

# Installation, Care and Maintenance of Capillary Gas Chromatography Columns

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# Installation, Care and Maintenance of Capillary Gas Chromatography Columns

**or....**

**"It's not what your column can do for you, but what you can do for your column"**

# Column Installation

**"Getting off to a good start"**

# Column Installation Procedure

- **Install the column**
- **Leak and installation check**
- **Column conditioning**
- **Setting linear velocity or flow rate**
- **Bleed profile**
- **Test mix**

# Column Installation

**What type of ferrule should I use?**

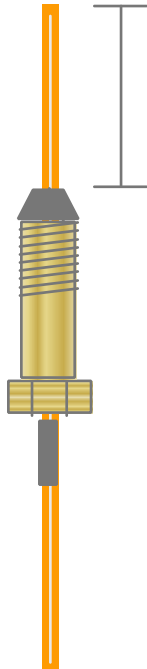
- **Graphite**
- **Graphite/Vespel**
- **Metal Ferrules UltiMetal Plus Flexible Metal Ferrules from Agilent for Capillary Flow Tech. and other GC connections**

# Column Installation

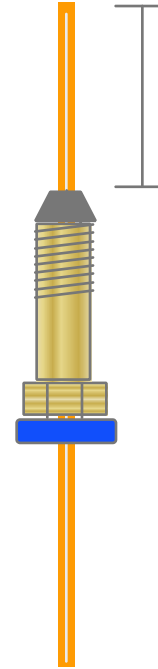
Measuring the right distance

Refer to manufacturers recommendations

**White out**



**Septa**



# Cutting The Column

**Gently scribe through the polyimide coating.  
Do not attempt to cut the glass.**

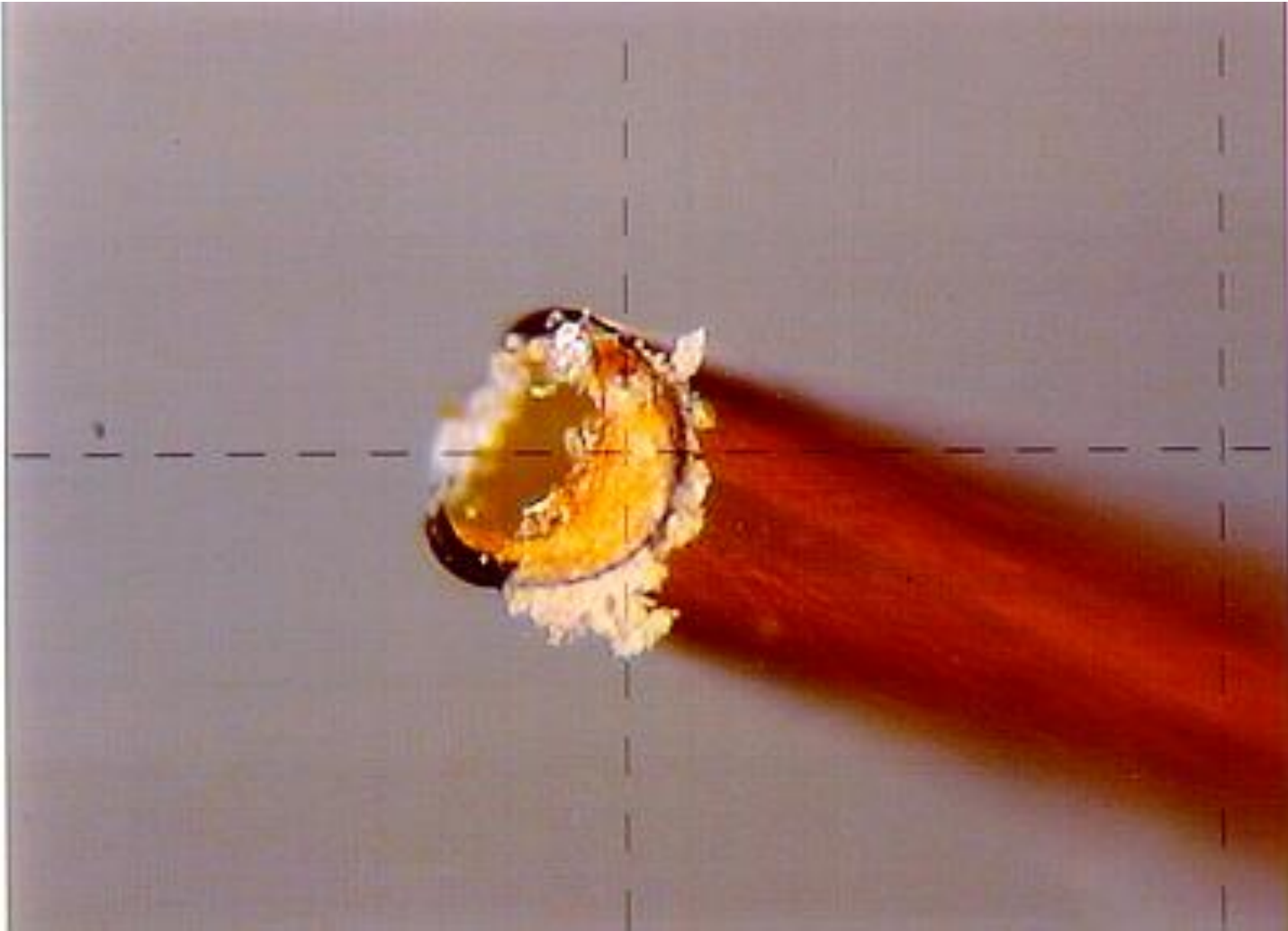
**Recommended tools:**

**Diamond or carbide tipped pencil; or sapphire  
cleaving tool, ceramic wafer  
Ocular or 6x magnification glass**

**Do not use:**

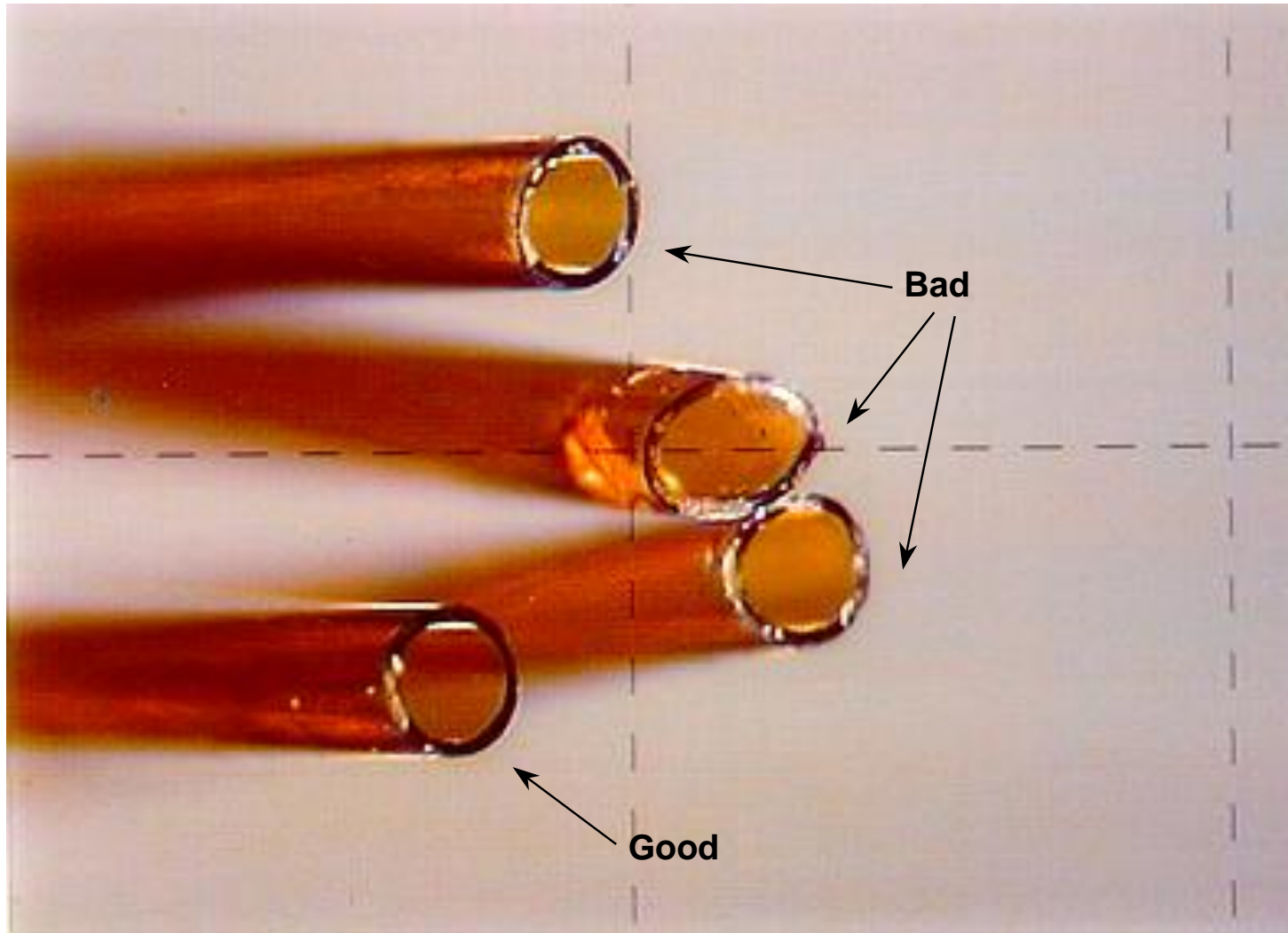
**Scissors, file, etc.**

# Example of a Bad Cut



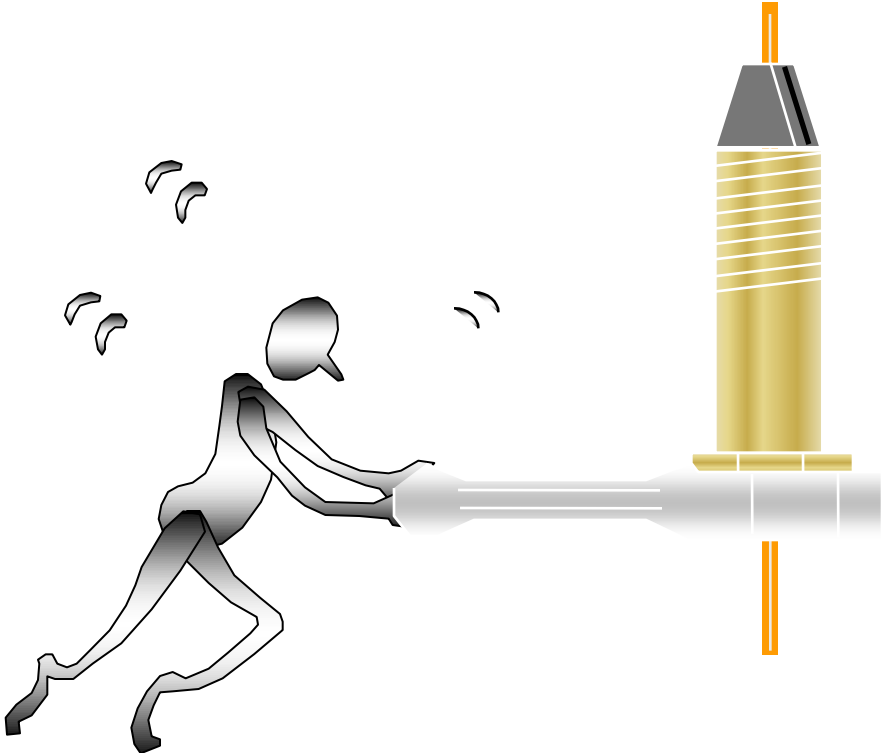


# Examples of Column Cuts

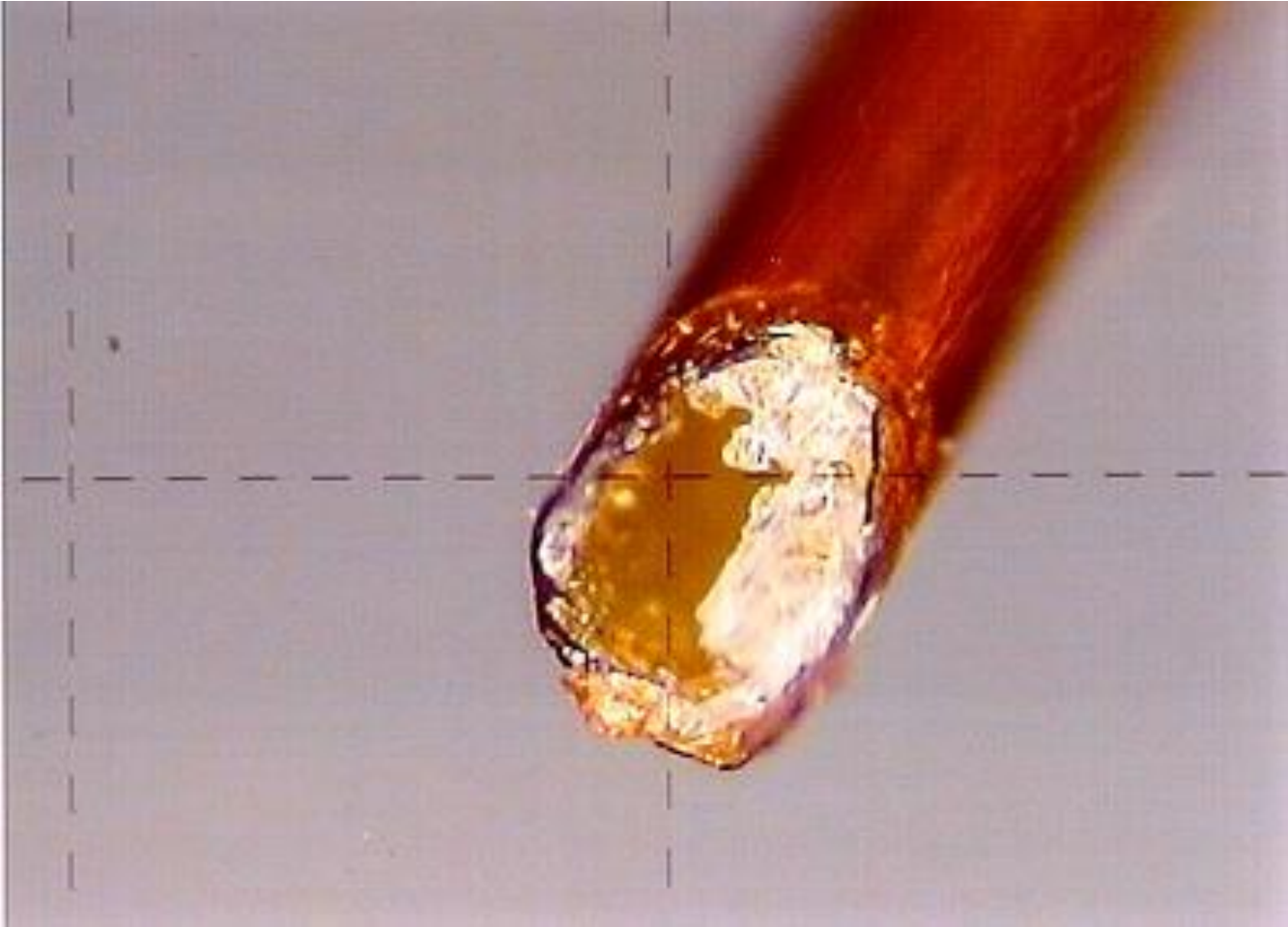


