Installation, Care and Maintenance of Capillary Gas Chromatography Columns

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Presenter – Mark Sinnott

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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

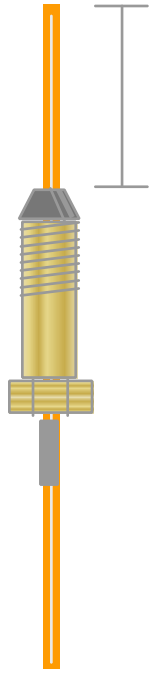
What type of ferrule should I use?

- **Graphite**
- **Graphite/Vespel**

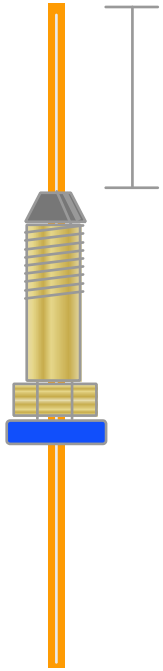
Column Installation

Measuring the right distance

White out



Septa



Cutting The Column

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

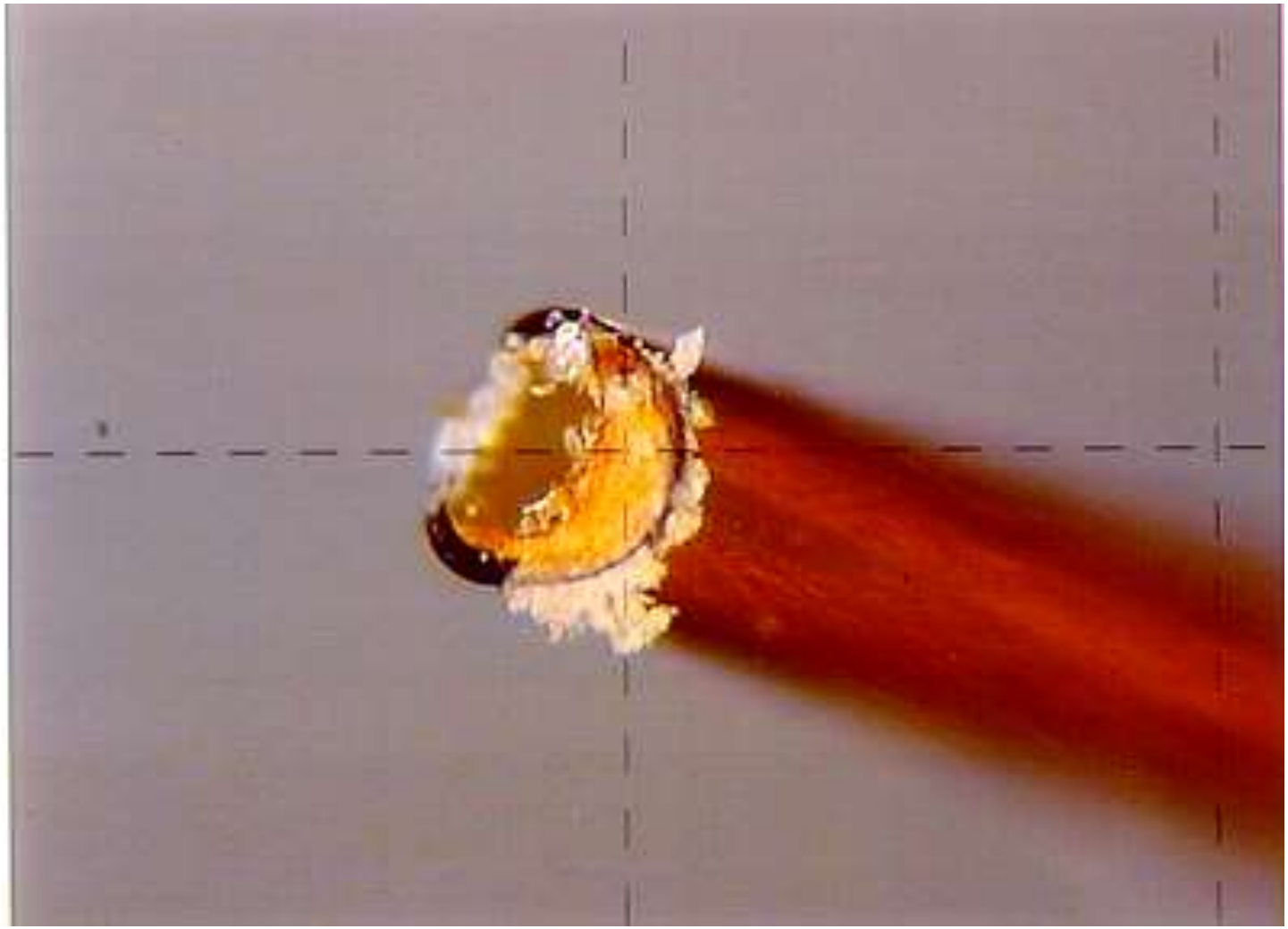
Recommended tools:

Diamond or carbide tipped pencil; or sapphire cleaving tool, ceramic wafer
Ocular

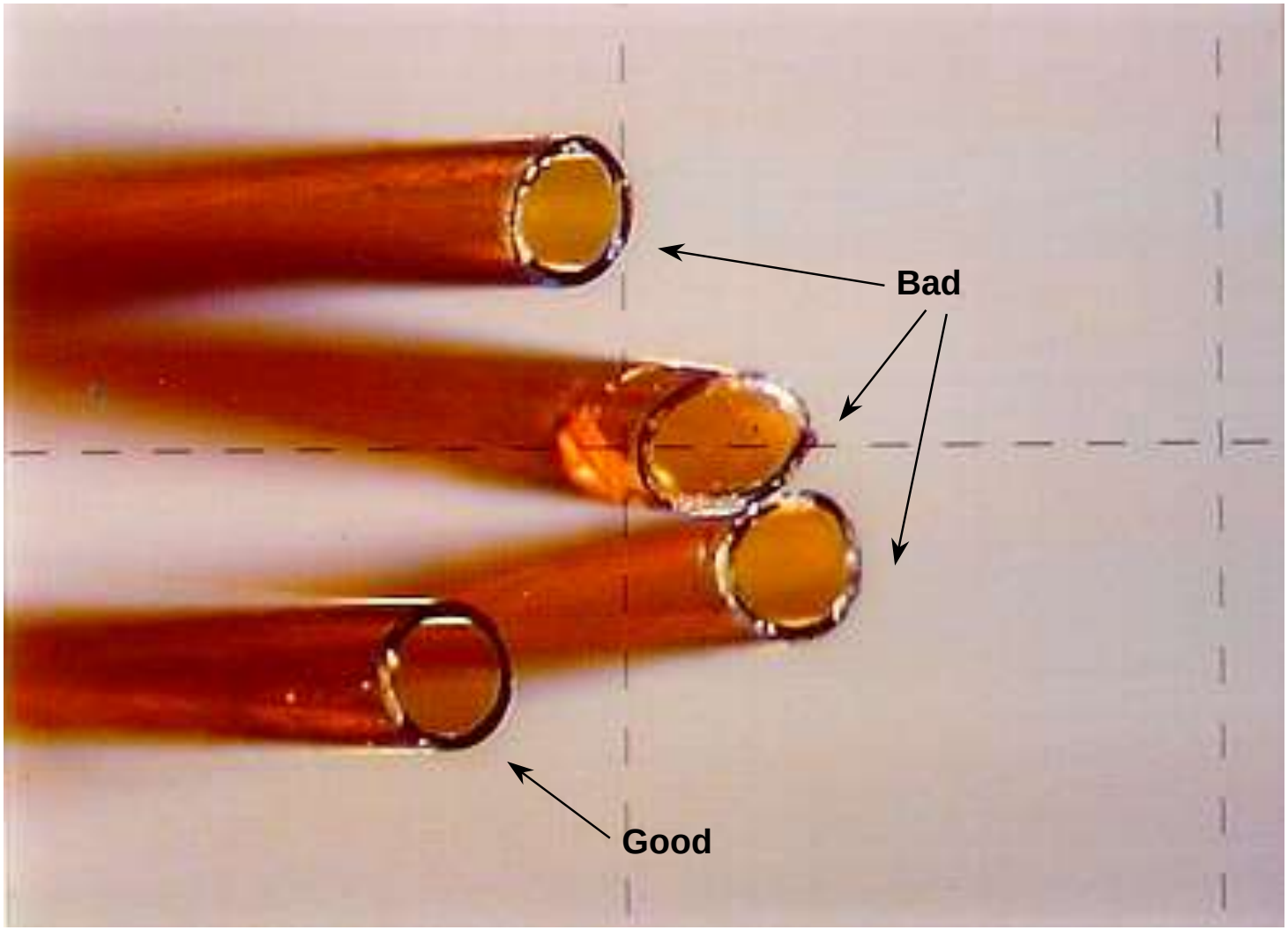
Do not use:

Scissors, file, etc.

