Change in Size



INJECTOR or COLUMN is active/contaminated

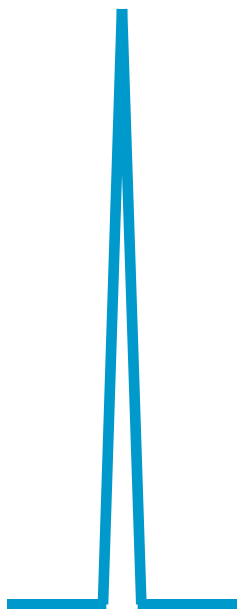
- Irreversible adsorption of active compounds (-OH, -NH, -SH)
- Decomposition of sample
- Temperature Change – Discrimination
- Evaporation from sample

\*Tip = If only some change, then ask which ones? If active compounds then activity. If tracks volatility then cold spots or inlet discrimination.



# Peak Fronting

Shark Fin Shaped or Just Slight



## **COLUMN** (contaminated)

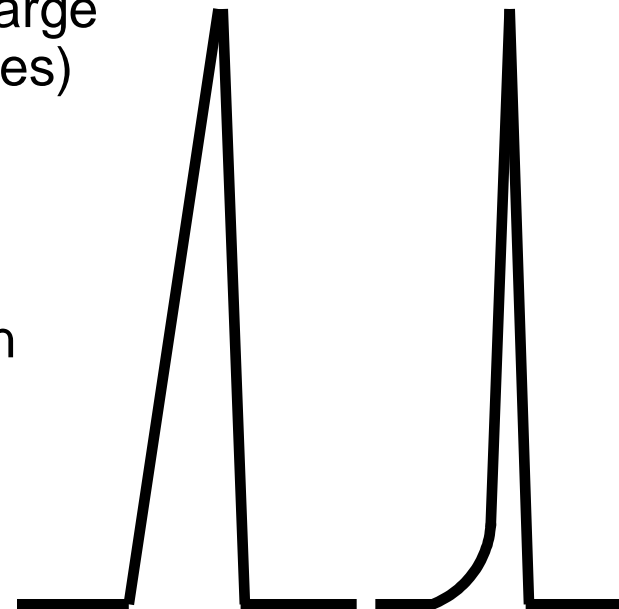
- Overload (More pronounced with large solute and phase polarity differences)

## **INJECTOR**

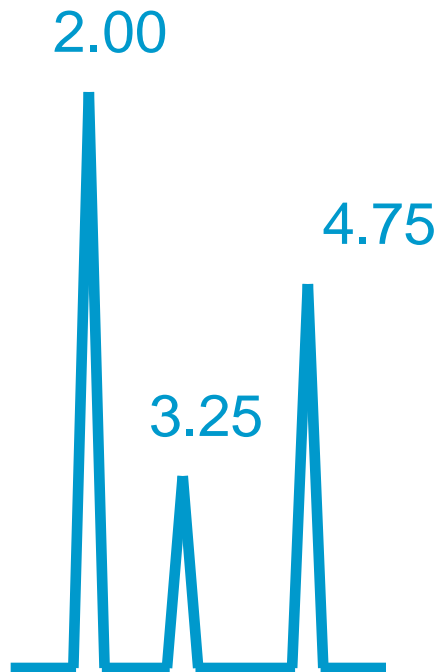
- Column installation
- Compound very soluble in injection solvent (need retention gap)
- Mixed sample solvent

## **OTHER**

- Co-elution
- Breakdown



# Retention Time Shift



## INJECTOR

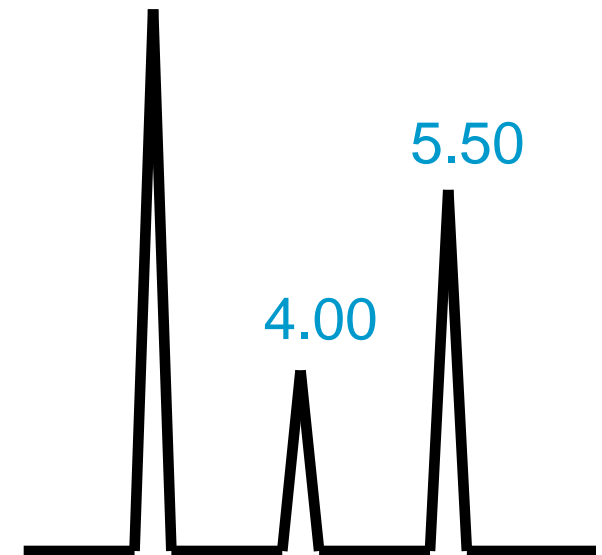
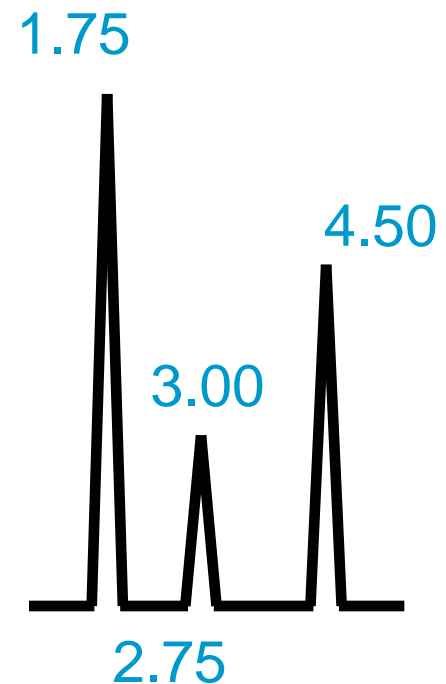
- Leak in the septum
- Change in injection solvent
- Large change in sample concentration