# Column Installation

How tight is tight?



# Over tightened Ferrule



# Column Installation

## Leak Check

**DO NOT USE SNOOP on Gc Columns**

**Electronic leak detector**

**IPA/Water**

**Inject a non-retained peak**

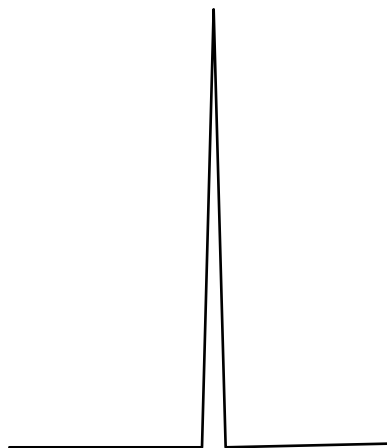
# Leak and Installation Check

Inject a non-retained compound on a DB-1

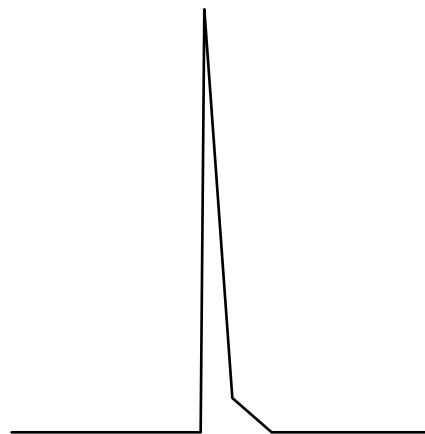
Detector	Compound
FID	Methane or Butane
ECD	MeCl <sub>2</sub> (headspace or diluted)
NPD	CH <sub>3</sub> CN-acetonitrile (headspace or diluted)
TCD	Air
MS	Air or Butane

The peak should be sharp and symmetrical

# Non-Retained Peak Shapes



Good Installation



Improper Installation or  
Injector Leak

**Check for: -Too low of a split ratio**

**-Injector or septum leak**

**-Liner problem:**

**(broken, leaking, misplaced)**

**-Column position in injector and detector**



# Calculating Linear Velocity

Inject a non-retained compound and obtain the retention time:

$$\bar{\mu} = \frac{L}{t_0}$$

$\bar{\mu}$  = Average linear velocity (cm/sec)  
 $L$  = Column length (cm)  
 $t_0$  = Retention time (sec)

$\bar{\mu}$  is *dependent* on column temperature

$\bar{\mu}$  is *independent* of column diameter

He 35-40 cm/sec

H<sub>2</sub> 45-60 cm/sec

# Calculating Flow Rate

Inject a non-retained compound and obtain the retention time:

$$\bar{F} = \frac{\pi r^2 L}{t_0}$$

$\bar{F}$  = Flow rate (mL/min)

$r$  = Column radius (cm)

$L$  = Column length (cm)

$t_0$  = Retention time (min)

