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"

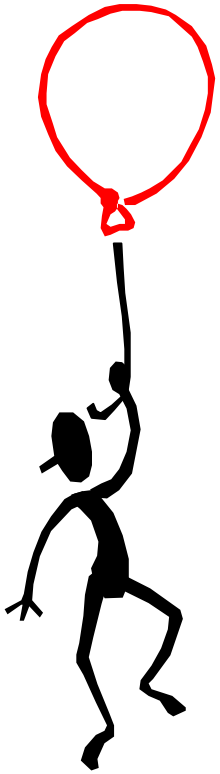
Column Installation

"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**

Cutting The Column

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

<u>Recommended tools:</u>

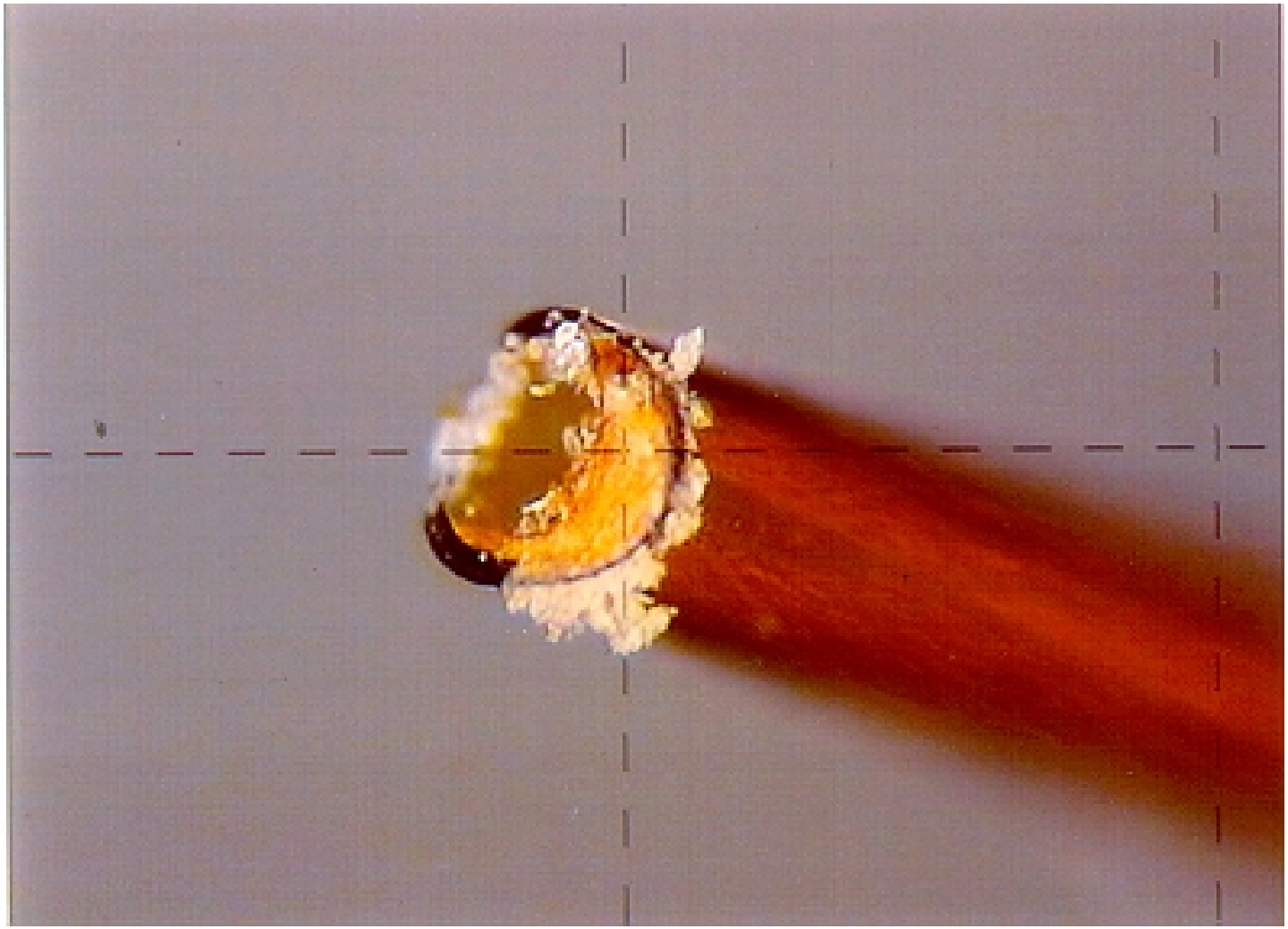
Diamond or carbide tipped pencil; or sapphire cleaving tool, ceramic wafer