## FLOW

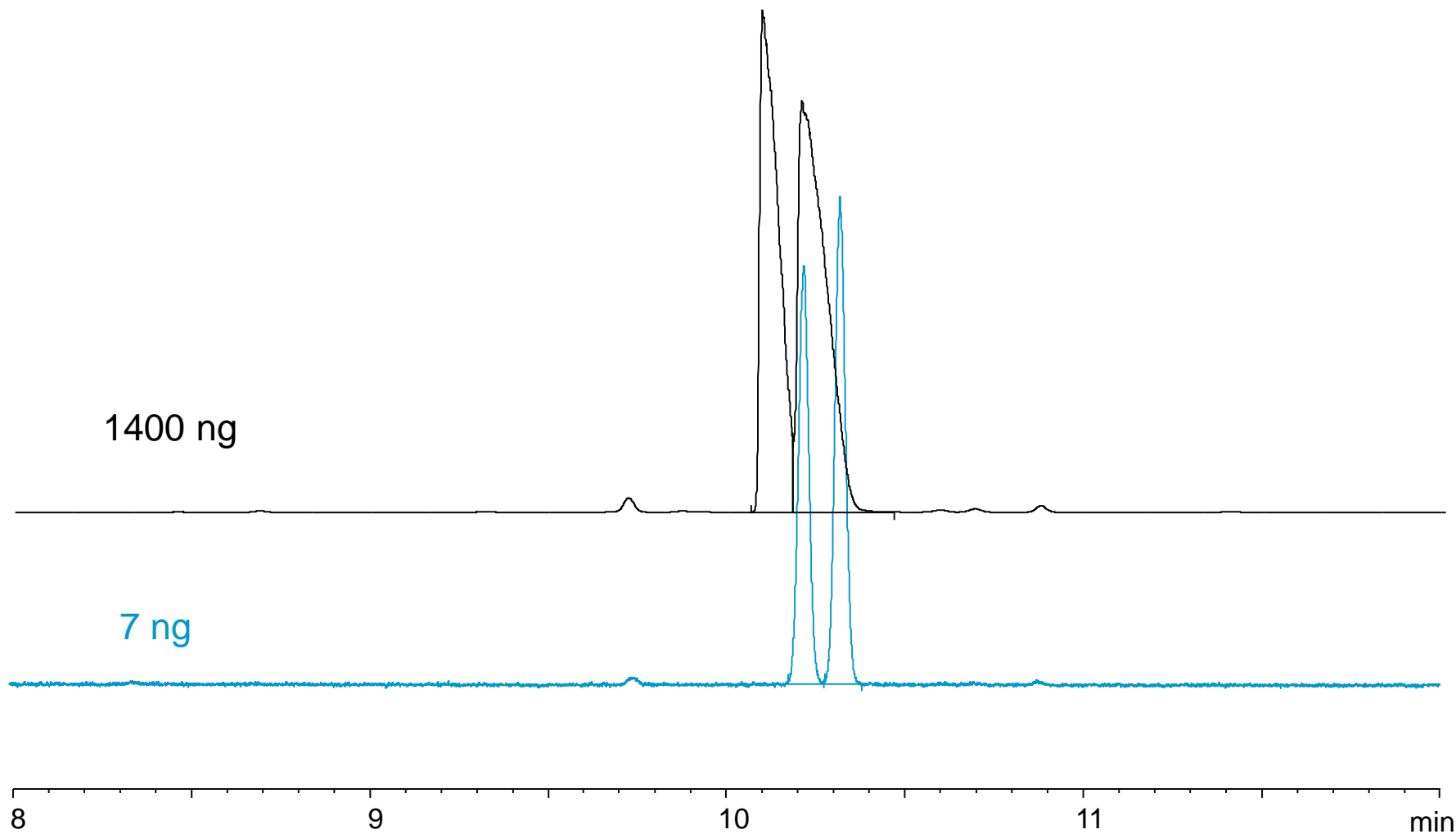
- Change in gas velocity

## COLUMN

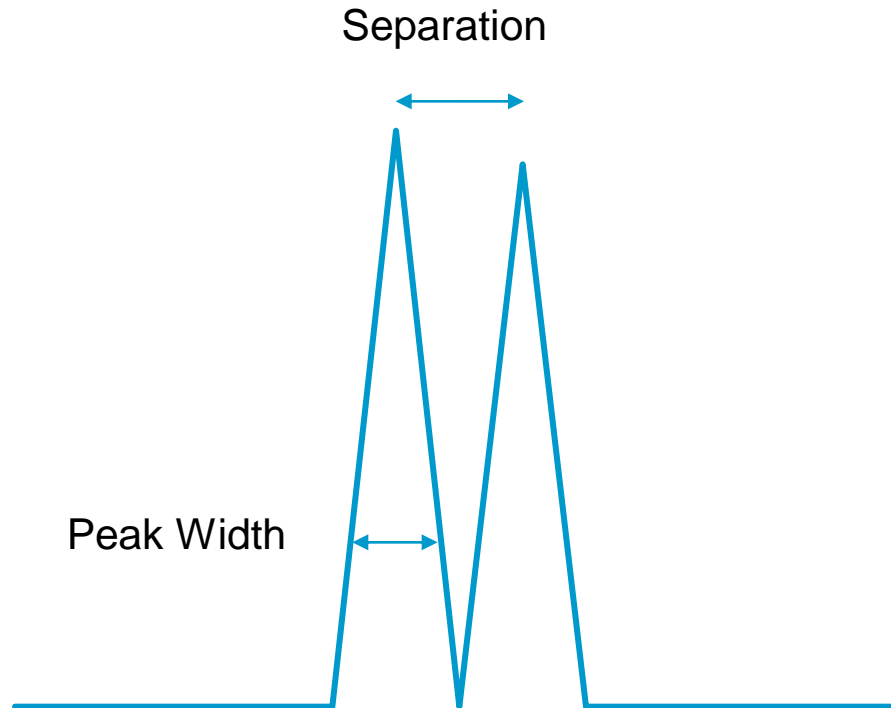
- Contamination
- Damaged stationary phase
- Loss of stationary phase
- Change in temperature



# Effect of Sample Overload on Retention Time and Peak Shape



# Loss of Resolution

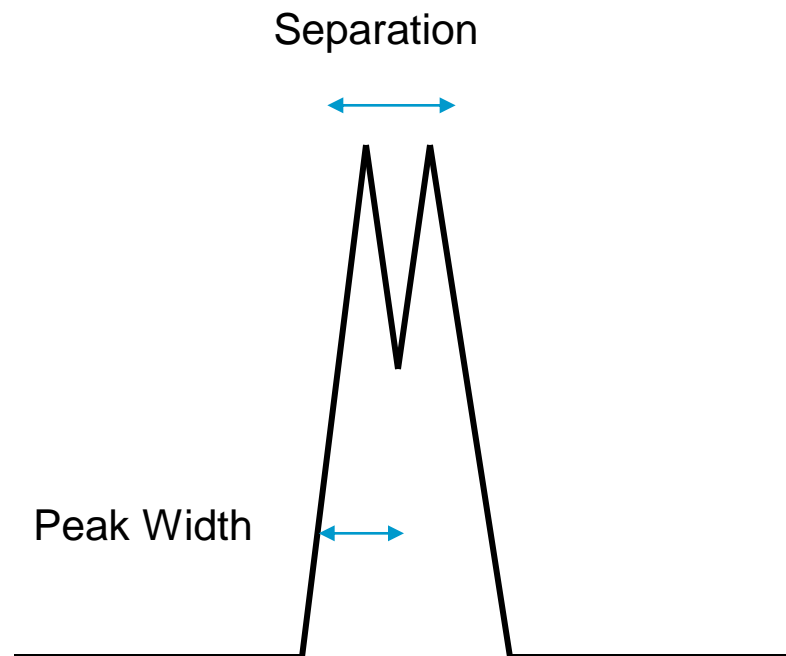


**Resolution is a function of separation and peak width**

# Loss of Resolution - Separation Decrease

## COLUMN

- Different column temperature
- Contamination (more phase?)
- Matrix components co-eluting
- Different column phase?



# Loss of Resolution - Peak Broadening

## FLOW

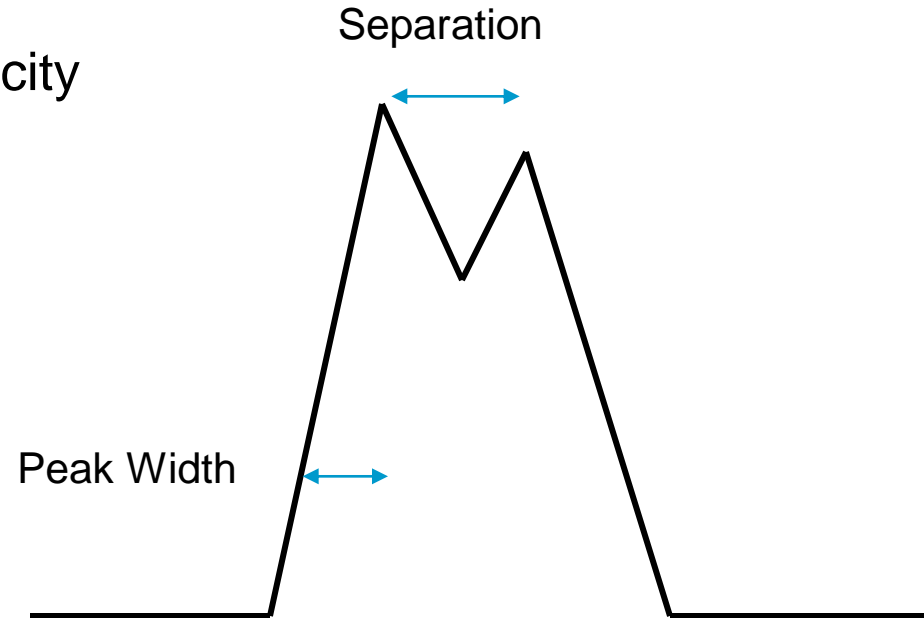
- Change in carrier gas velocity
- Make-up gas

## COLUMN

- Contamination
- Phase degradation

## INJECTOR (efficiency)

- Settings, Liner, Installation, etc.



# Baseline Disturbances

Sudden Changes, Wandering, or Drifting

WANDER



COLUMN or DETECTOR

-Not fully conditioned or stabilized (electronics)

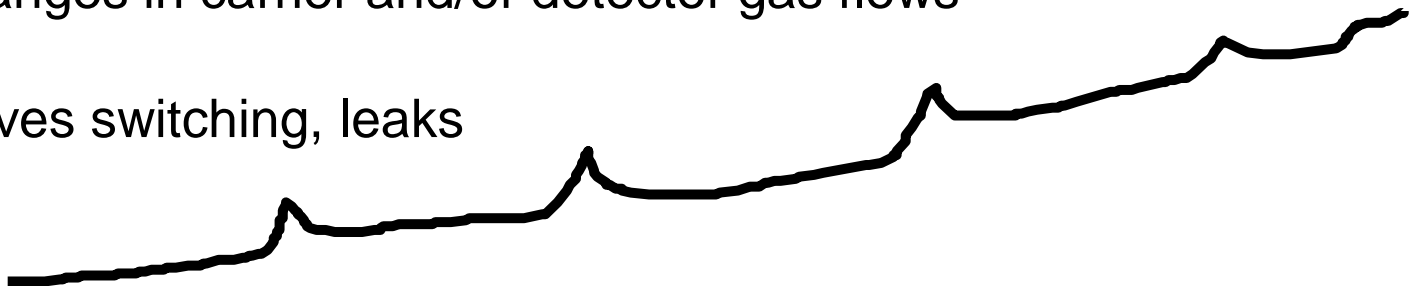
-Contamination

FLOW

-Changes in carrier and/or detector gas flows

-Valves switching, leaks

DRIFT



# Noisy Baseline

MILD



SEVERE



FLOW

- Contaminated gas
- Incorrect detector settings

COLUMN

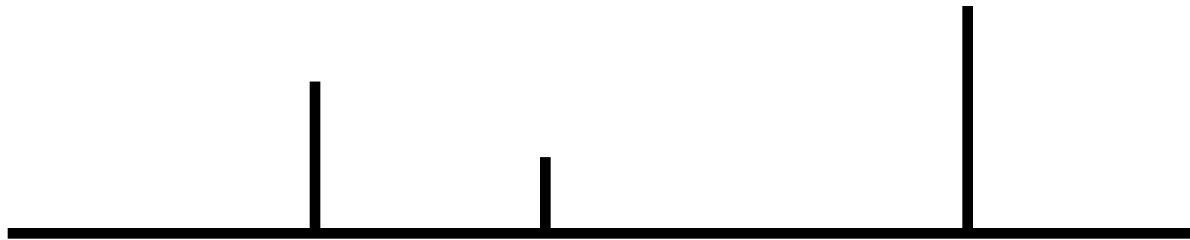
- Bleed if at high temperature
- In detector flame (poor installation)

DETECTOR

- Air leak - ECD, TCD
- Electronics malfunction



# Spiking Baseline



## DETECTOR

- Particles entering the detector
- Random: poor connection
- Regular: nearby "cycling" equipment (electronics)

# Quantitation Problems

## DETECTOR

- Poor stability (electronics) or Baseline disturbances (contamination)
- Outside detector's linear range or wrong settings

Activity (adsorption) in INJECTOR or COLUMN  
392538403

## INJECTOR

- Technique, settings, conditions
- Syringe worn

## OTHER

- Co-elution
- Matrix effects
- Sample evaporation – leaky vials
- Sample decomposition

# What is NOT caused by a Column???

## Peaks!!

Any reproducible, sharp 'chromatographed' peak!