$\bar{F}$  is dependent on column temperature

Measuring flow with a flow meter is often inaccurate





# Column Conditioning

**System must be leak free before conditioning column**  
**Follow Manufactures Recommendations**

**Heat the column to the lower of:**

**Isothermal maximum temperature OR**

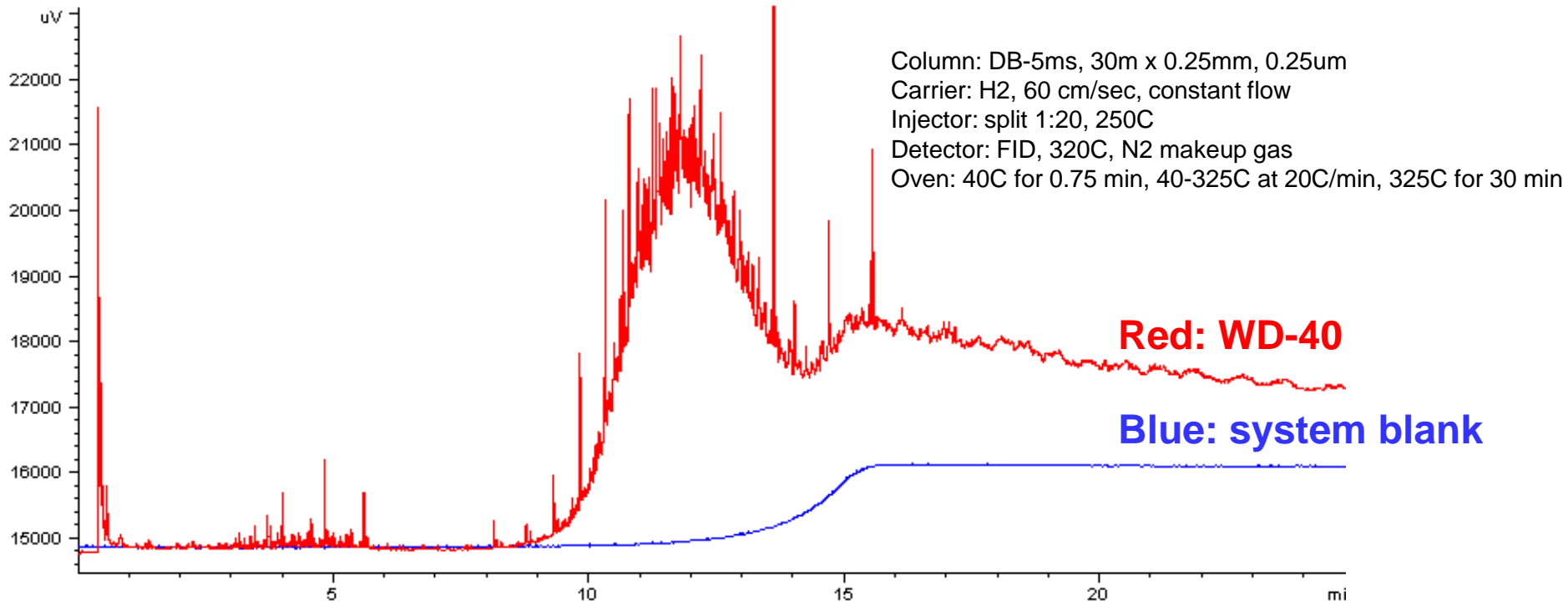
**20° to 30°C above highest operation temperature**

**Temperature programming is not necessary**

**Stop conditioning when the stable baseline is obtained:**

**1 to 2 hours in most cases**

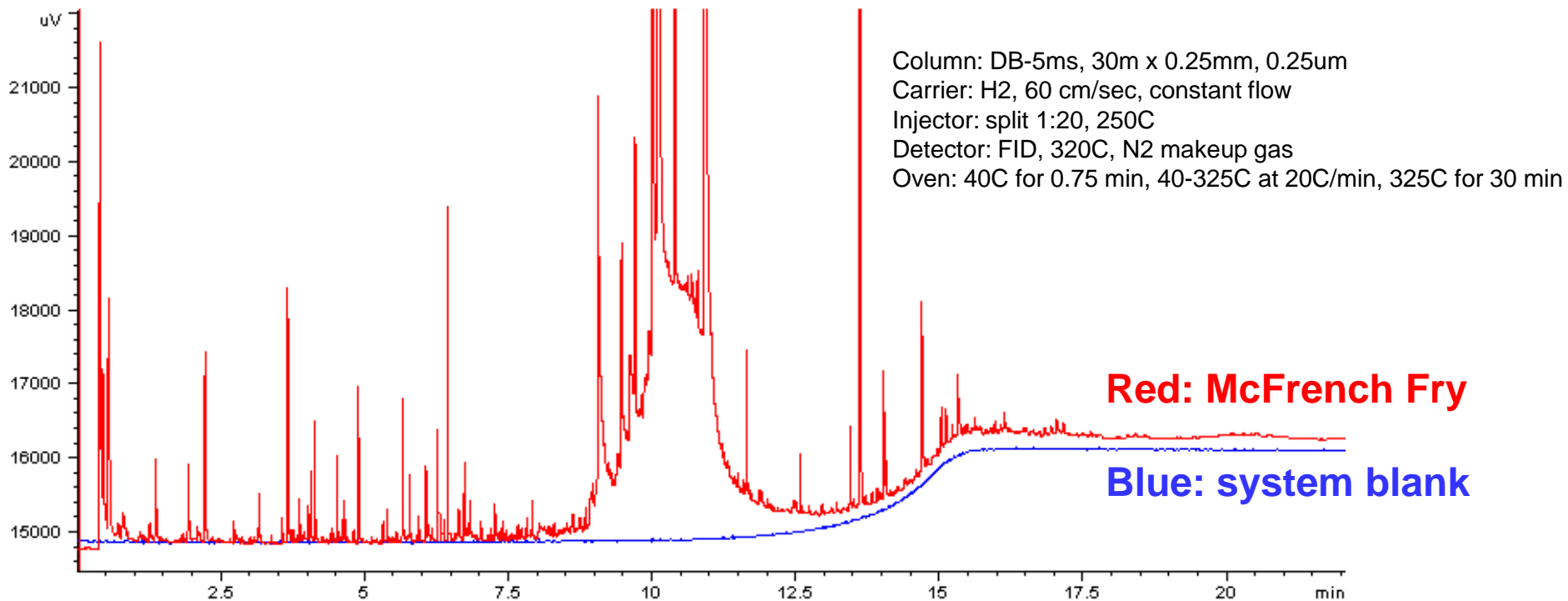
# Contamination of System by Residue on Fingers During Column Installation



## Procedure:

- (1) One very small drop of liquid placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.

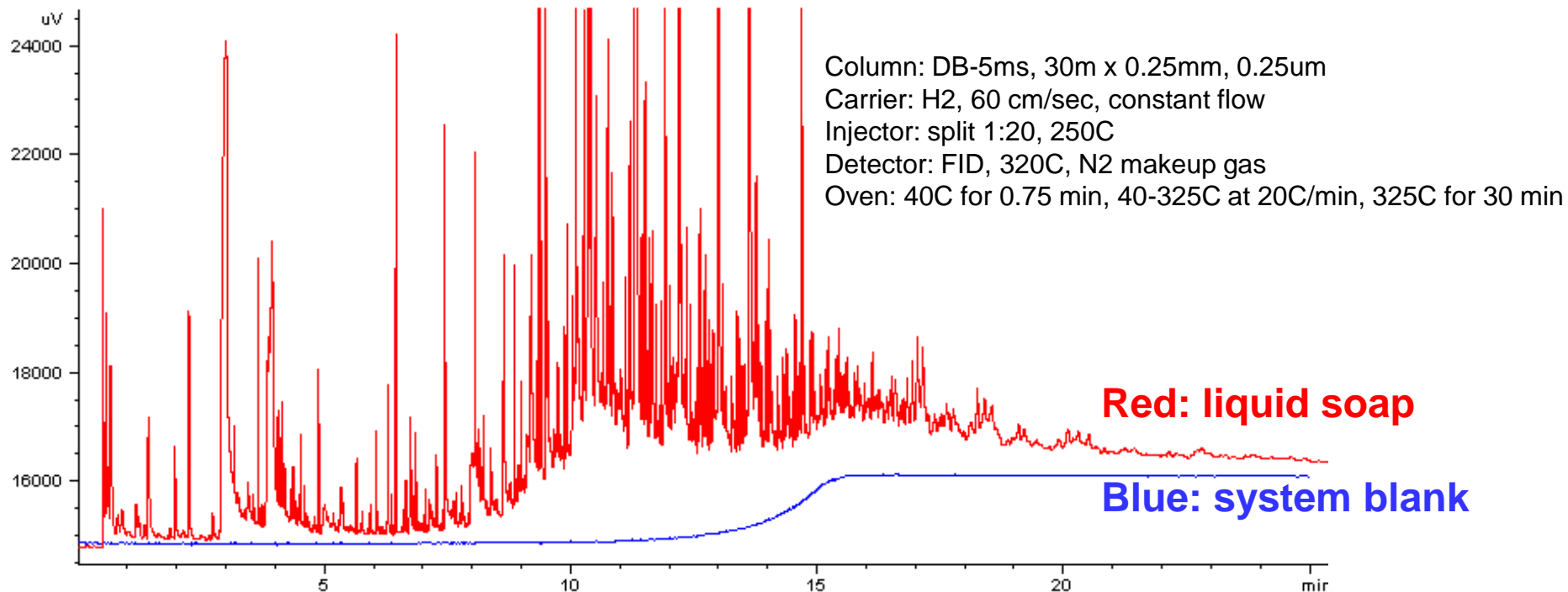
# Contamination from French Fry Grease



## Procedure:

- (1) Held french fry for 5 seconds.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.