Example of a Bad Cut

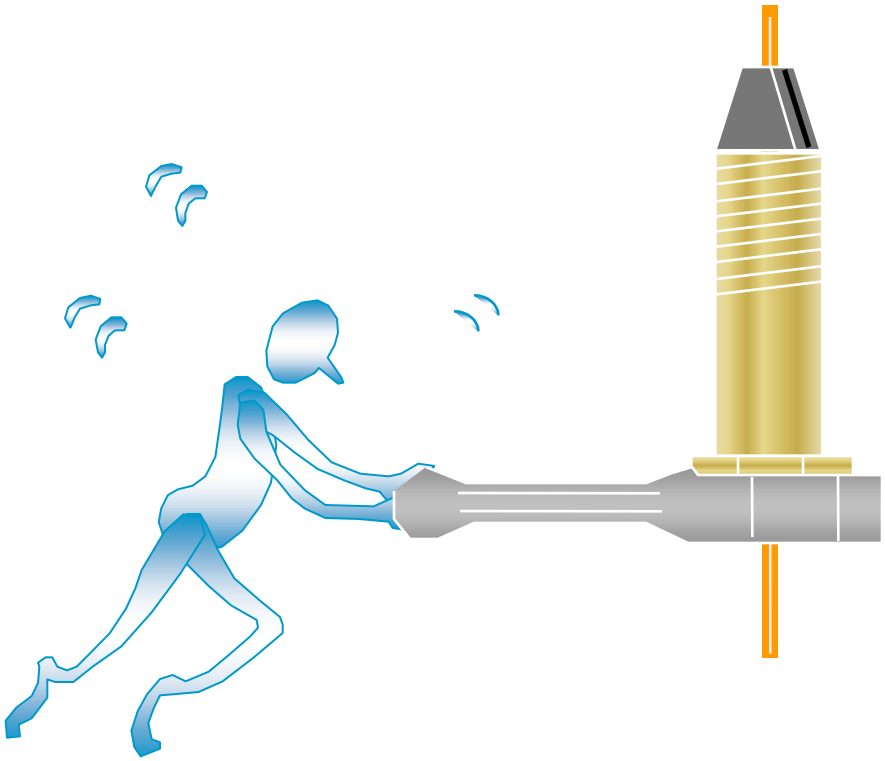


Examples of Column Cuts

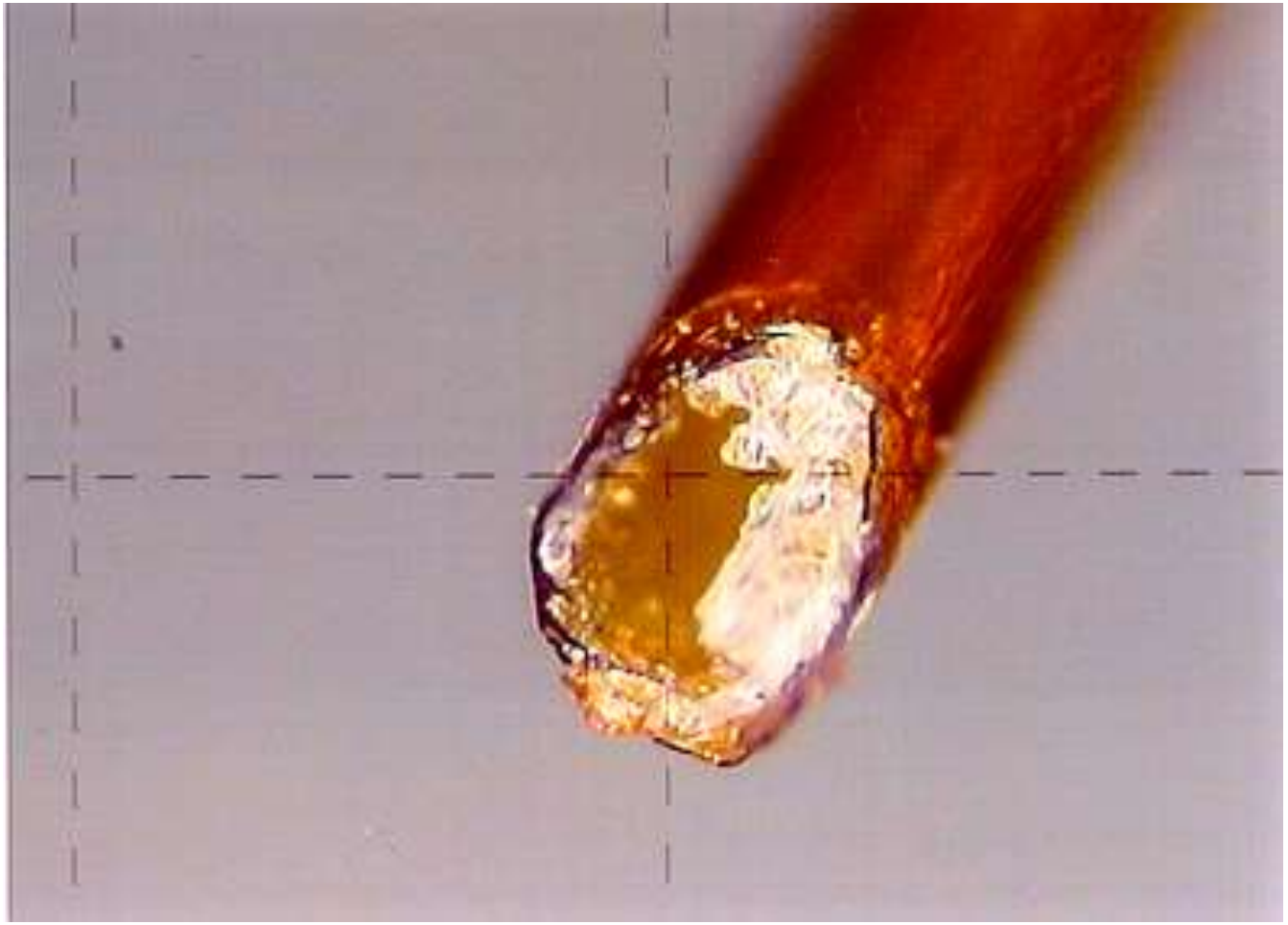


Column Installation

How tight is tight?



Overtightened Ferrule



Column Installation

Leak Check

DO NOT USE SNOOP

Electronic leak detector
IPA/Water
Inject a non-retained peak

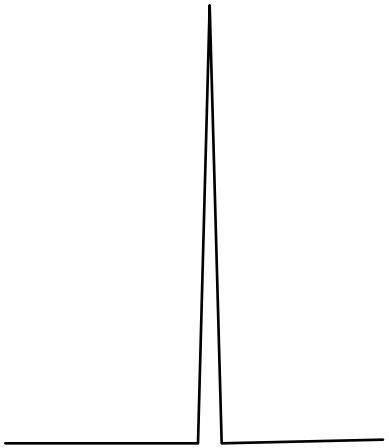
Leak and Installation Check

Inject a non-retained compound vs DB-1

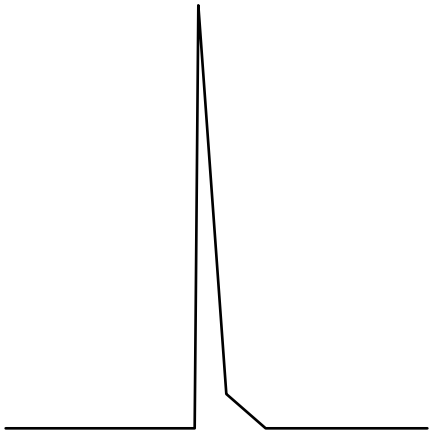
Detector	Compound
FID	Methane or Butane
ECD	$MeCl_2$ (headspace or diluted)
NPD	CH_3CN-acetonitrile (headspace or diluted)
TCD	Air
MS	Air or Butane

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:

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

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

He 20-40 cm/sec
H_2 35-55 cm/sec

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

Calculating Flow Rate

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

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

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

r = Column radius (cm)

L = Column length (cm)

t_o = Retention time (sec)

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

Measuring flow with a flow meter is often inaccurate

Column Conditioning

System must be leak free before conditioning column

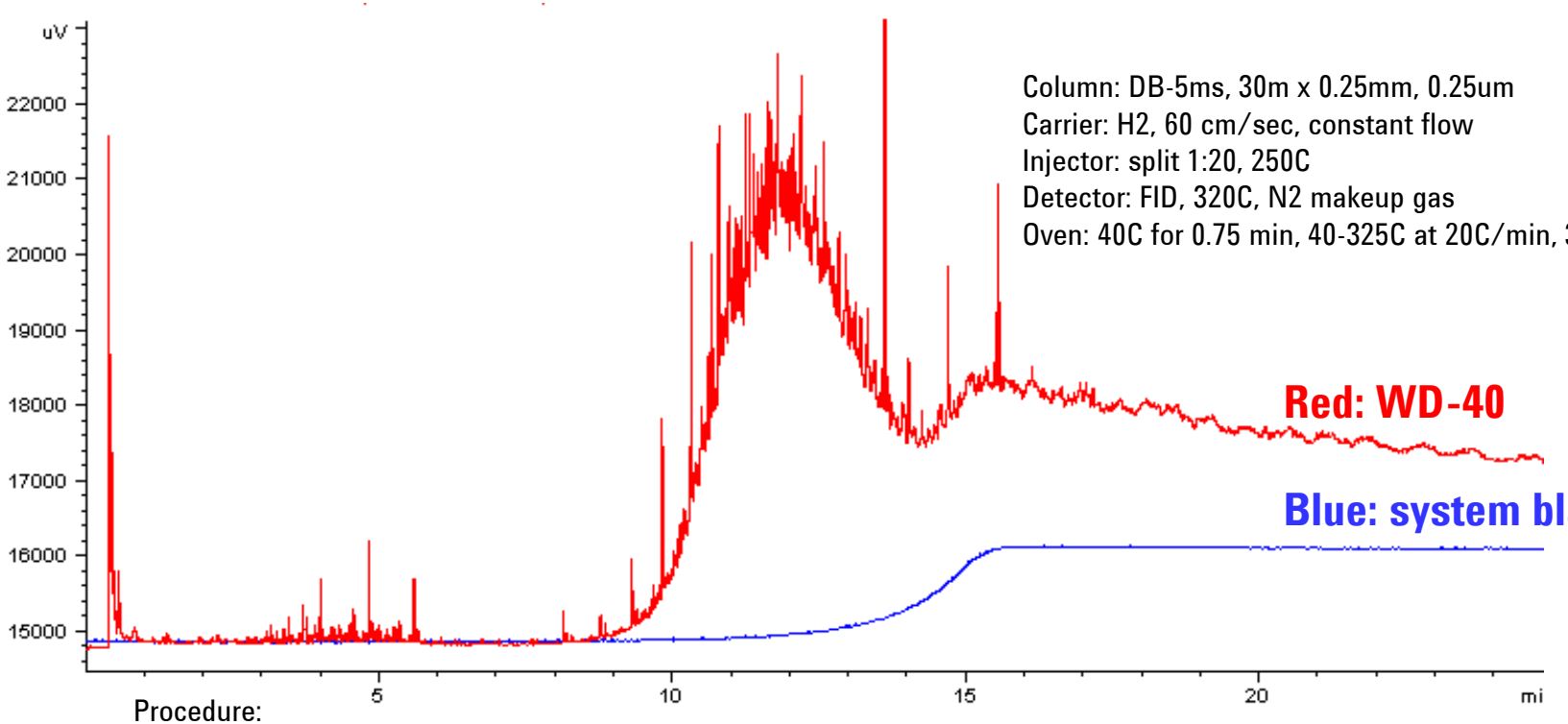
Heat the column to the lower of:

- **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



Column: DB-5ms, 30m x 0.25mm, 0.25um
Carrier: H2, 60 cm/sec, constant flow
Injector: split 1:20, 250C
Detector: FID, 320C, N2 makeup gas
Oven: 40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

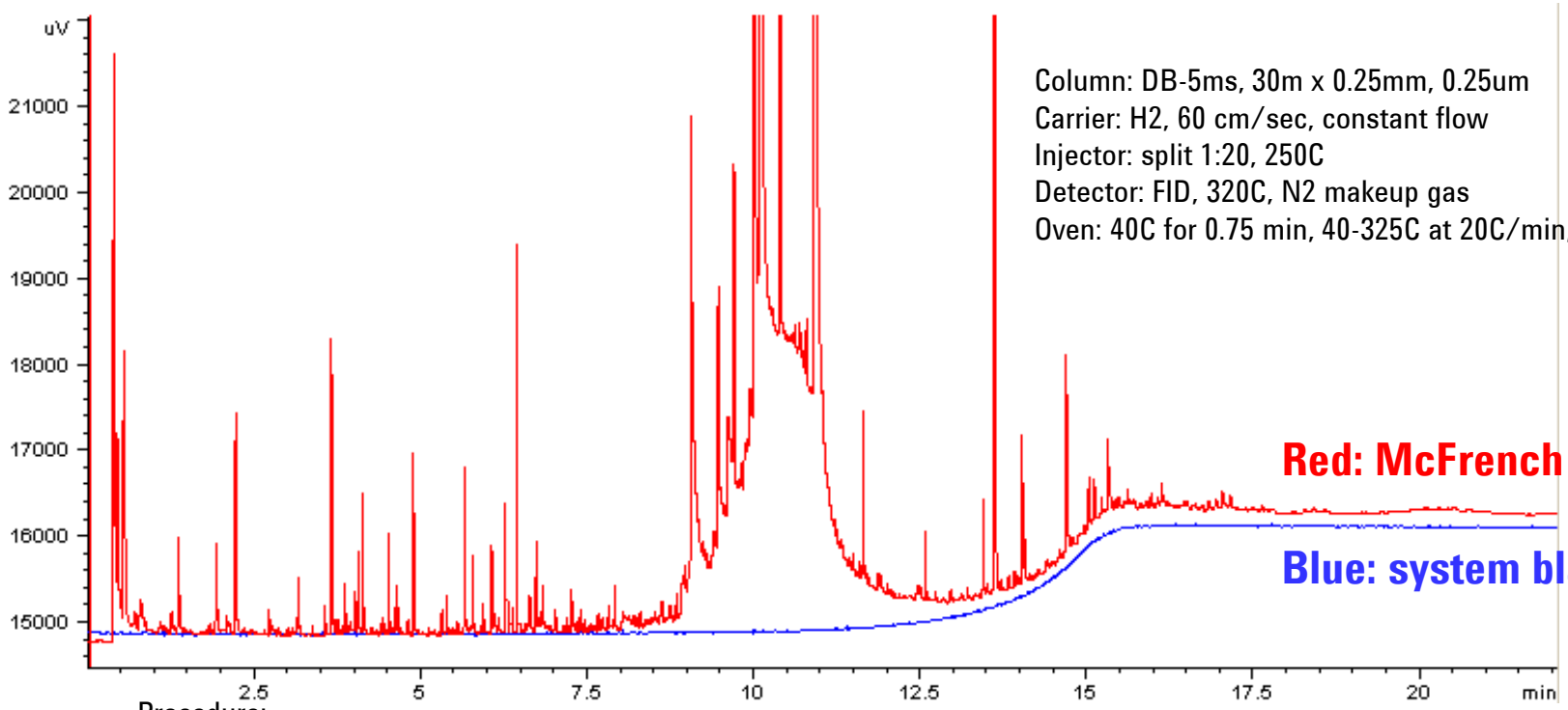
Red: WD-40

Blue: system blank

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



Column: DB-5ms, 30m x 0.25mm, 0.25um
Carrier: H2, 60 cm/sec, constant flow
Injector: split 1:20, 250C
Detector: FID, 320C, N2 makeup gas
Oven: 40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min