Ocular

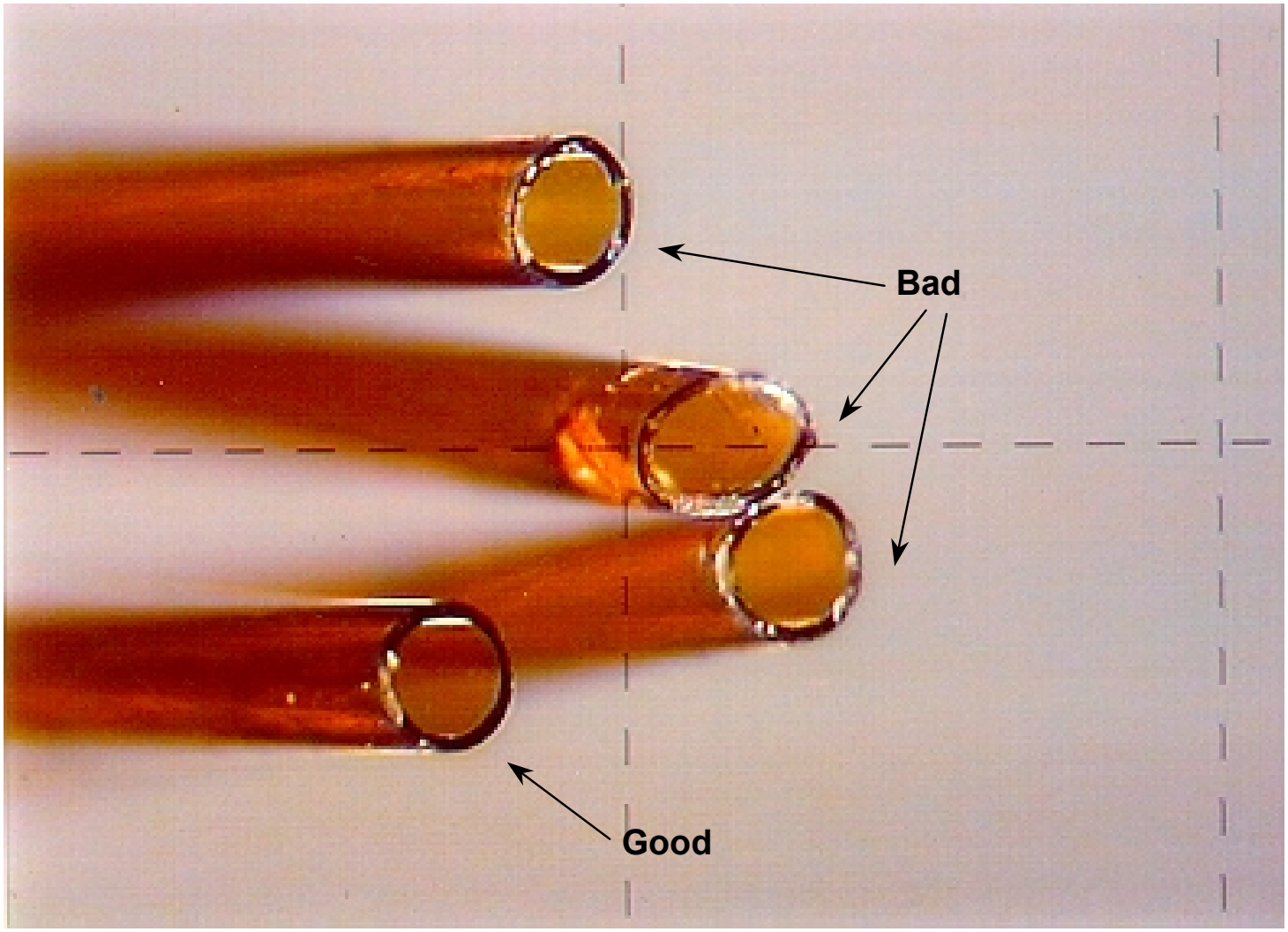
<u>Do not use:</u>

Scissors, file, etc.