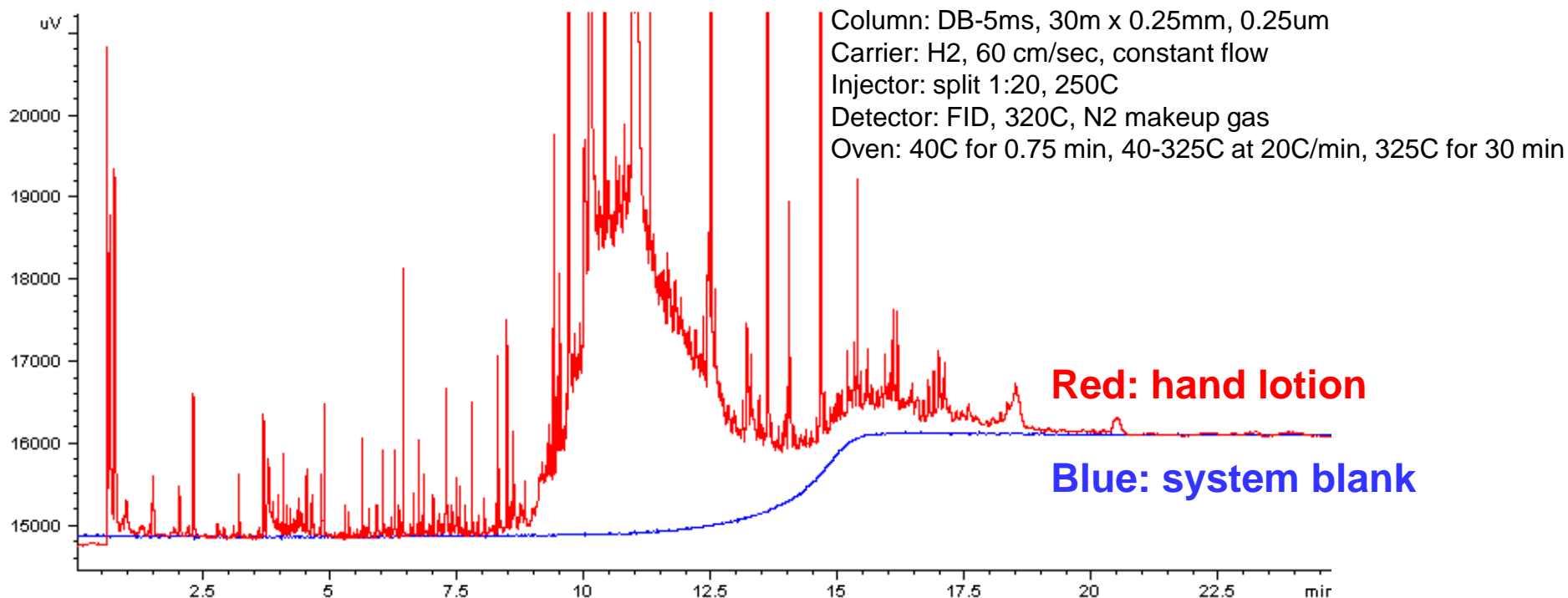
# Contamination from Liquid Soap



## Procedure:

- (1) One very small drop of liquid placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.

# Contamination from Hand Lotion



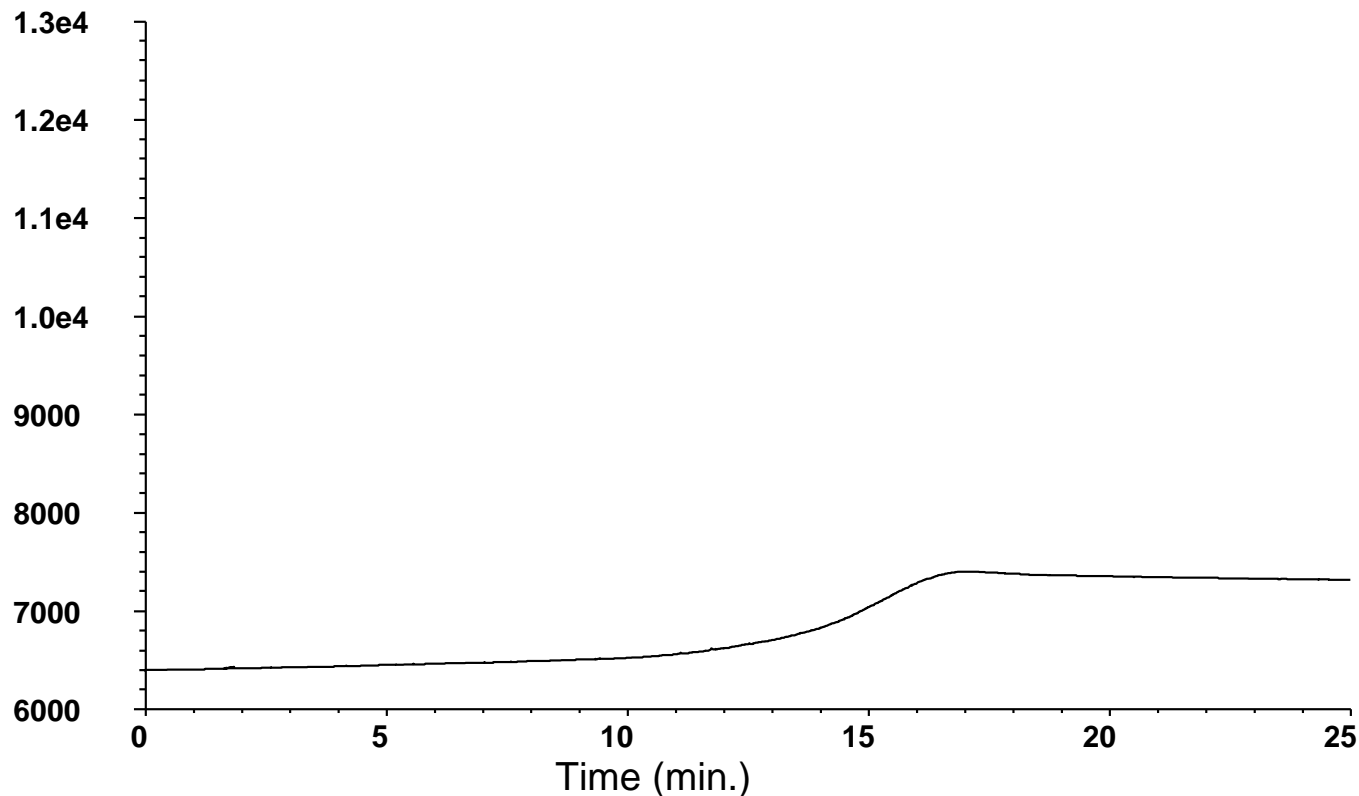
## Procedure:

- (1) One very small drop of liquid placed on one fingertip.
- (2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
- (3) Lightly touched the part of the column sticking up above the ferrule.
- (4) Installed column into injector.
- (5) Set oven temperature to 40C.
- (6) Started oven temperature program as soon as oven reached 40C.



# Generating a Bleed Profile

Temperature program the column without an injection\*



\*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.

# Test Mixes

Used to determine how "good" the column is



# Column Performance Summary

PART NO: 1225032  
 COLUMN I.D. NO.: 3303121  
 LIQUID PHASE: DB-5  
 FILM THICKNESS: 0.25 µm  
 COLUMN DIMENSIONS:  
 30 m X 0.252 mm  
 TEMPERATURE LIMITS:  
 -60° C TO 325° C (350° C PROGRAM)