Siloxanes

Degradation product peaks: Endrin Aldehyde, Endrin Ketone, DDE, DDP....

Carryover of sample compounds

Splitting of peaks

# Troubleshooting “Tools”

Bleed Profile: *baseline problems*

Inject a non-retained peak: *peak shape problems*

Test mix: *all problems*

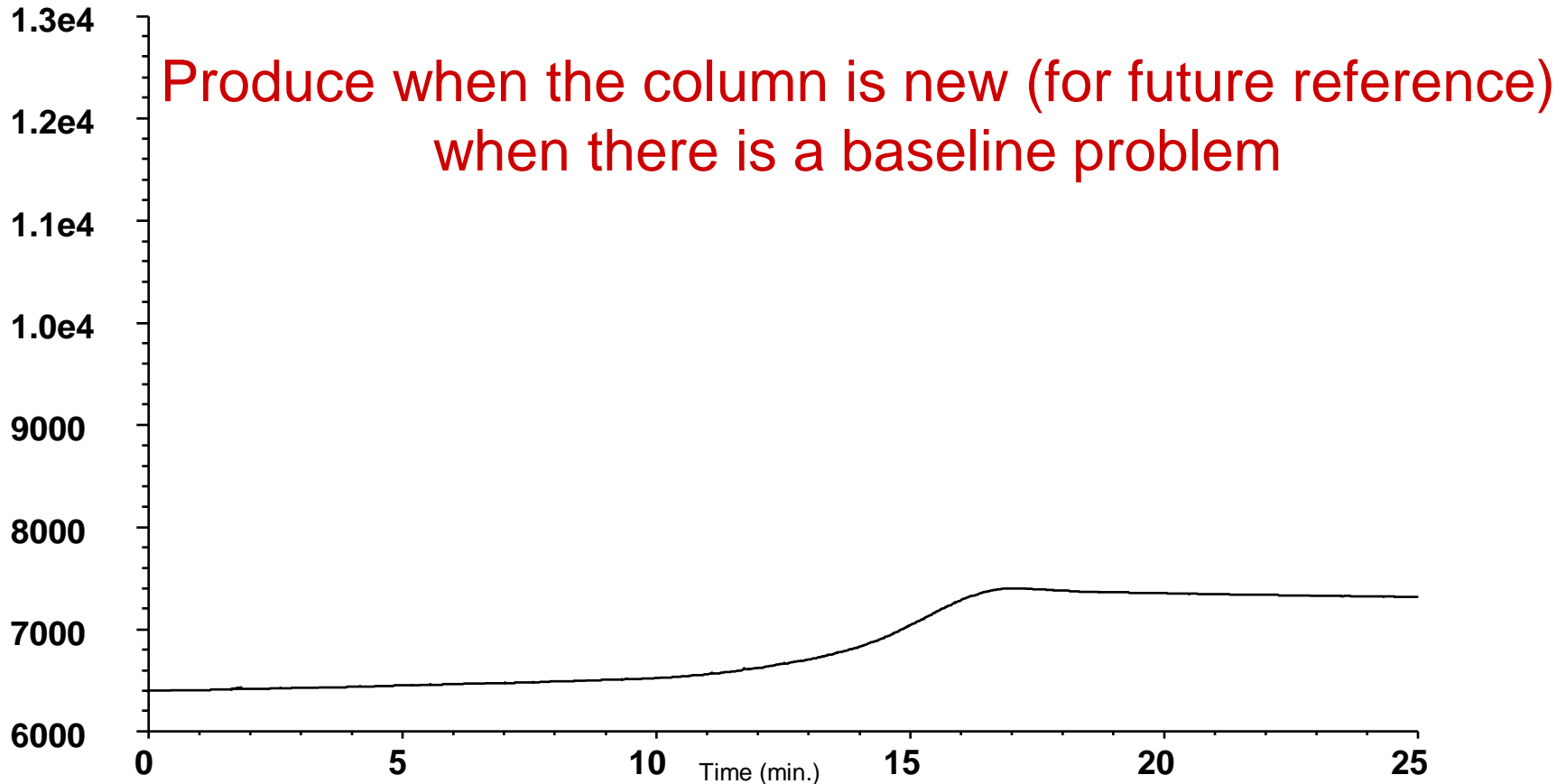
Isolate the components: *all problems*

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Condensation Test: *baseline problems*

Jumper Tube Test: *baseline problems*

# Generating a Bleed Profile

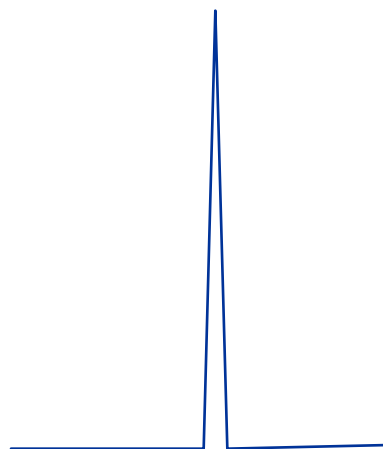


\*DB-1 30m x .32mm I.D., .25 $\mu$ m

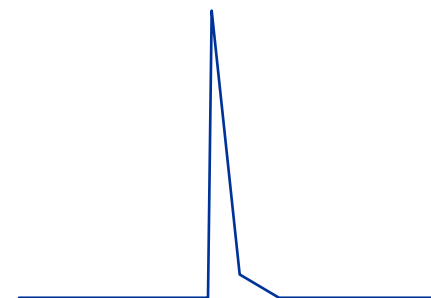
Temperature program // 40°C, hold 1 min // 20°/min to 320°C, hold 10 min.

# Non-Retained Peak Shapes

Used to Check  
Flowpath



Good Installation



Improper Installation or  
Injector Leak

Potential problems:

- Injector or septum leak

- Too low of a split ratio

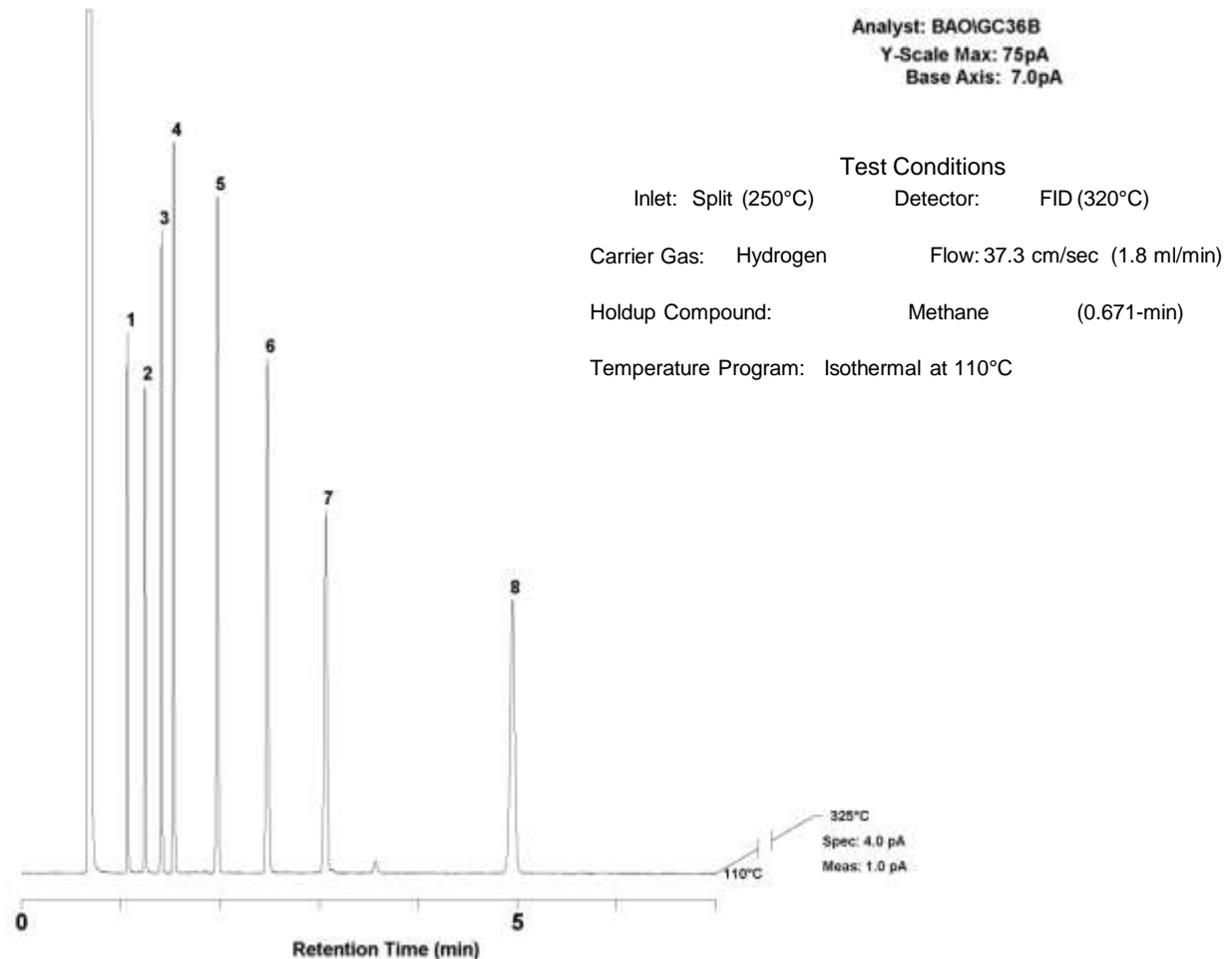
- Liner problem

  - (broken, leaking, misplaced)

- Column position in injector and detector

# Test Mix

Used to determine how “good” the column is or if the problem is related to the chemical properties of the analytes.



# Test Mixture Components

## Compounds

Hydrocarbons

Alcohols

FAME's, PAH's

Acids

Bases

## Purpose

Efficiency

Retention

Activity

Retention

Acidic Character

Basic Character



# Own Test Mixture

- More specific to your application
- Selective detectors
- Concentrations specific to your application
- Use same instrument conditions
- Easiest to simply inject a calibration standard
- Store for future measure of column performance

# Isolate the Components

Simplify the system:

- example - Direct injection instead of P&T sample introduction

Put in a known good column

Move column to a different GC, inlet or detector

# Condensation Test



Used\* to isolate the cause of:

- Erratic baselines
- Ghost peaks or carryover

**\*Use when problems are worse after periods of GC non-use**

# Condensation Test

## Procedure

Leave GC at 40-50°C for > 8 hours

Blank run

Repeat a blank run immediately after the first blank run is complete

Compare the two blank runs

# Condensation Test

## Results

First blank run is worse:

- Contaminants (from injector, lines, traps or carrier gas) carried into the column

Blank runs the same: *contaminants are not strongly focused on the front of the column*

# Jumper Tube Test

## Purpose

Helps to locate the source of contamination or noise

Isolates GC components

# Jumper Tube Test

## Isolate the Detector

Remove column from the detector

Cap detector and turn on

Blank run

# Jumper Tube Test

## Isolation of Detector - Results



Detector OK



Detector is the problem





# Jumper Tube Test

## Isolate the Injector

Connect the injector and detector

- 1-2 meters deactivated fused silica tubing

Turn on carrier gas

Blank run

# Jumper Tube Test

## Isolate the Injector - Results



Injector OK



Injector, lines or carrier  
gas contaminated

# Jumper Tube Test

## Isolate the Column

Reinstall the column

Setup as before

Blank Run

# Jumper Tube Test

## Isolate the Column - Results

Problem returns: It's the column

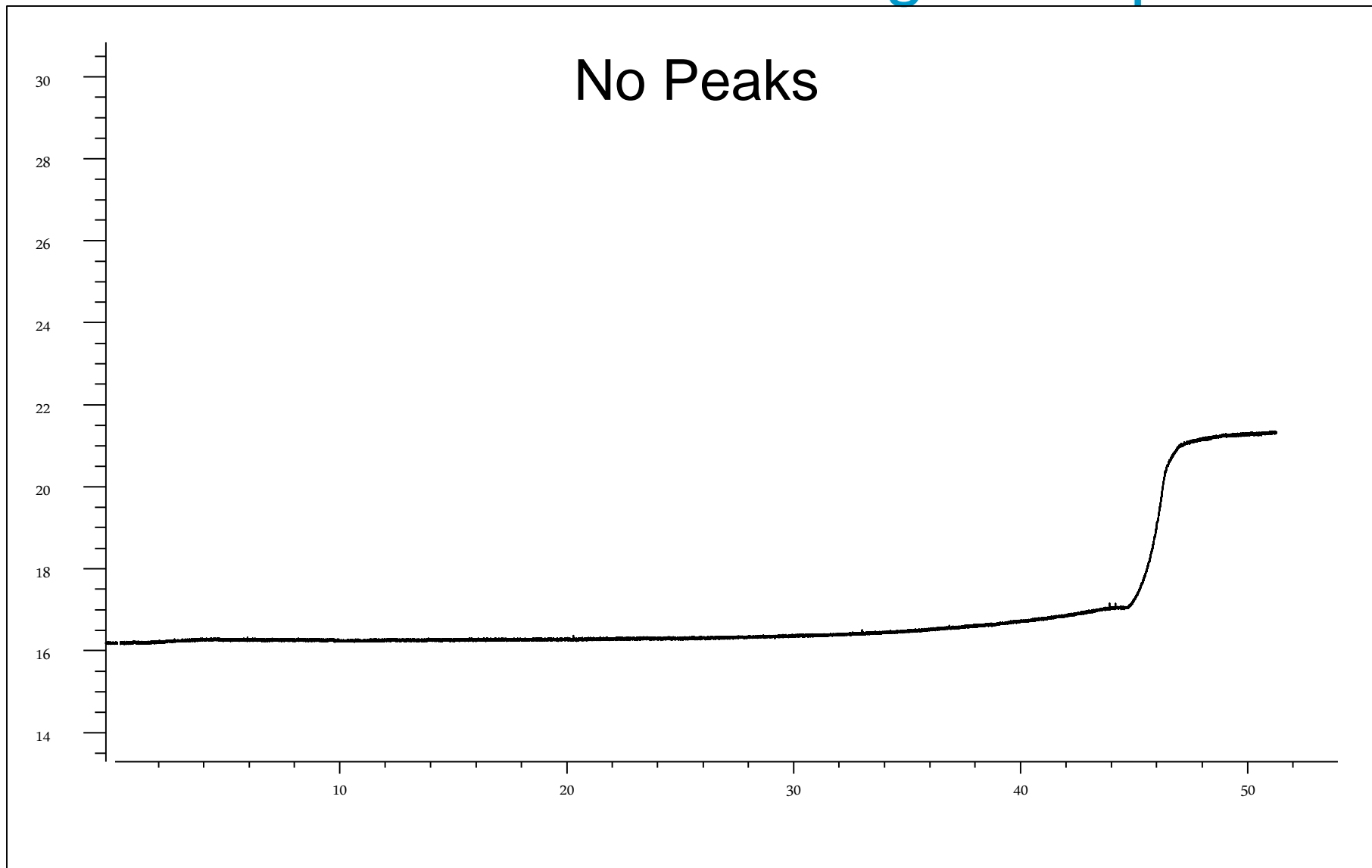
Problem gone: Previous leak, solid debris, or installation problem

And Now Let's do Some

# TROUBLESHOOTING

# Troubleshooting-Example #1

# A Real Troubleshooting Example



# Logical Steps Taken to Find Peaks

(most of our problems are leaks and plugs)

## Is the flame Lit?

- put glass piece over FID outlet----*Answer in this case, Water condenses*
- look at output in instrument guage-- is the digital value greater than 0.0?  
*Answer in this case is approximately 16.2 pico amps*

## Is there flow through the column?

- disconnect column from detector and measure flow with bubble solution or meter  
*Answer in this case was YES THERE IS FLOW*

## Assess the observations

- *Flame is lit and we have flow from end of column*
- *Hypothesis: Sample not getting on column-syringe plugged?*