Red: McFrench Fry

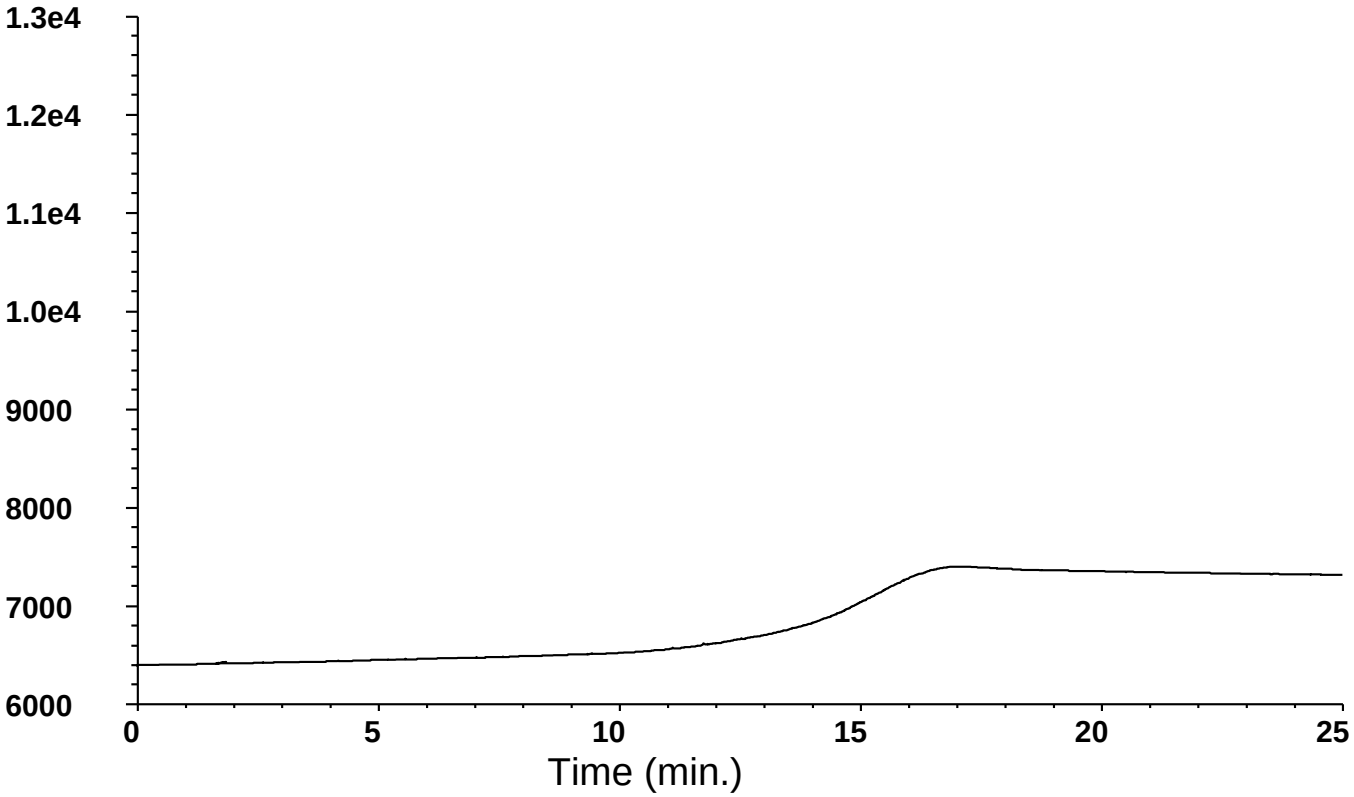
Blue: system blank

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.

Generating a Bleed Profile

Temperature program the column without an injection*



***DB-1 30m x .32mm I.D., .25µm**
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

Catalog: 1225032

Serial: US5345175H

Stationary Phase: DB-5

Description: 30m x 0.252mm x 0.25µm

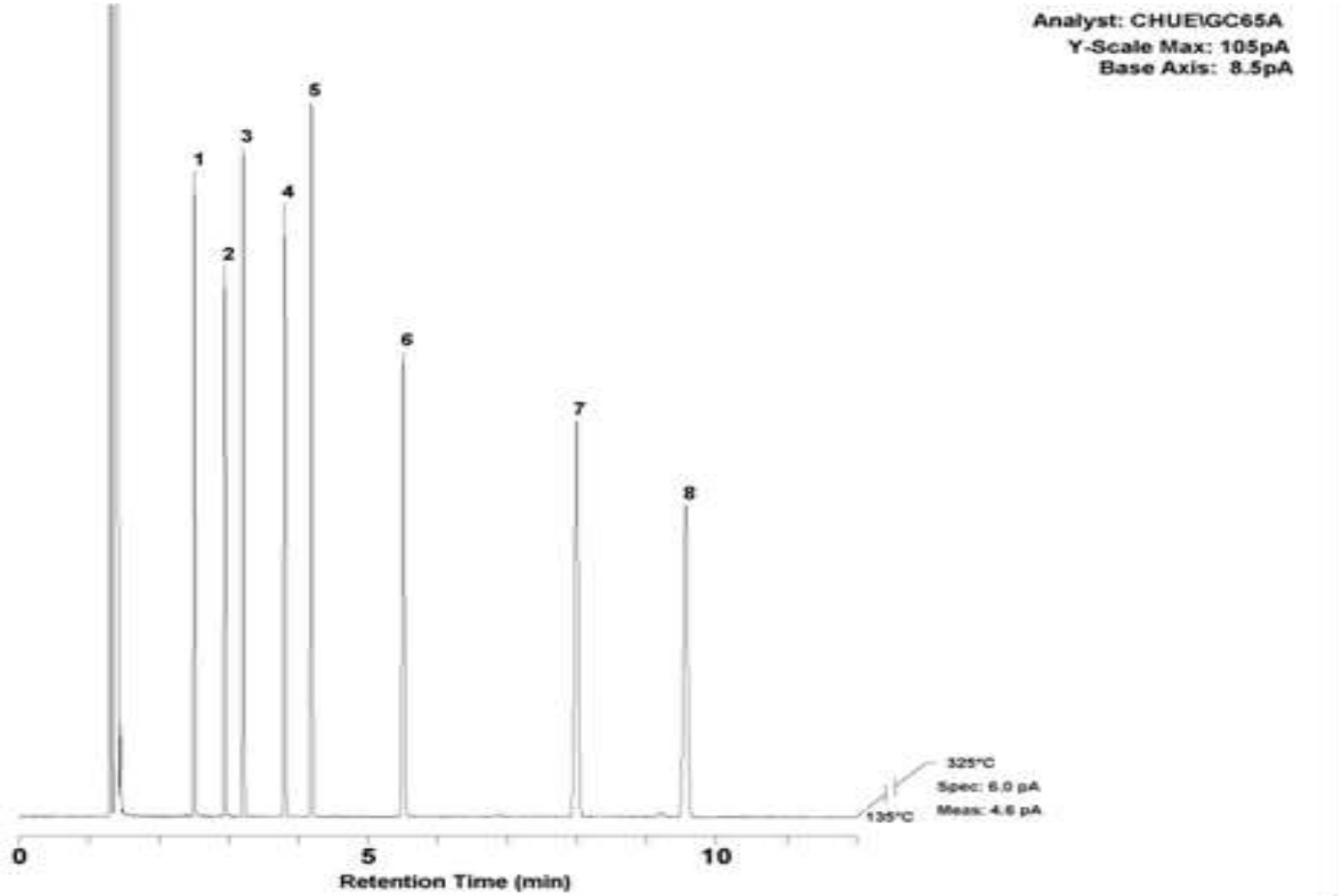
Temperature Limits: -60°C to 325°C (350°C Pgm)

Performance Results

		Spec.	Meas.
Theoretical Plates/Meter:			
PENTADECANE		3900	4533
UTE%:			
PENTADECANE			97.8%
Retention Index:			
1-UNDECANOL		1371.0 to 1372.0	1371.6
ACENAPHTHYLENE		1459.3 to 1460.3	1460.0

Compound Identification	Retent. Time	Part. Ratio	1/2-Width
1. 1,6-HEXANEDIOL	2.507	.93	.017
2. 4-CHLOROPHENOL	2.945	1.27	.020
3. METHYL NONANOATE	3.210	1.47	.020
4. 4-PROPYLANILINE	3.803	1.93	.025
5. TRIDECANE	4.187	2.22	.026
6. 1-UNDECANOL	5.505	3.23	.034
7. ACENAPHTHYLENE	7.992	5.15	.053
8. PENTADECANE	9.557	6.35	.061

Chromatographic Performance



Test Mixture Components

Compounds	Purpose
Hydrocarbons	Efficiency
	Retention
FAME's, PAH's	Retention
Alcohols	Activity
Acids	Acidic Character
Bases	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.....