THEORETICAL PLATES/METER:	MIN SPEC	ACTUAL
PENTADECANE	3900	4389

COATING EFFICIENCY:	MIN SPEC	MAX SPEC	ACTUAL
PENTADECANE	90.0	95.5	

RETENTION INDEX:	MIN SPEC	MAX SPEC	ACTUAL
1-UNDECANOL	1371.04	1372.04	1371.43
ACENAPHTHYLENE	1459.34	1460.34	1459.53

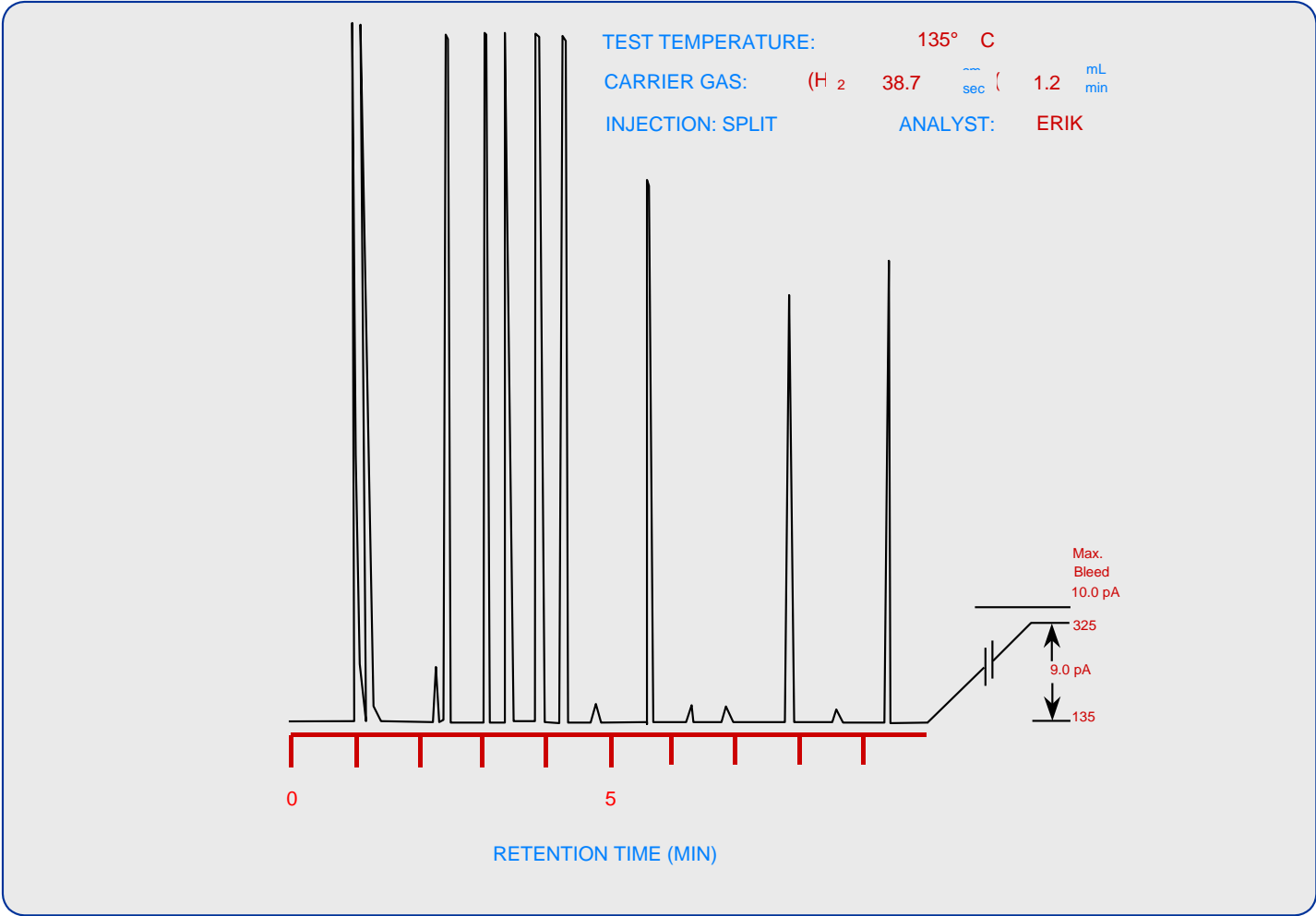
  

PEAK HEIGHT RATIO:	
4-CHLOROPHENOL/ METHYL NONANOATE	0.83
4-PROPYLANILINE/ METHYL NONANOATE	1.14

COMPOUND IDENTIFICATION	RETENTION TIME (R)	PARTITION RATIO (k)	PEAK WIDTH (W 1/2)
1,6-HEXANEDIOL	2.51	0.9	0.019
4-CHLOROPHENOL	2.95	1.3	0.022
METHYL NONANOATE	3.21	1.5	0.022
4-PROPYLANILINE	3.81	1.9	0.026
TRIDECANE	4.20	2.2	0.027
1-UNDECANOL	5.52	3.3	0.036
ACENAPHTHYLENE	8.00	5.2	0.053
PENTADECANE	9.58	6.4	0.062
Approximately 5-10 ng on column			
o	1.29		



# Chromatographic Performance



# Test Mixture Components

## Compounds

Hydrocarbons

FAME's, PAH's

Alcohols

Acids

Bases

## Purpose

Efficiency

Retention

Retention

Activity

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**



# An Ounce of Prevention.....

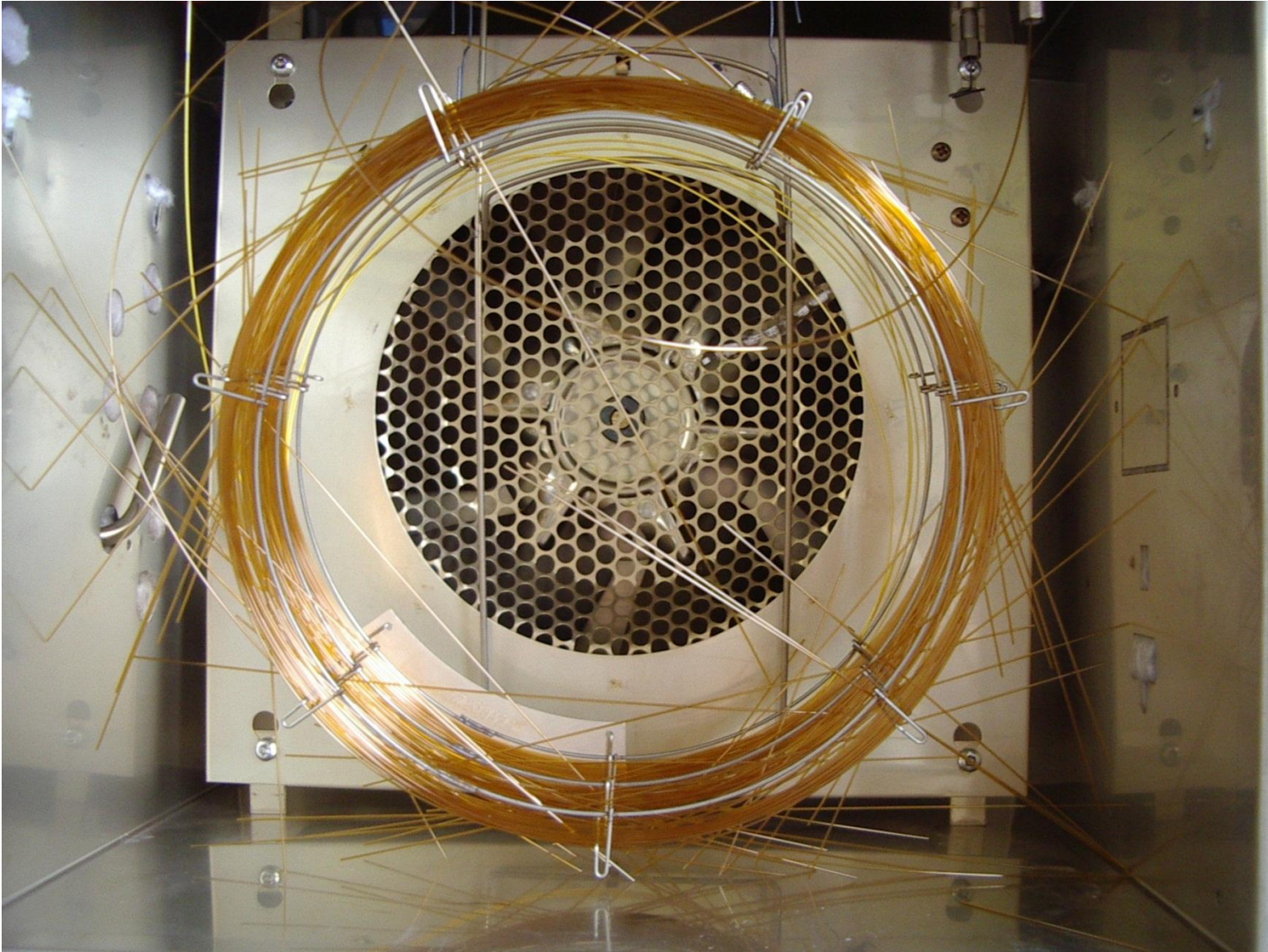
# Common Causes of Column Performance Degradation

- **Physical damage to the polyimide coating**
- **Thermal damage**
- **Oxidation (O<sub>2</sub> damage)**
- **Chemical damage by samples DCM and H<sub>2</sub>**
- **Contamination**

# Physical Damage to The Polyimide Coating

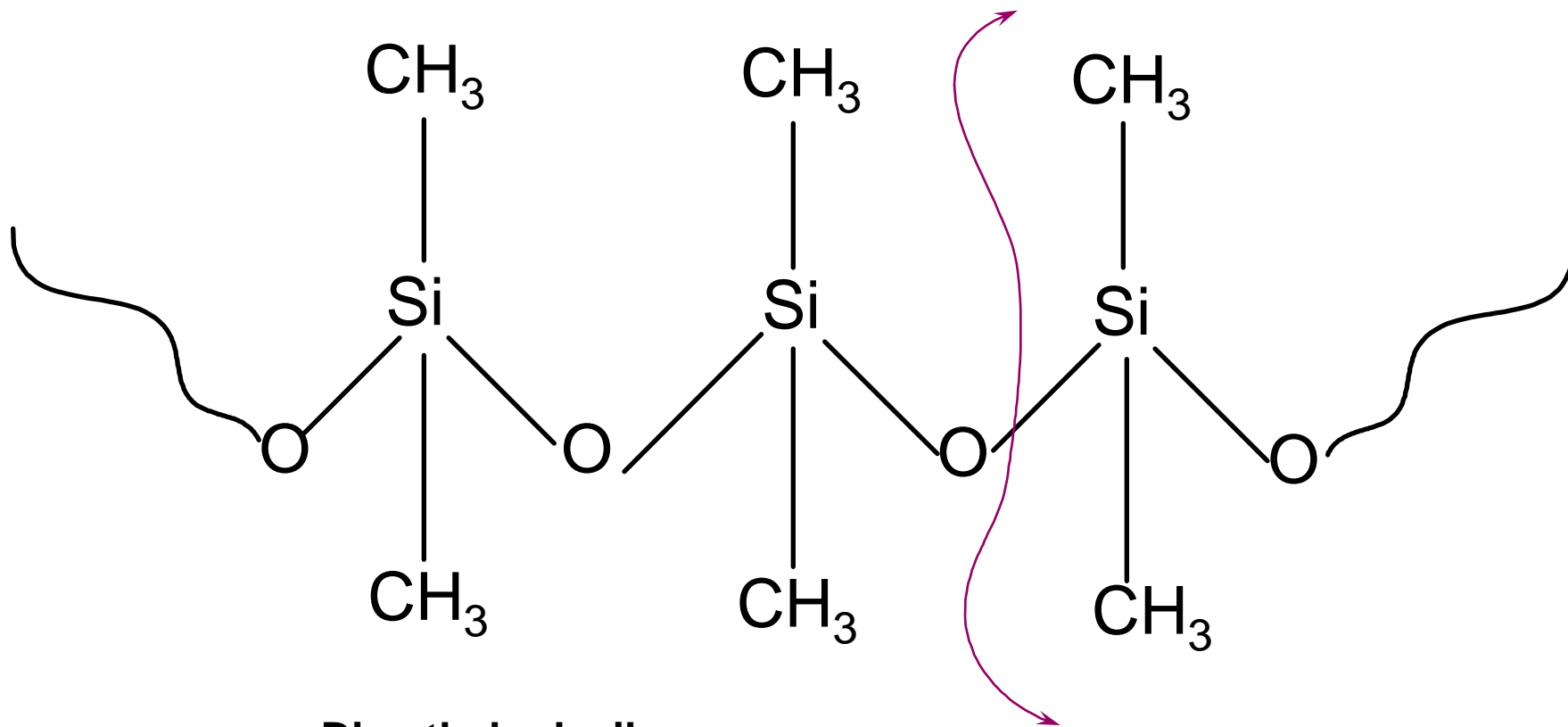
- **Smaller diameter tubing is more flexible than larger diameter tubing.**
- **Avoid scratches and abrasions**
- **Immediate breakage does not always occur upon physical damage**

# NOT what you want your column to look like!



# Thermal Damage

Degradation of the stationary phase is increased at higher temperatures. Breakage along the polymer backbone.