Take syringe out and make injection manually on a dry paper towel

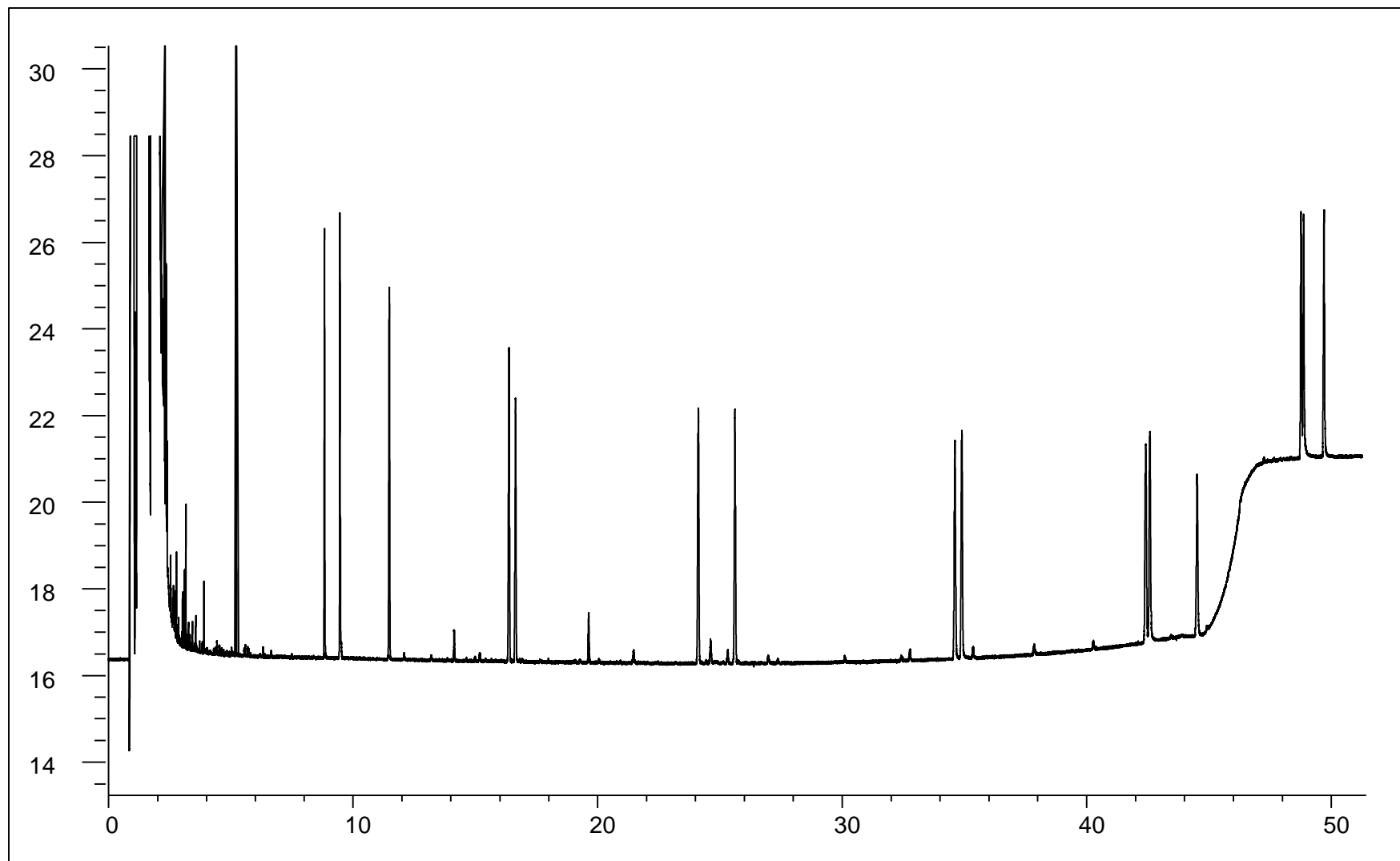
*Answer – towel stays dry (Syringe was clogged with septum)*

Pull plunger out top, add solvent and replace plunger will usually dislodge septum particle (should hear a little pop) If you can't dislodge plug, Replace syringe