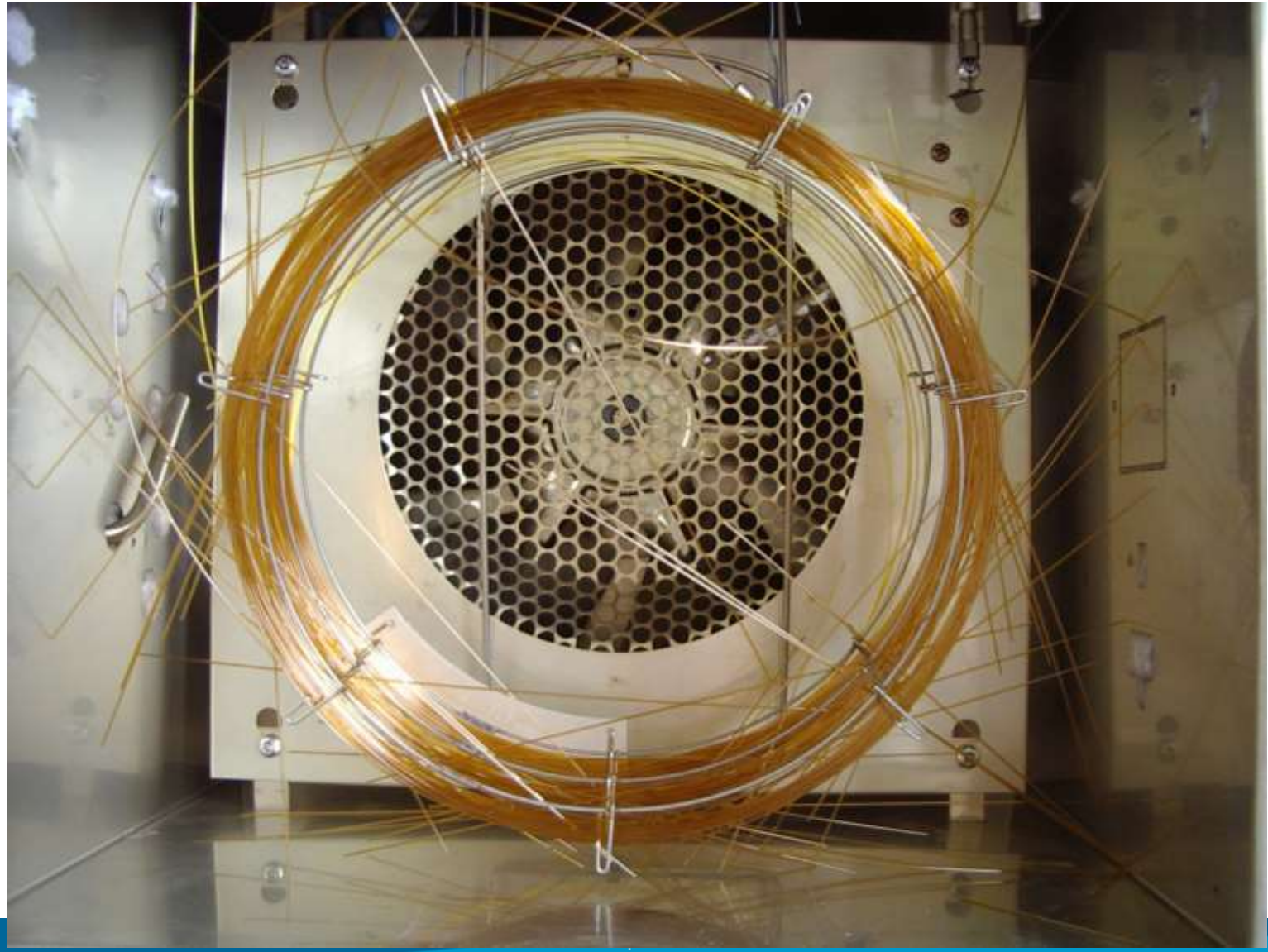
Common Causes of Column Performance Degradation

- Physical damage to the polyimide coating
- Thermal damage
- Oxidation (O_2 damage)
- Chemical damage by samples
- Contamination

Physical Damage to The Polyimide Coating

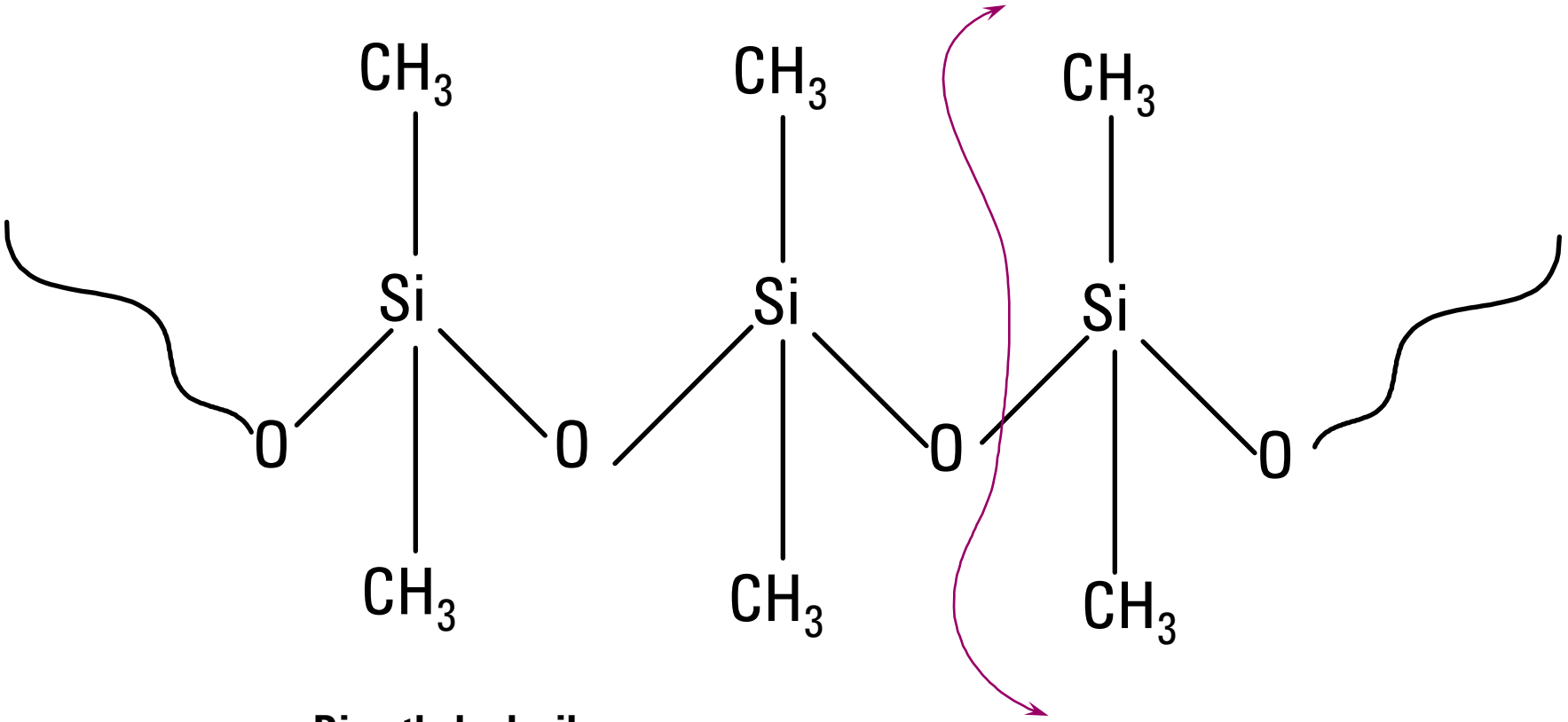
- **The smaller the tubing diameter, the more flexible it is.**
- **Avoid scratches and abrasions**
- **Immediate breakage does not always occur upon physical damage**