Dimethylpolysiloxane



# Thermal Damage

## What To Do If It Happens

- **Disconnect column from detector**
- **“Bake out” overnight at isothermal limit**
- **Remove 10-15 cm from column end**
- **On most columns one wrap = 1/2 meter**

# Thermal Damage

- **Rapid degradation of the stationary phase caused by excessively high temperatures**

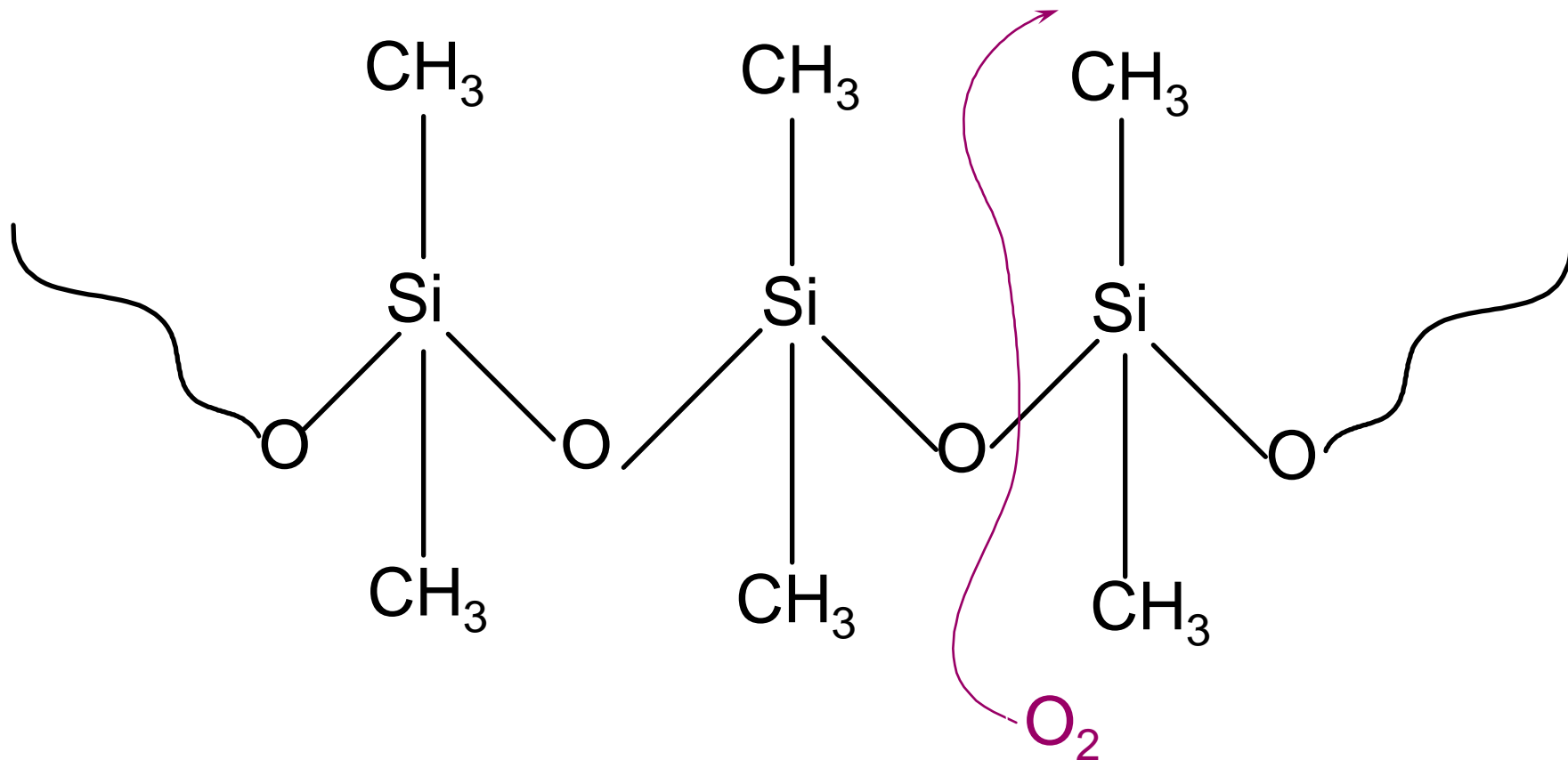
**Isothermal limit = Indefinite time**

**Programmed limit = 5-10 minutes 325/350**

- **Temporary "column failure" below lower temperature**  
**Polar or Specialty columns have lower oven temps.**

# Oxidation (O<sub>2</sub> Damage)

Oxygen in the carrier gas rapidly degrades the stationary phase. The damage is accelerated at higher temperatures. Damage along the polymer backbone is irreversible.



Dimethylpolysiloxane

# Oxygen Damage

- **Causes rapid damage to the column**
- **Usually results in irreversible column damage**

# How to Prevent Column Damage by Oxygen

- High quality carrier gas (4 nine's or greater)
- Leak free injector and carrier lines
  - Change septa
  - Maintain gas regulator fittings
- Appropriate impurity traps
- Use Gas Clean Filters from Agilent

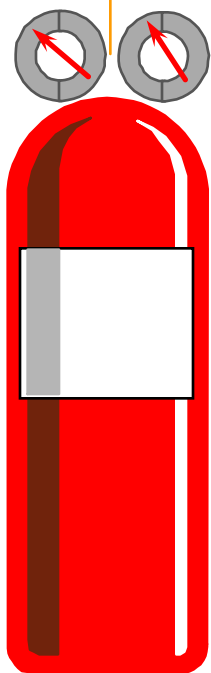


# Configurations for Carrier Gas Purifiers

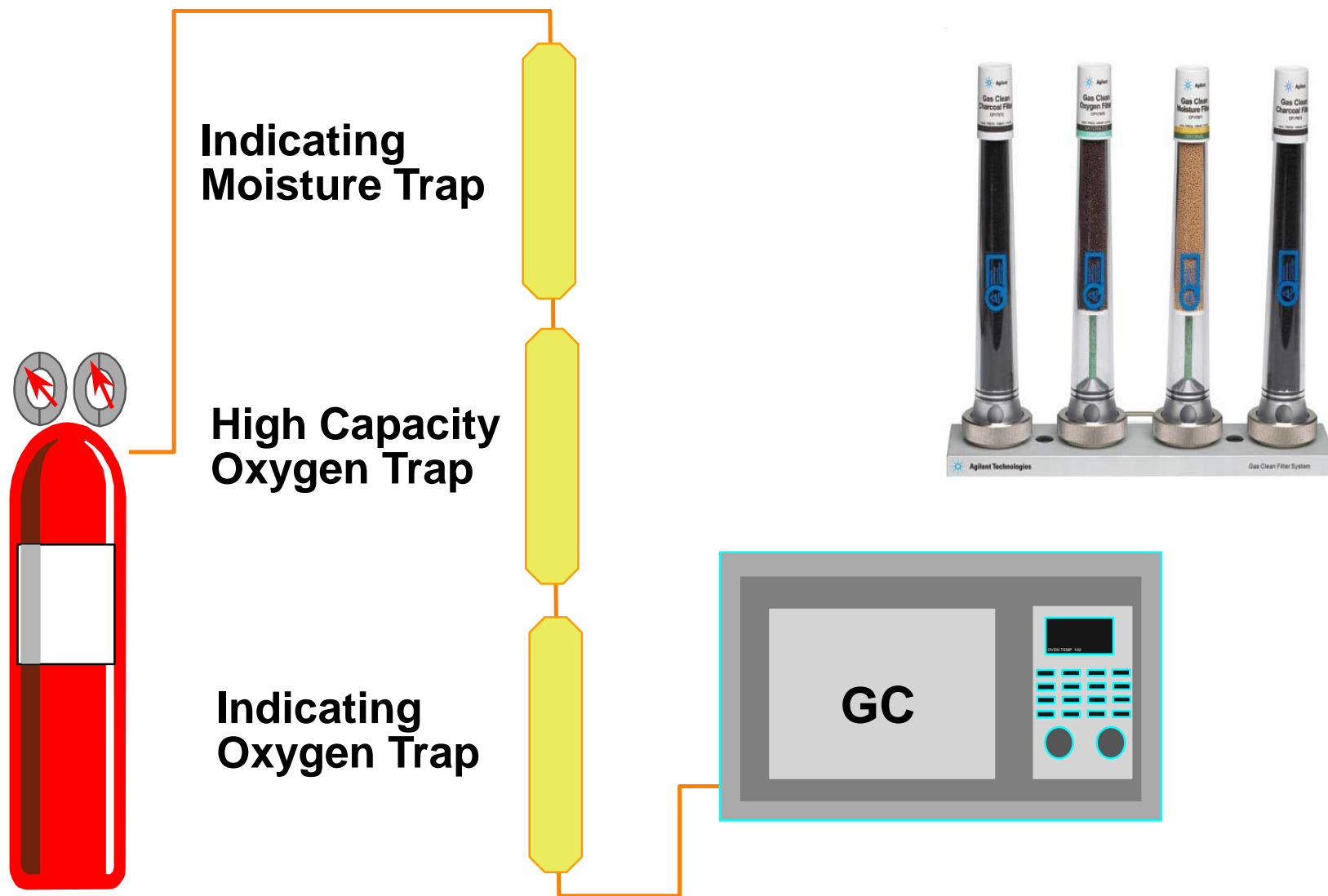
Indicating  
Moisture Trap

High Capacity  
Oxygen Trap

Indicating  
Oxygen Trap



# Configurations for Carrier Gas Purifiers



# Chemical Damage

**Bonded and cross-linked columns have excellent chemical resistance except for inorganic acids and bases**

HCl

NH<sub>3</sub>

KOH

NaOH

H<sub>2</sub>SO<sub>4</sub>

H<sub>3</sub>PO<sub>4</sub>

HF

etc.