## Reassemble the Injector & Re-inject



# Peaks !!

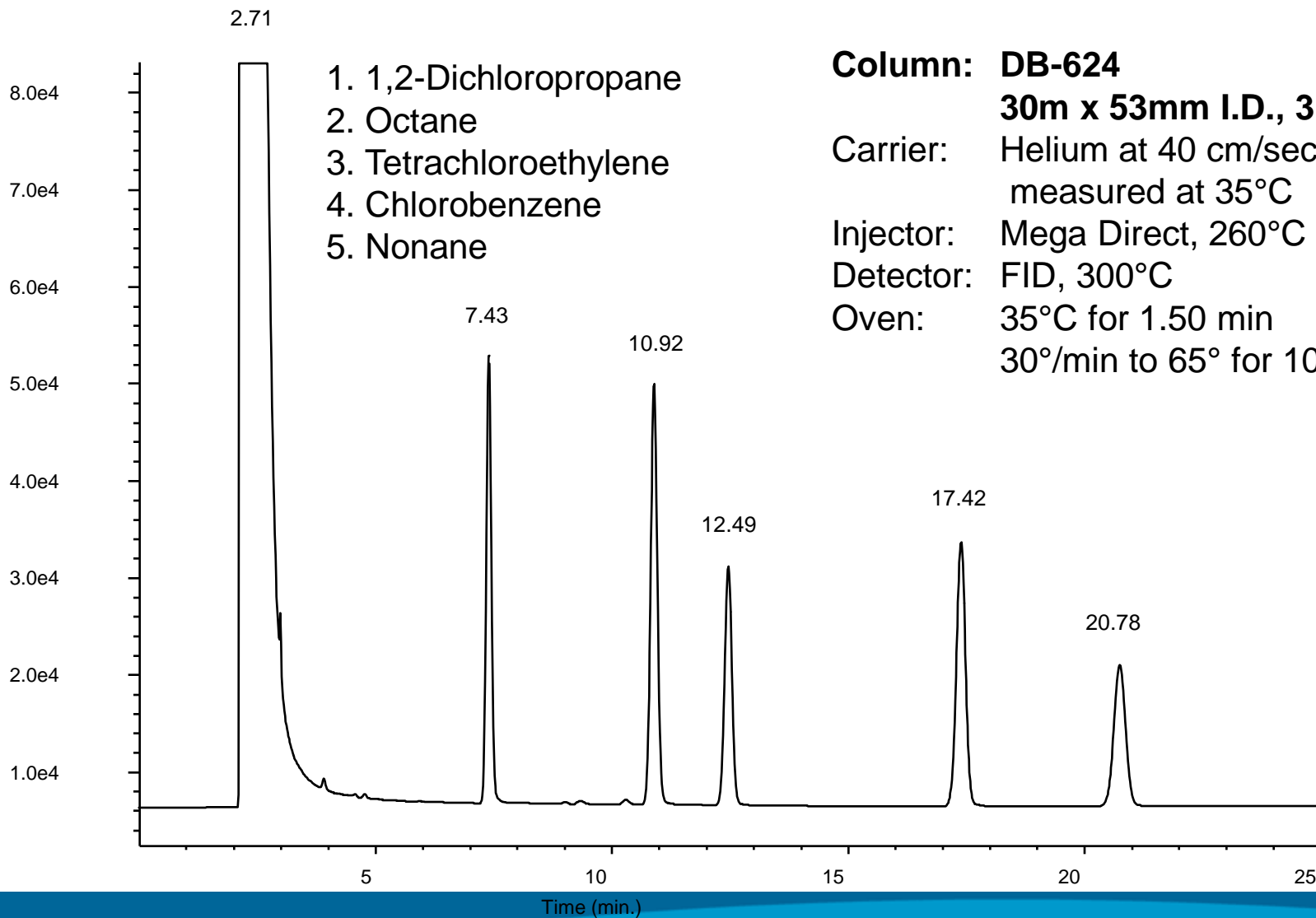


# Troubleshooting-Example #2

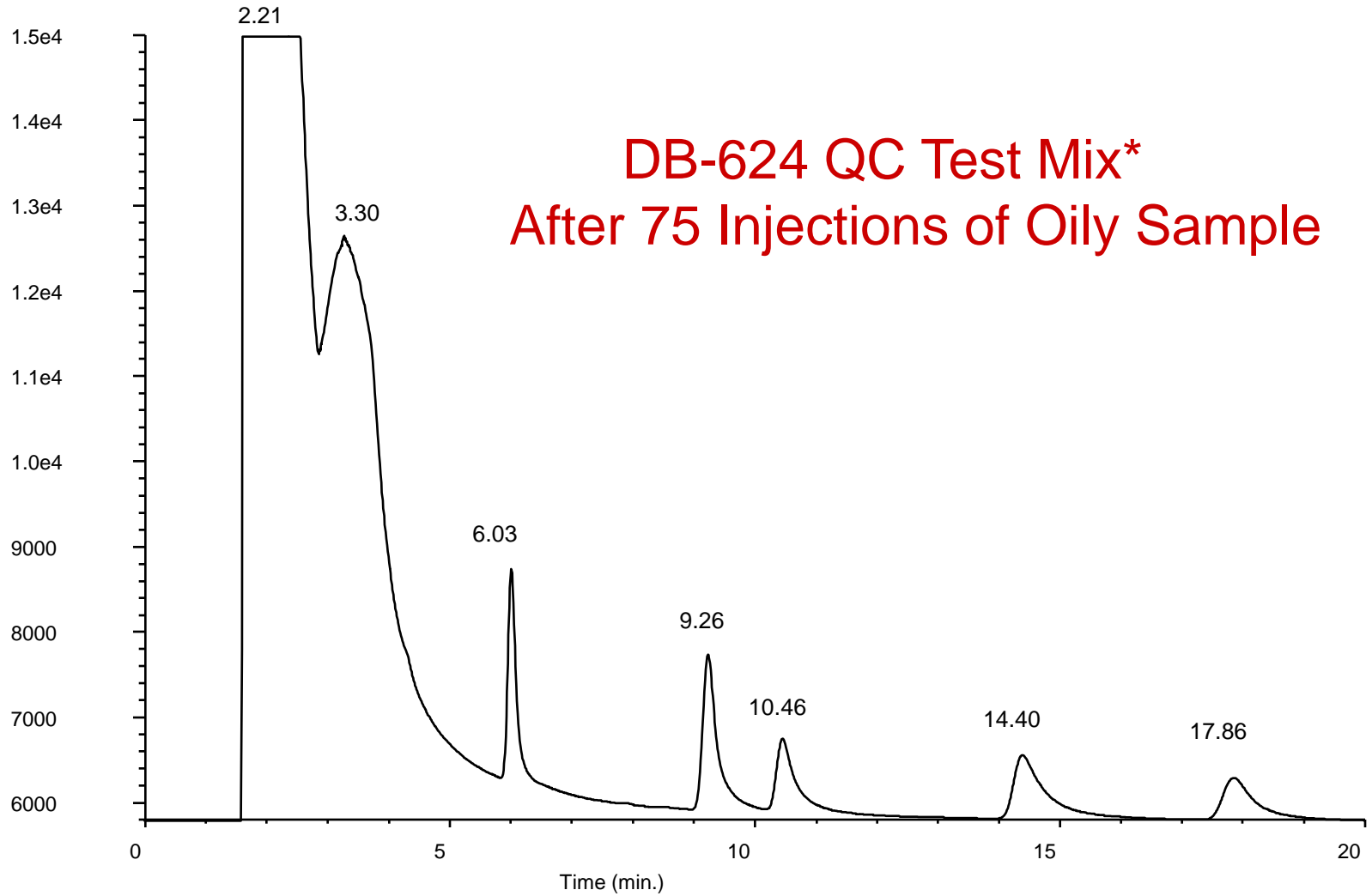
# DB-624 COLUMN

## QC Test Mix

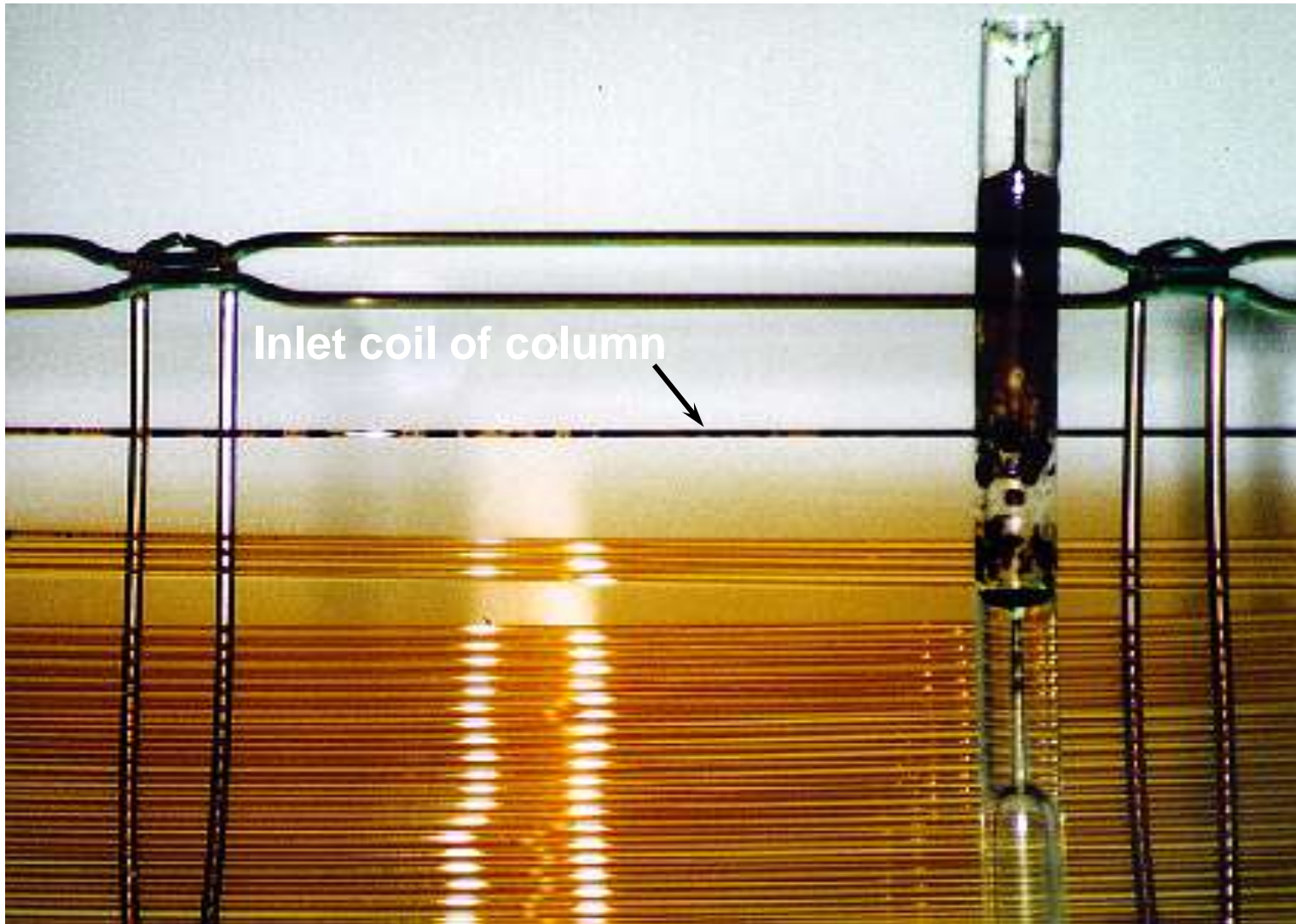
**Column:** DB-624  
**30m x 53mm I.D., 3.0 $\mu$ m**  
**Carrier:** Helium at 40 cm/sec  
measured at 35°C  
**Injector:** Mega Direct, 260°C  
**Detector:** FID, 300°C  
**Oven:** 35°C for 1.50 min  
30°/min to 65° for 10 min