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

Thermal Damage

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

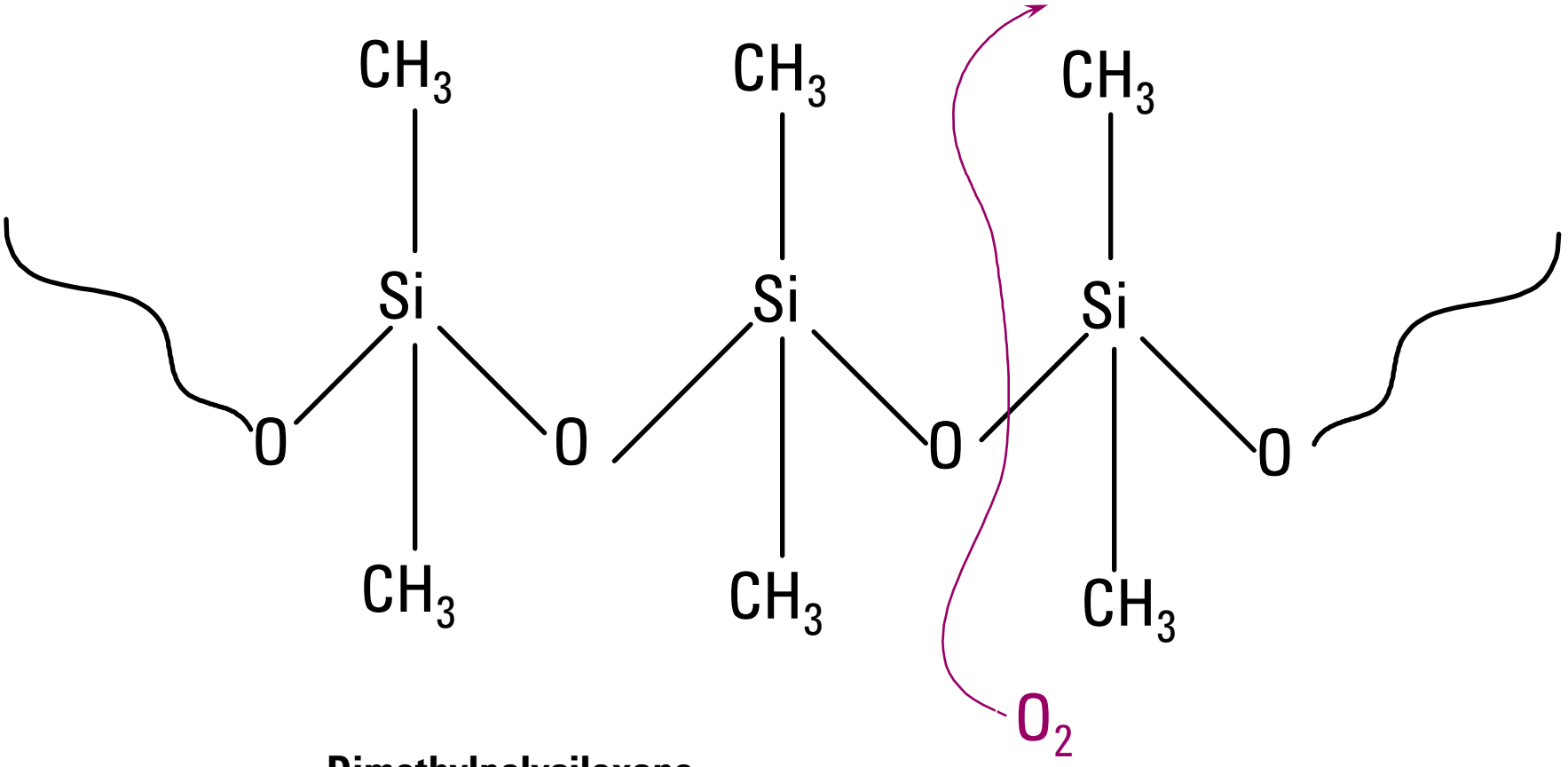
  Isothermal limit = Indefinite time

  Programmed limit = 5-10 minutes

- **Temporary "column failure" below lower temperature limit**

Oxidation (O2 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

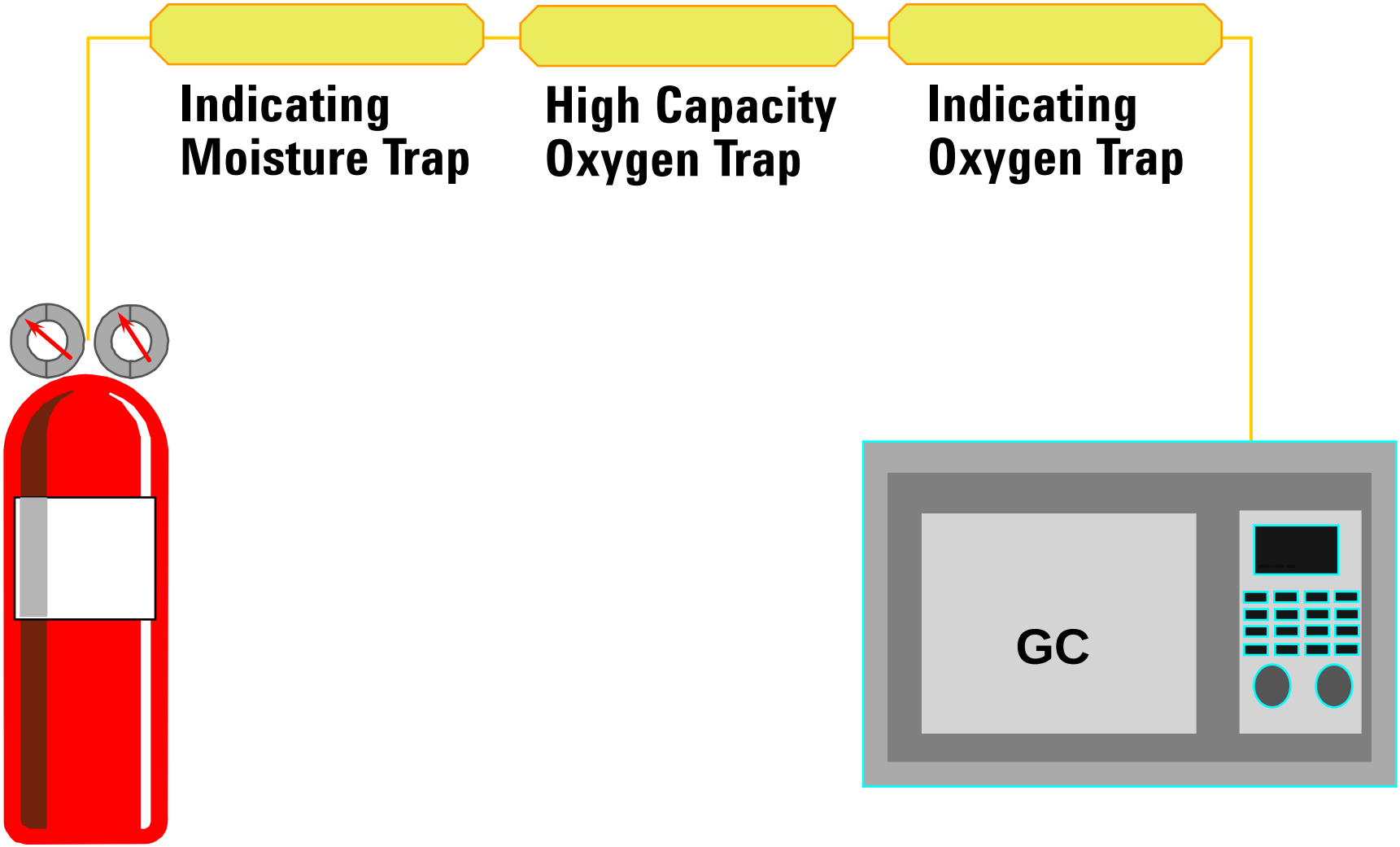
Oxygen Damage
What To Do If It Happens

- **Rapid damage to the column**
- **Usually results in irreversible column damage**