**Chemical damage will be evident by excessive bleed, lack of inertness or loss of resolution/retention**



# Chemical Damage

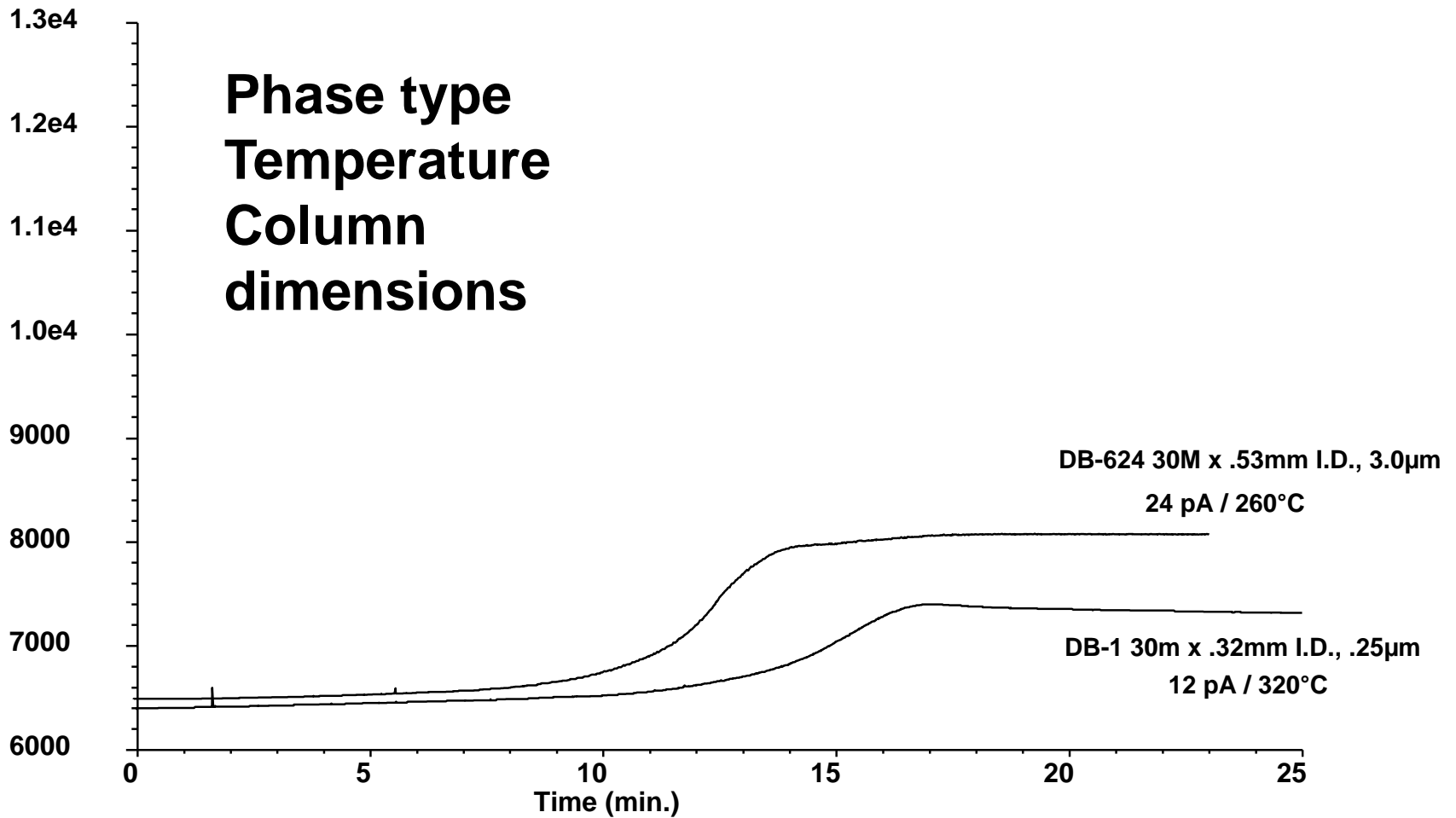
## What To Do If It Happens

- Remove 1/2 - 1 meter from the front of the columns
- Removing one wrap is = to 1/2 meter on a 7" column
- Severe cases may require removal of up to 5 meters

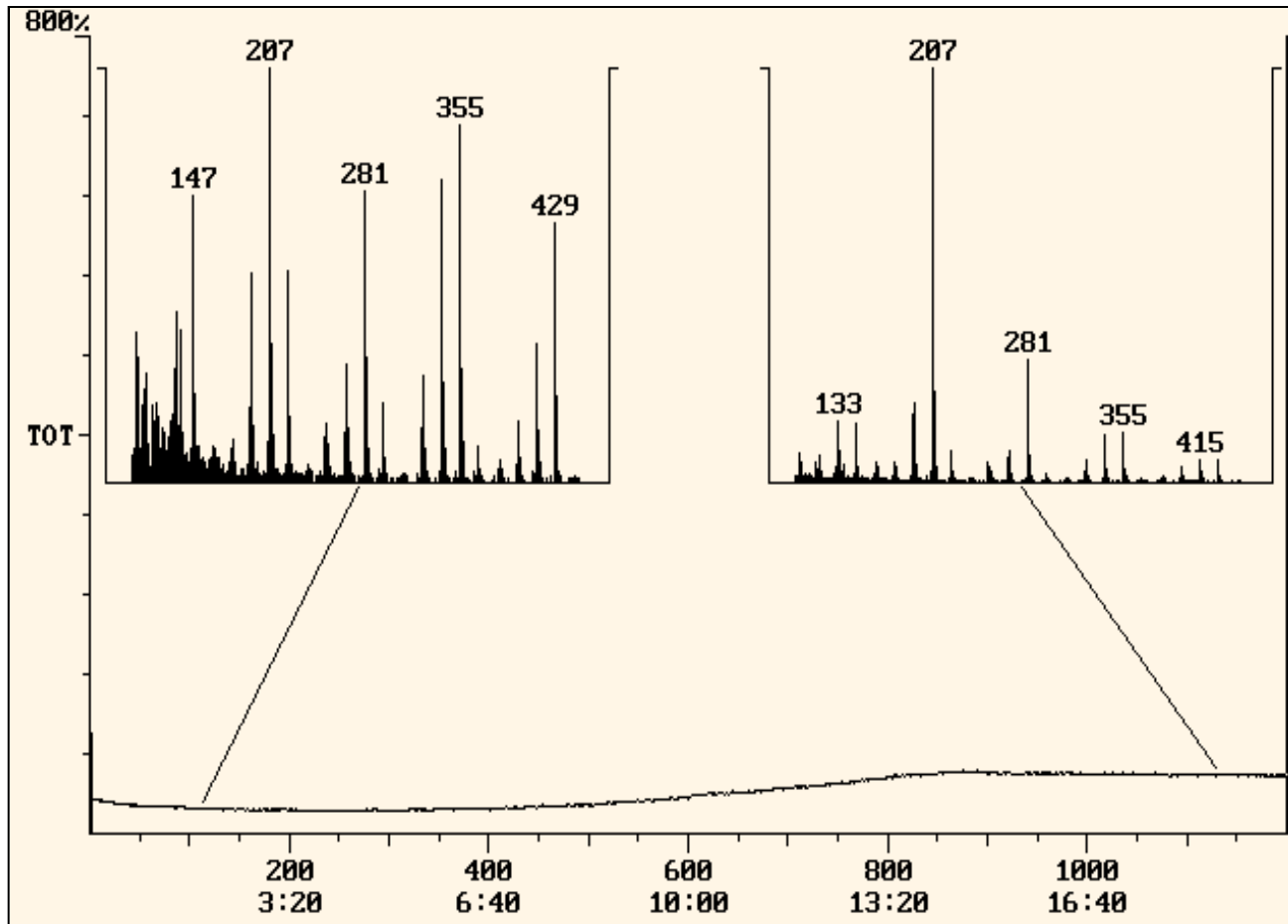
# What is Normal Column Bleed

Normal background signal generated by the elution of normal degradation products of the column stationary phase

# Column Bleed is Influenced by:



# Mass Spectrum of Phenylmethylpolysiloxane Column Bleed (Normal Background)



Mass spectral library search is not always accurate

# What is a Bleed Problem?

## IT IS:

An abnormal elevated baseline at **High Temperature**

## IT IS NOT:

A high baseline at **Low Temperature**

Wandering or drifting baseline at any temperature

Discrete peaks

# Column Contamination

- **Fouling of GC and column by contaminants**
- **Mimics nearly every chromatographic problems**

# Symptoms of Contamination

- **Poor peak shape**
- **Loss of separation (resolution)**
- **Changes in retention**
- **Reduced peak size**
- **Baseline disturbances (semi-volatiles only)**



# Typical Samples That Contain a Large Amount of Residues

**Biological (Blood, Urine, Tissue, Plants)**

**Soils**

**Foods**

**Waste Water**

**Sludges**

**All samples contain residues!! (even standards!)**





# Other Sources of Contamination

- **Septum and ferrule particles**
- **Gas and trap impurities**
- **Unknown sources (vials, syringes, etc.)**

## Non-Volatile Residues

Any portion of the sample that does not elute from the column or remains in the injector.