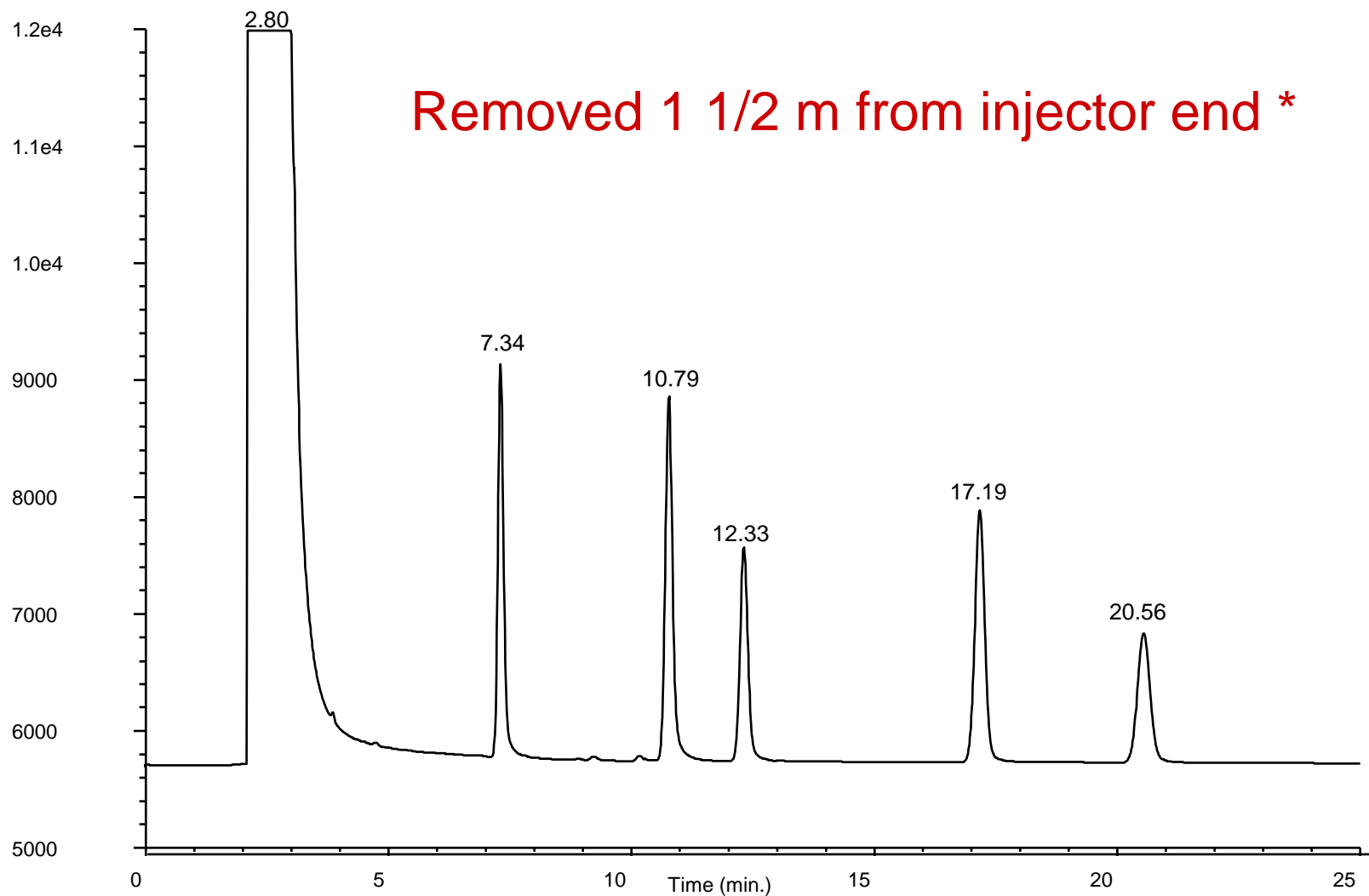
# Example of Column Contamination



# Column and Liner Contamination



# Example of Column Contamination



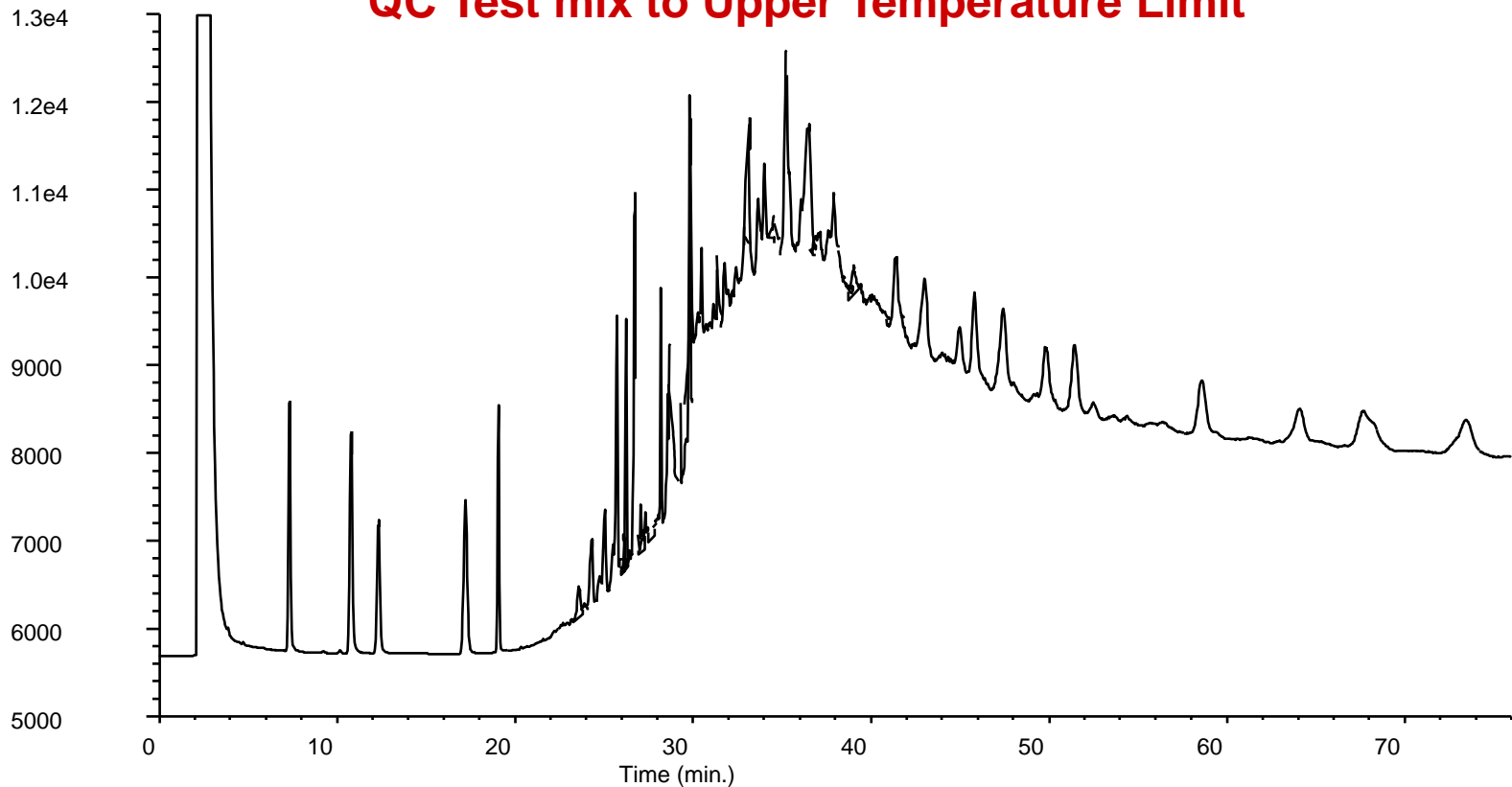
\*Before Column rinse and bake

Temperature program // 35°C hold 1.50 min // 30°/min to 65°C, hold 10 min

# Looks Fixed Doesn't it?

# Example of Column Contamination

**1 1/2 mtrs removed\***  
**QC Test mix to Upper Temperature Limit**



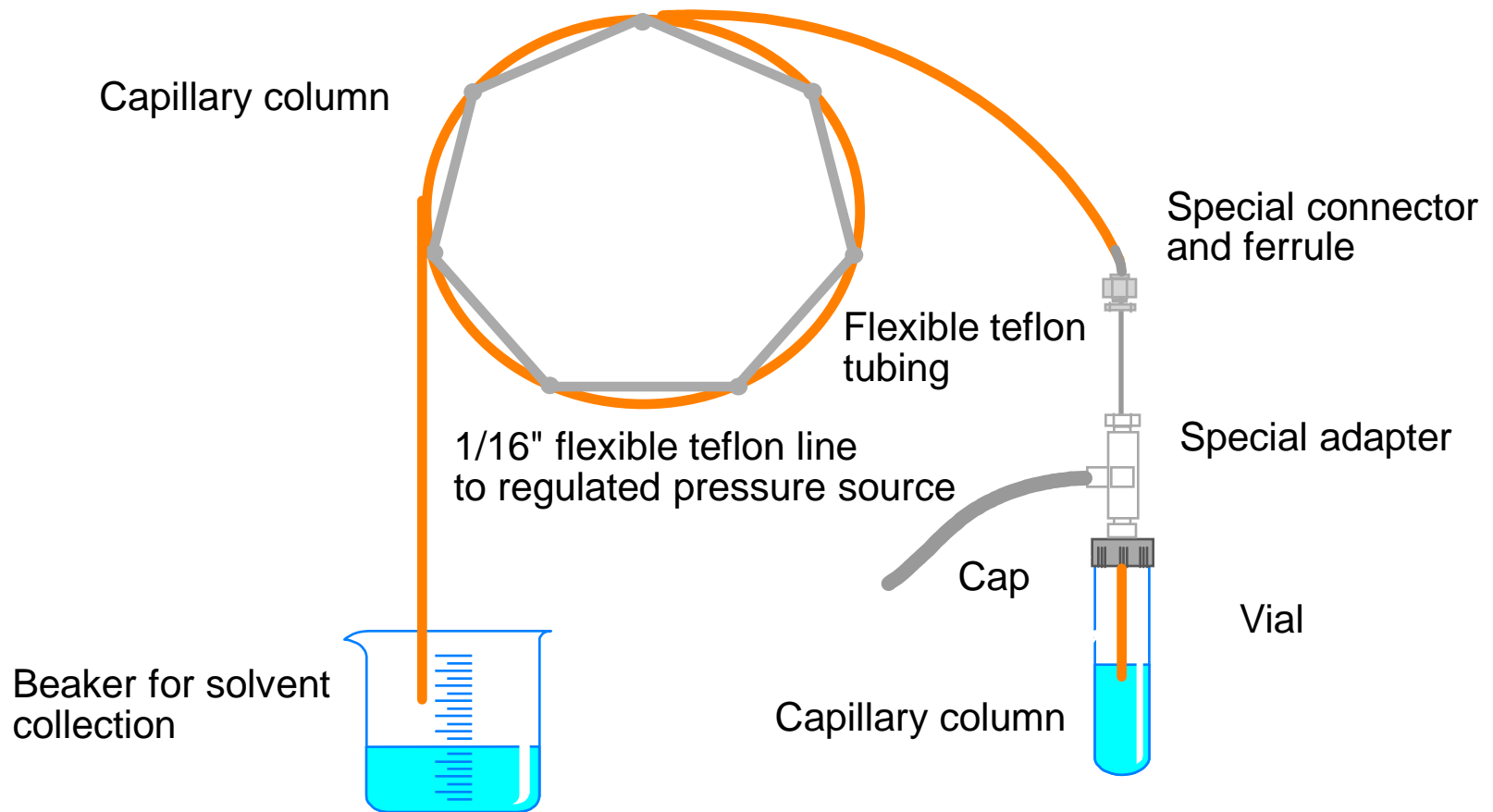
**\*Before Column rinse and bake.**

**Temperature program // 35°C, hold 1.50 min // 30°/min to 65°C,  
hold 15 min // 20°/min to 260°, hold 50 min**



# Backflush Column

Rinse with 10ml each:  
Methanol, Methylene Chloride, Hexane

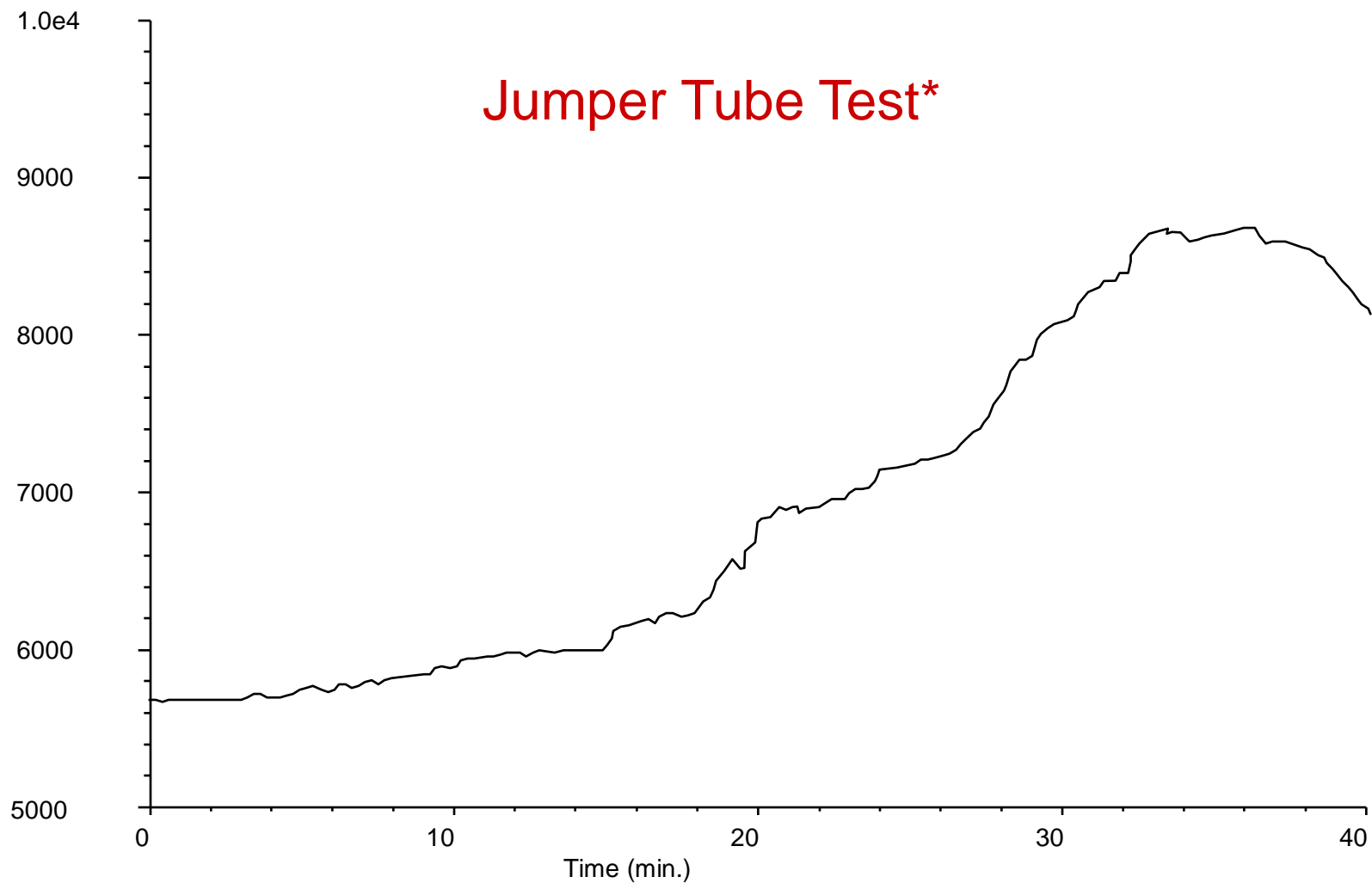


# Jumper Tube Test

## Used to Isolate Source of Contamination

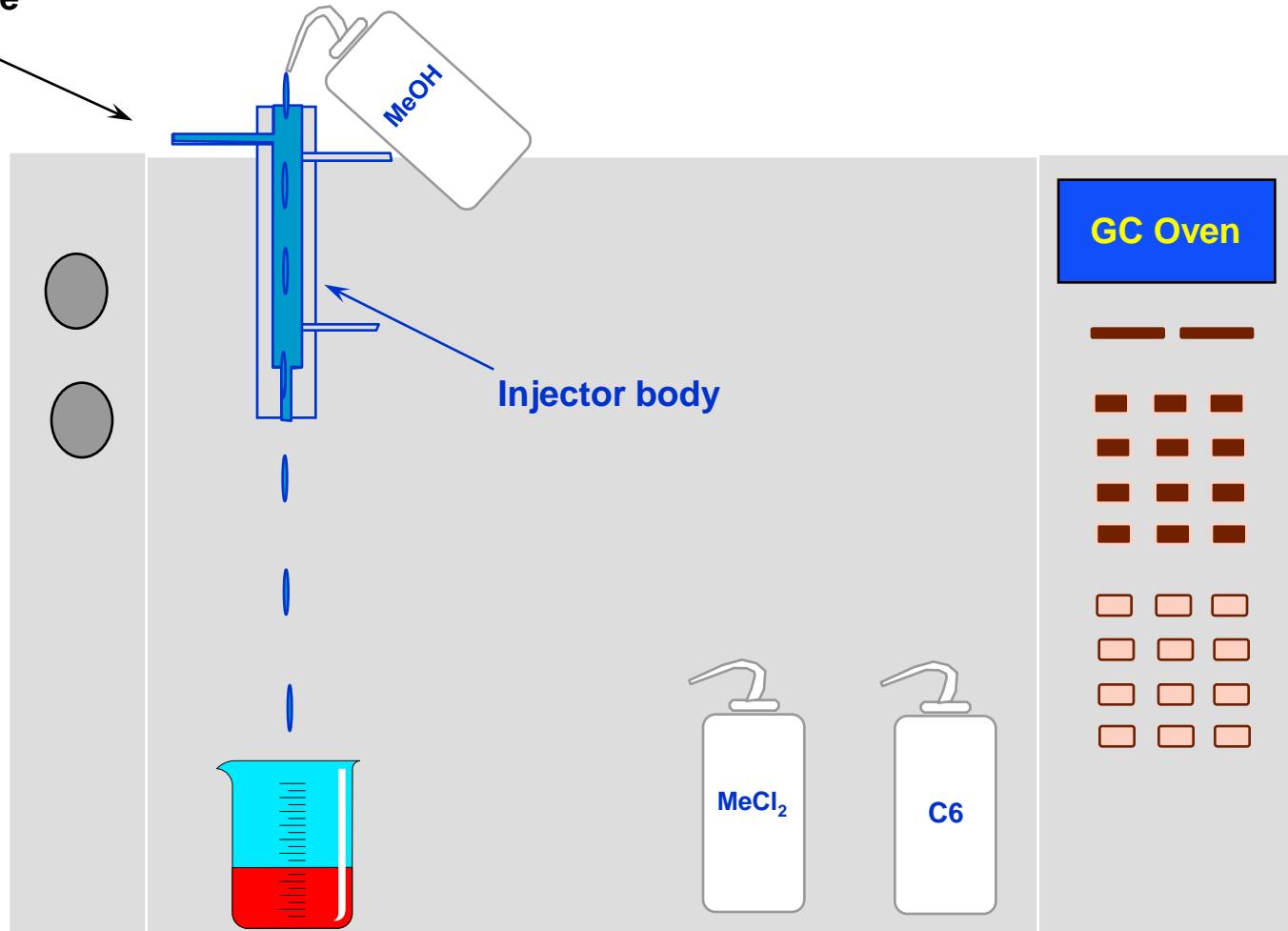
- . Cap off the detector and establish normal gas flows and temperature.
- . Plot the baseline using a temperature program. If flat.....
- . Connect 1 meter of deactivated tubing between the injector and detector
- . Plot the baseline using a temperature program. If flat.....
- . Install the column.
- . Plot the baseline using a temperature program.

# Contaminated Inlet



# Rinsing Injector

Carrier gas line



# Troubleshooting Tips

## 1. Isolate the problem.

(Blank Run, Inject Un-retained Compound, Jumper Tube Test)

## 2. Change only one variable at a time.

## 3. Compare before/after chromatograms.

(Peak shape, response, retention, baseline rise, background, look for trends, etc.)

## 4. Utilize Technical Support.

# Remember

Complete system = Carrier Gas + Injector +  
Column + Detector + Data System

Multiple cause and effect

Do not change too many variables at once

# TECHNICAL SUPPORT

1-800-227-9770, #3, #3, #1

**E-mail:** [gc-column-support@agilent.com](mailto:gc-column-support@agilent.com)