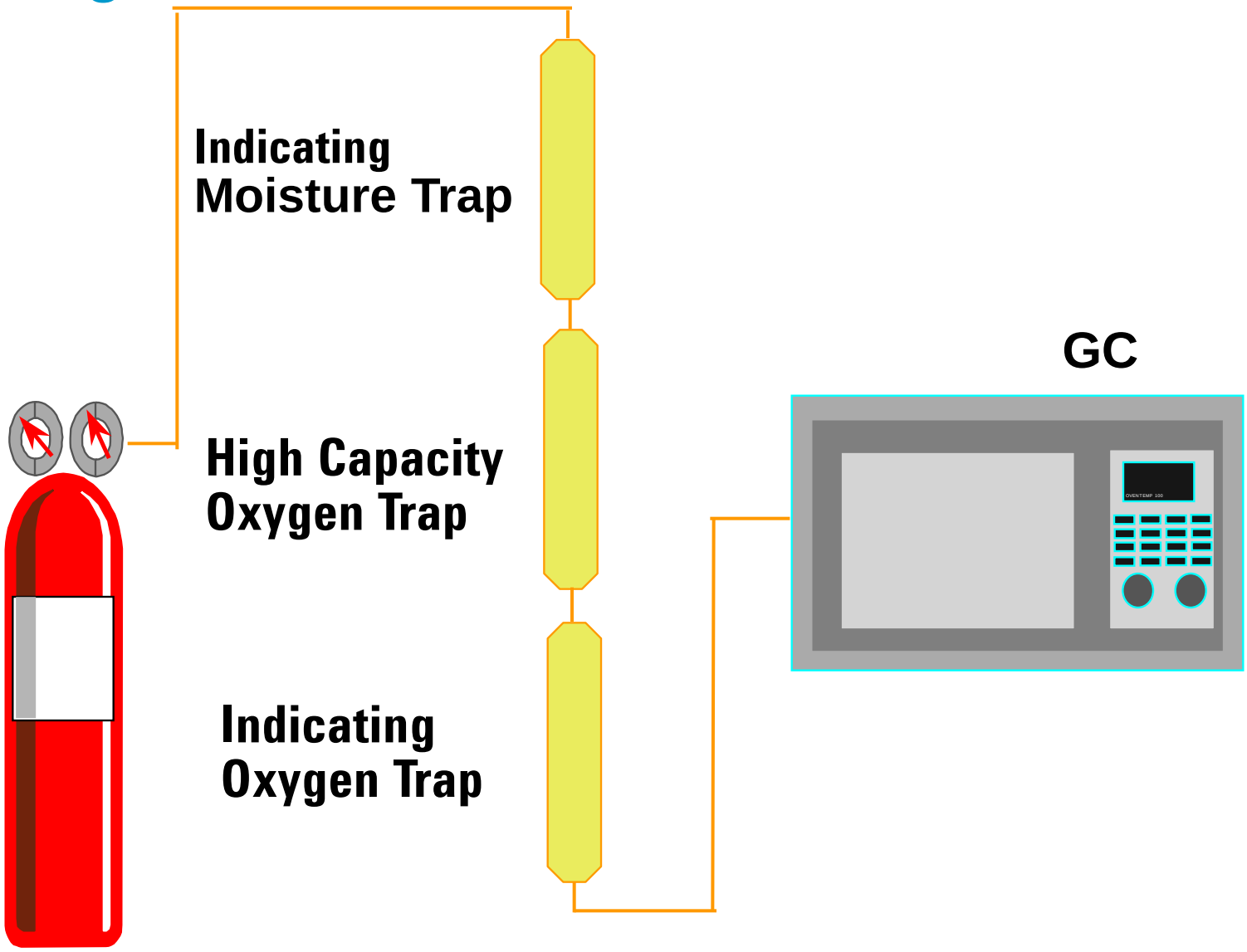
How to Prevent Column Damage by Oxygen

- **High quality carrier gas (4 nine's or greater)**
- **Leak free injector and carrier lines**
  - **Change septa**
  - **Maintain gas regulator fittings**
- **Appropriate impurity traps**

Configurations for Carrier Gas Purifiers



Configurations for Carrier Gas Purifiers



Chemical Damage

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

HCl NH_3 KOH $NaOH$

H_2SO_4 H_3PO_4 HF etc.

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

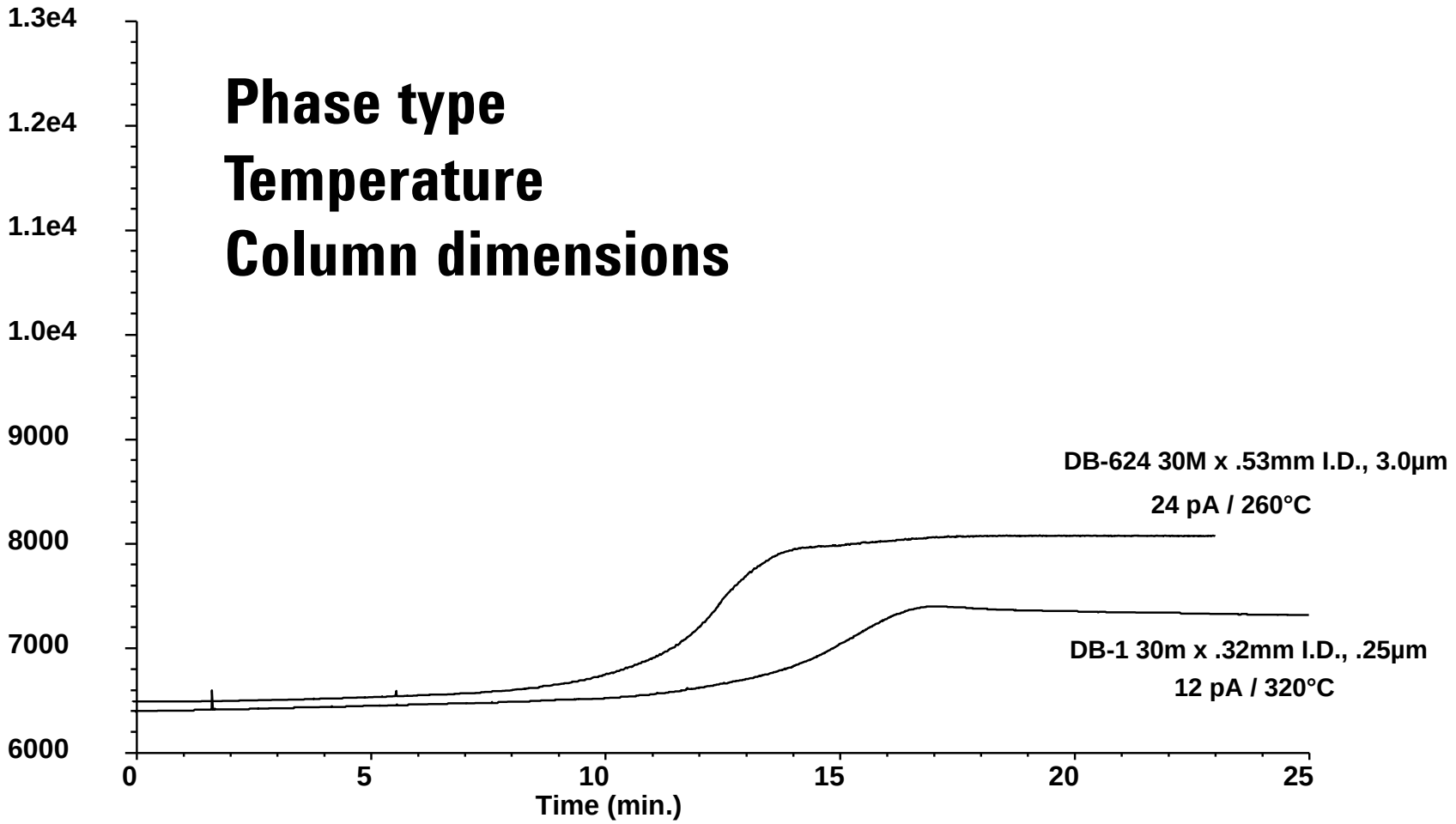
Chemical Damage
What To Do If It Happens

- Remove 1/2 - 1 meter from the front of the columns
- Severe cases may require removal of up to 5 meters