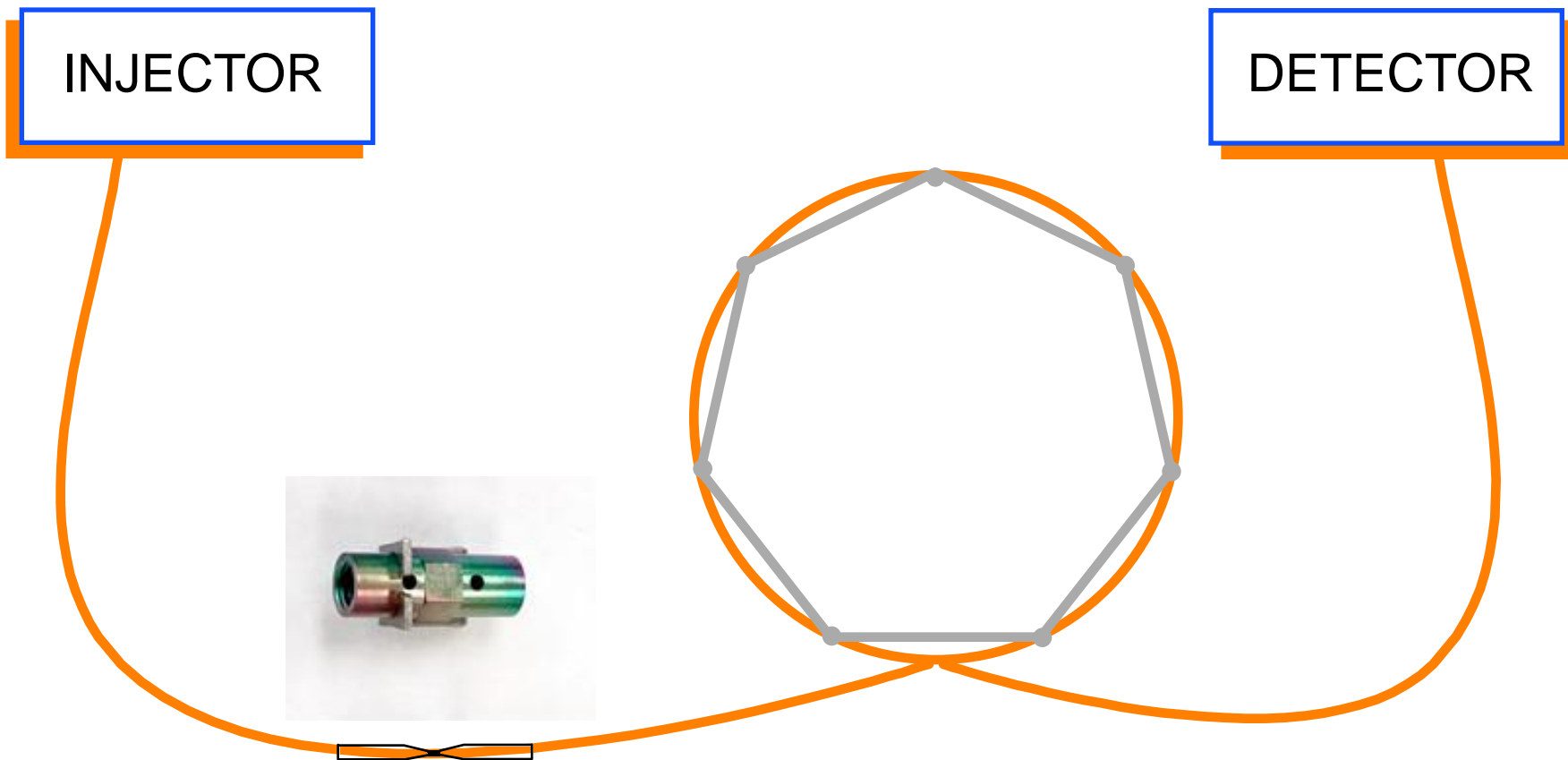
## Semi-Volatile Residues

Any portion of the sample that elutes from the column after the current chromatographic run.

# Methods to Minimize Non-Volatile Residue Problems

- **Sample cleanup**
- **Packed injection port liners**
- **Guard columns**

# Guard Column or Retention Gap



**The guard column is 3 - 5 meters of deactivated fused silica tubing with the same diameter as the analytical column. It is connected with a zero dead volume union.**

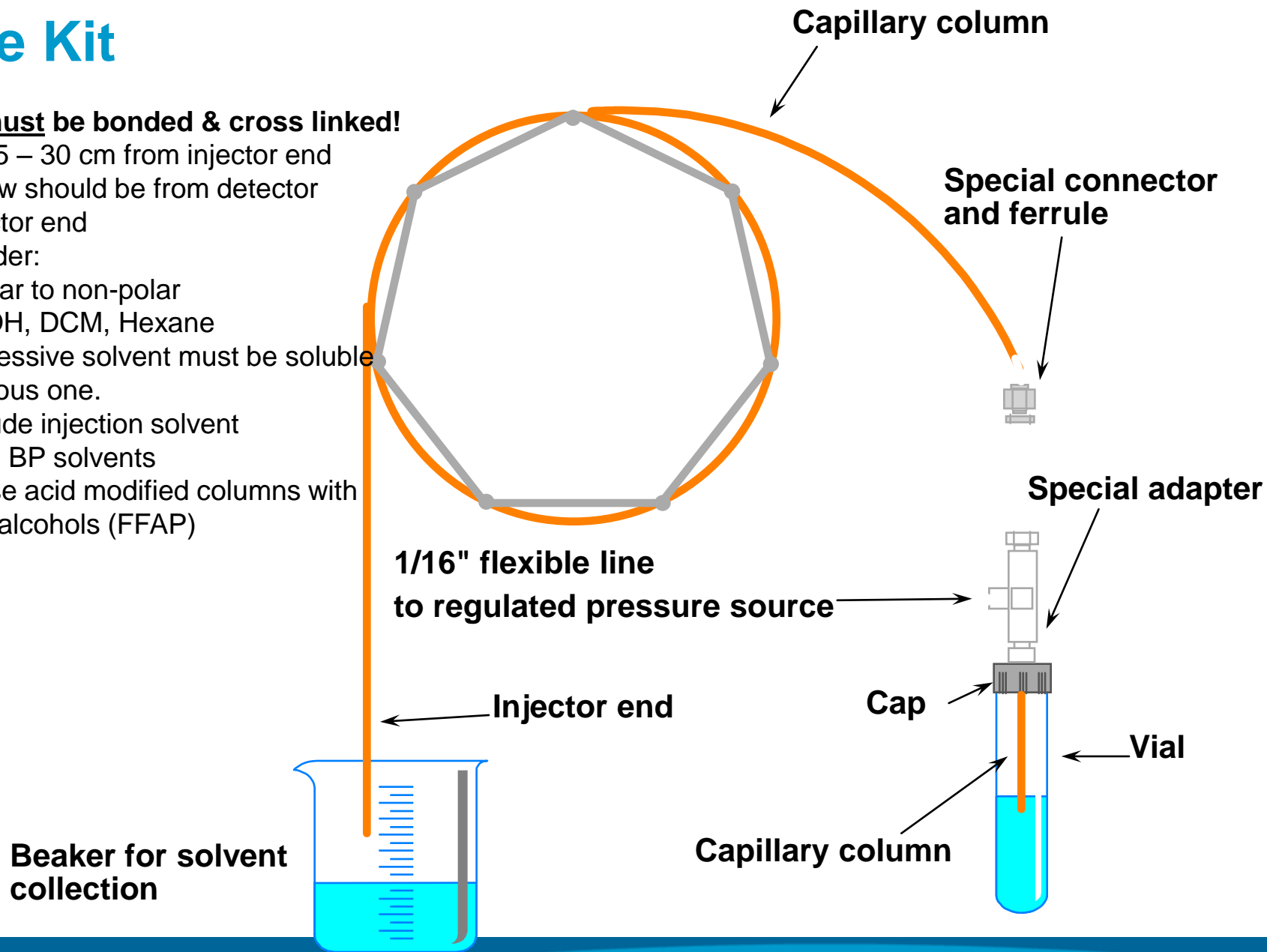
# Non-Volatile Contamination

## What To Do If It Happens

- Do not “bake out” the column
- Front End Maintenance
  - clean or change the injector liner
  - clean the injector
  - cut off 1/2 -1 meter of the front of the column
- Turn the column around
- Solvent rinse the column
- Cut the column in half

# Rinse Kit

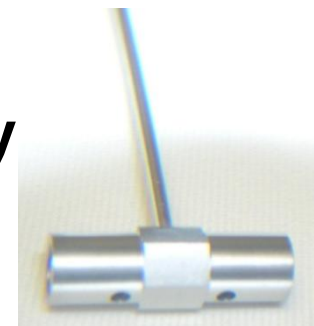
- > **Column must be bonded & cross linked!**
- > Remove 15 – 30 cm from injector end
- > Solvent flow should be from detector end to injector end
- > Solvent order:
  - from polar to non-polar
  - i.e. MeOH, DCM, Hexane
- > Each successive solvent must be soluble in the previous one.
- > Try to include injection solvent
- > Avoid High BP solvents
- > Do not rinse acid modified columns with water or alcohols (FFAP)



# Semi-Volatile Contamination

## What To Do If It Happens

- **“Bake out” the column**
  - **Limit to 1-2 hours**
  - **Longer times may polymerize some contamination and reduces column life**
- **Solvent rinse the column**
- **Consider utilizing Back Flush Technology**



# Column Storage

- **Place septa over the ends**
- **Return to column box**



## Always Remember to:

- **Start with a good installation**
- **Maintain an oxygen free system**
- **Avoid physical, thermal, and chemical damage**
- **Take steps to prevent contamination**

**Thank you and remember to  
call Technical Support!!**

**1-800-227-9770 Select Opt.  
#3, #3 then #1**

**1-866-422-5571 (FAX)**

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