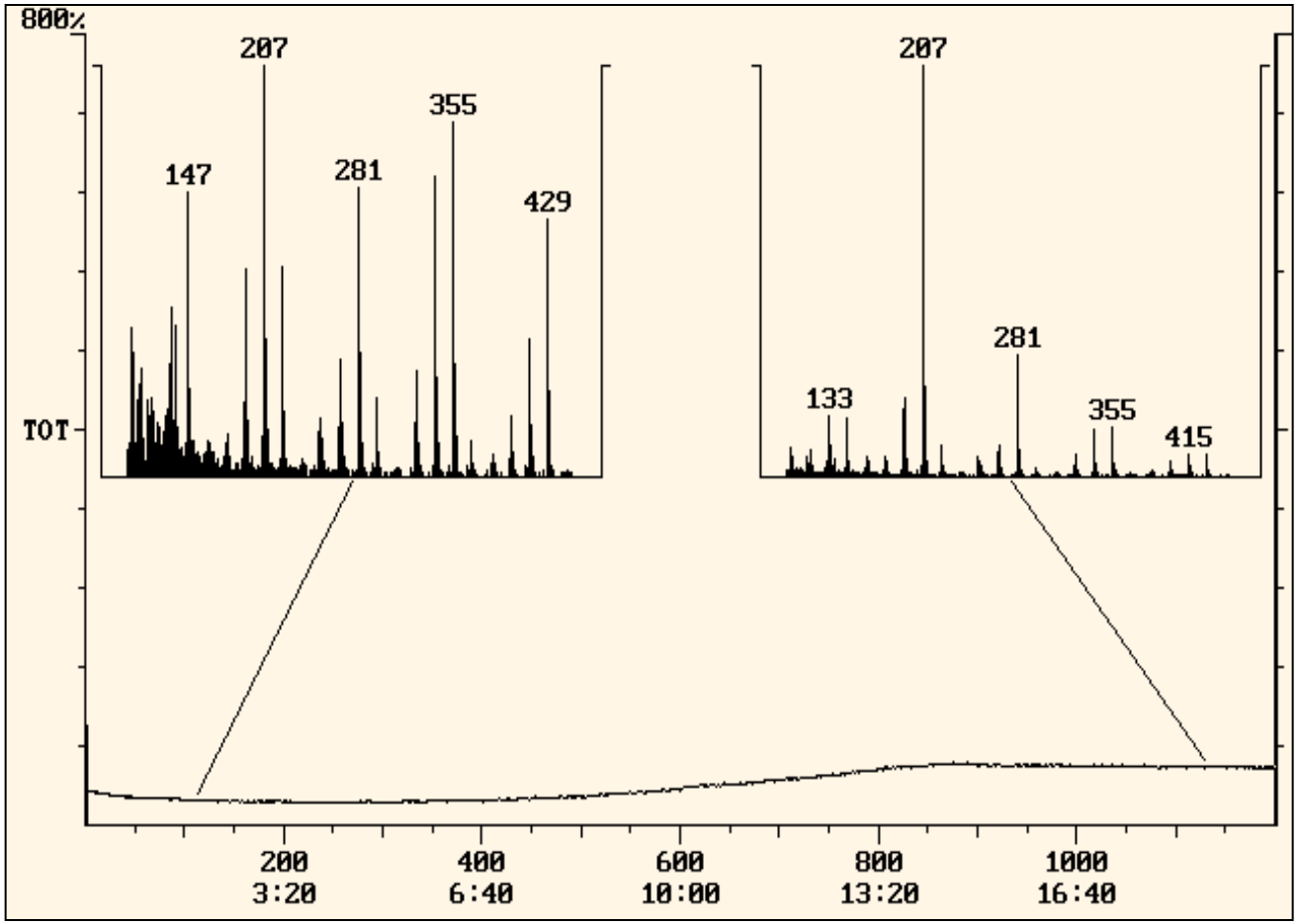
What is Normal Column Bleed

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?

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**

<u>All</u> 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.

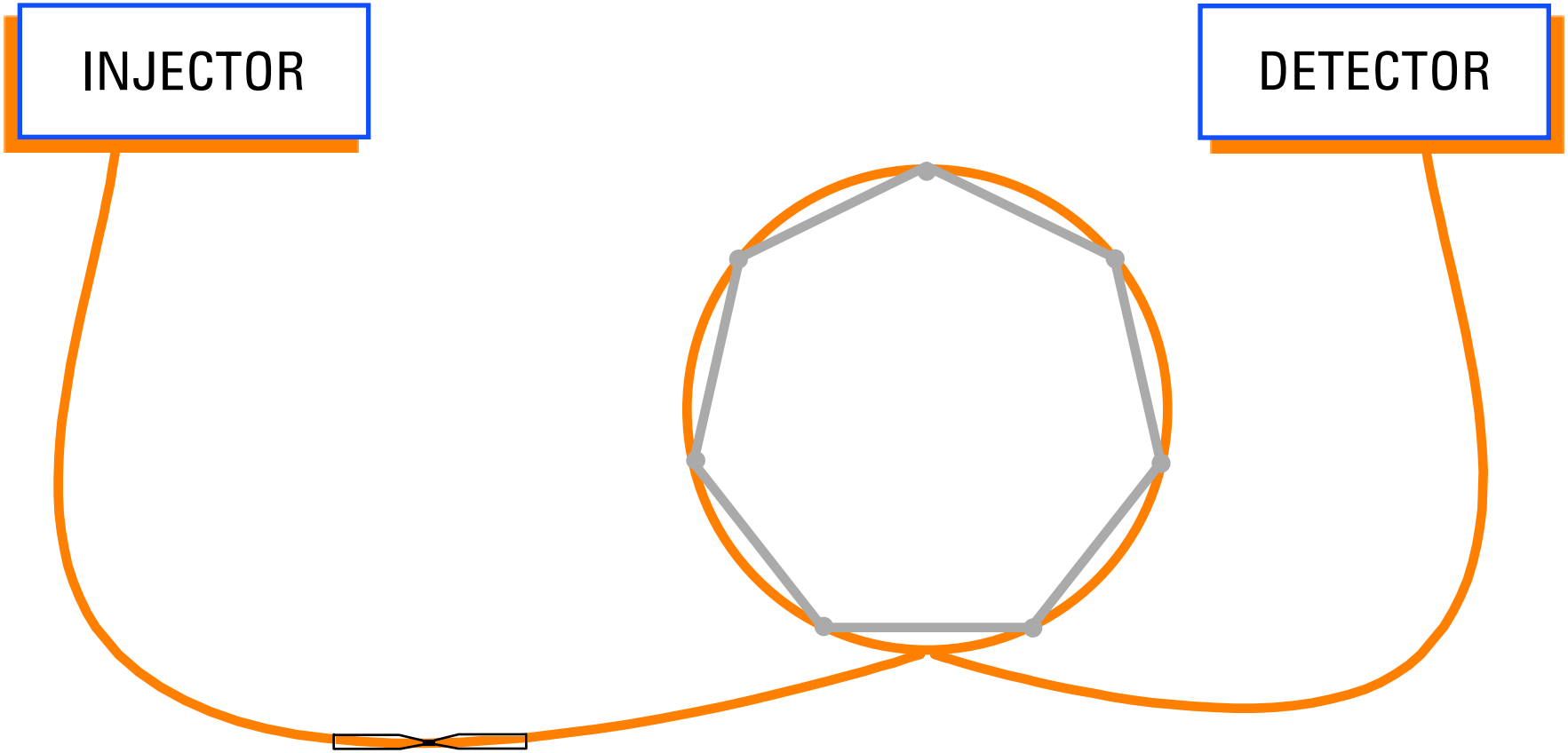
Semi-Volatile Residues

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

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

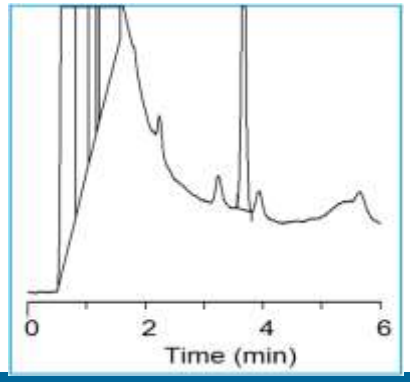
Methods to Minimize Non-Volatile Residue Problems

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

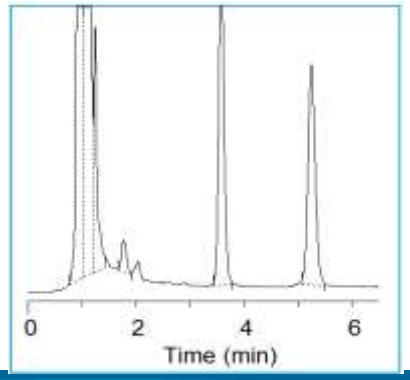
Why Perform Sample Preparation?

- To acquire desired sensitivity/selectivity
- To reduce contamination of inlet supplies and columns
- To protect your investment if using very sensitive and expensive instrumentation

Pesticides in Avocado *without* SP



Pesticides in Avocado with SP



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**
- **Use Sample Prep where possible**

Agilent/J&W Technical Support

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302-993-5304 (phone)*

**** Select option 41***

866-422-5571 (fax)

email: gc-column-support@agilent.com

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