Example of a Bad Cut



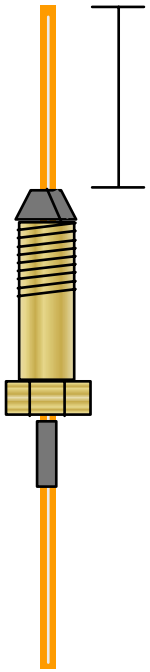
Examples of Column Cuts



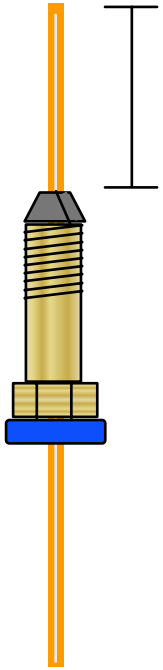
Column Installation

Measuring the right distance

White out

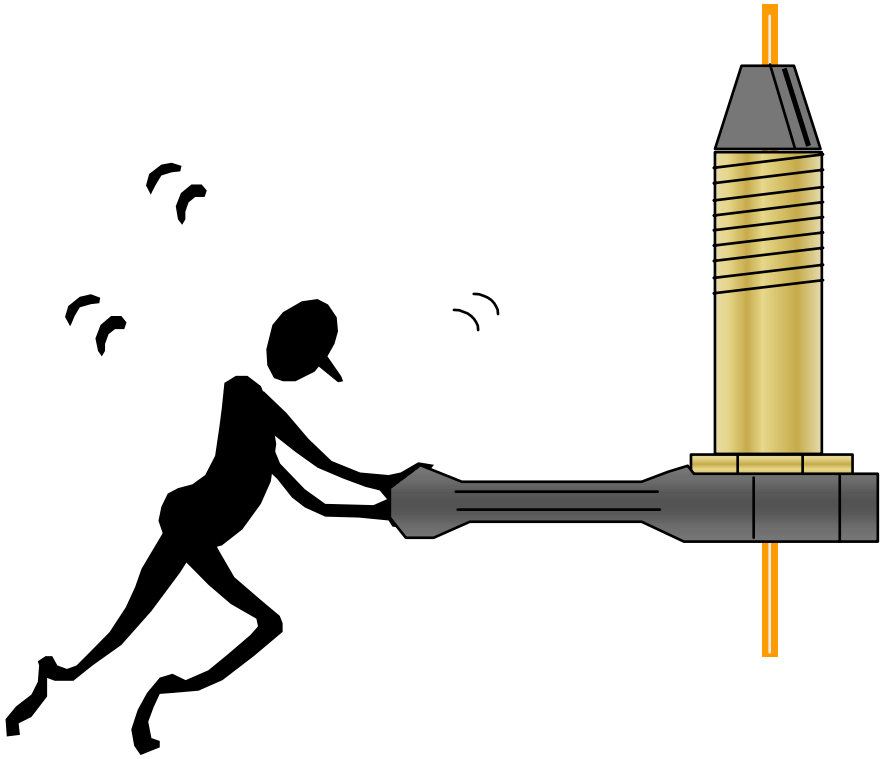


Septa

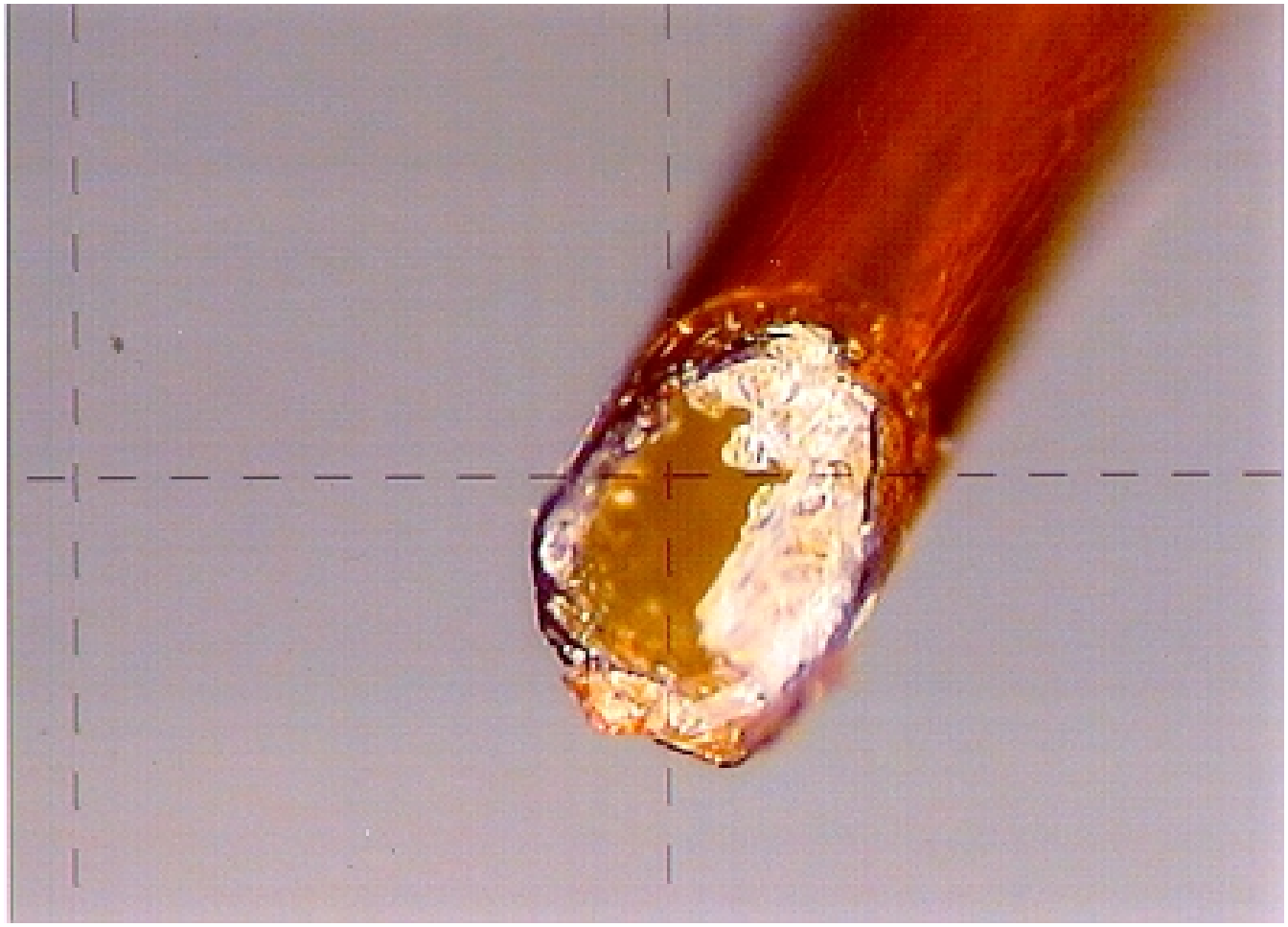


Column Installation

How tight is tight?



Overtightened Ferrule



Column Installation

Leak Check

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

DO NOT USE SNOOP

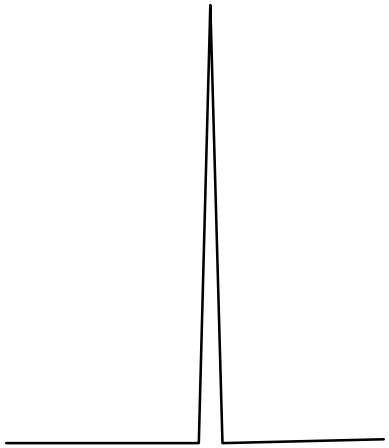
Leak and Installation Check

Inject a non-retained compound vs DB-1

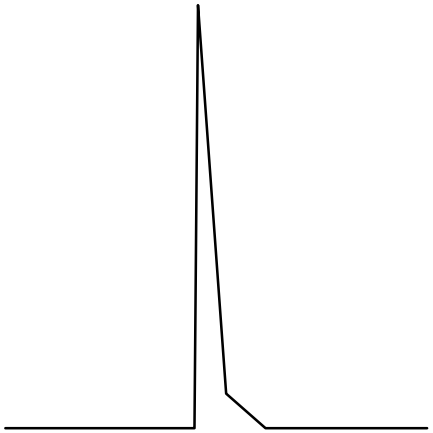
Detector	Compound
FID	Methane or Butane
ECD	$MeCl_2$ (headspace or diluted)
NPD	CH_3CN (headspace or diluted)
TCD	Air
MS	Air or Butane

The peak should be sharp and symmetrical

Non-Retained Peak Shapes



Good Installation



Improper Installation or Injector Leak

Check for: Injector or septum leak

Too low of a split ratio

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 35-40 cm/sec
H_2 45-60 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:

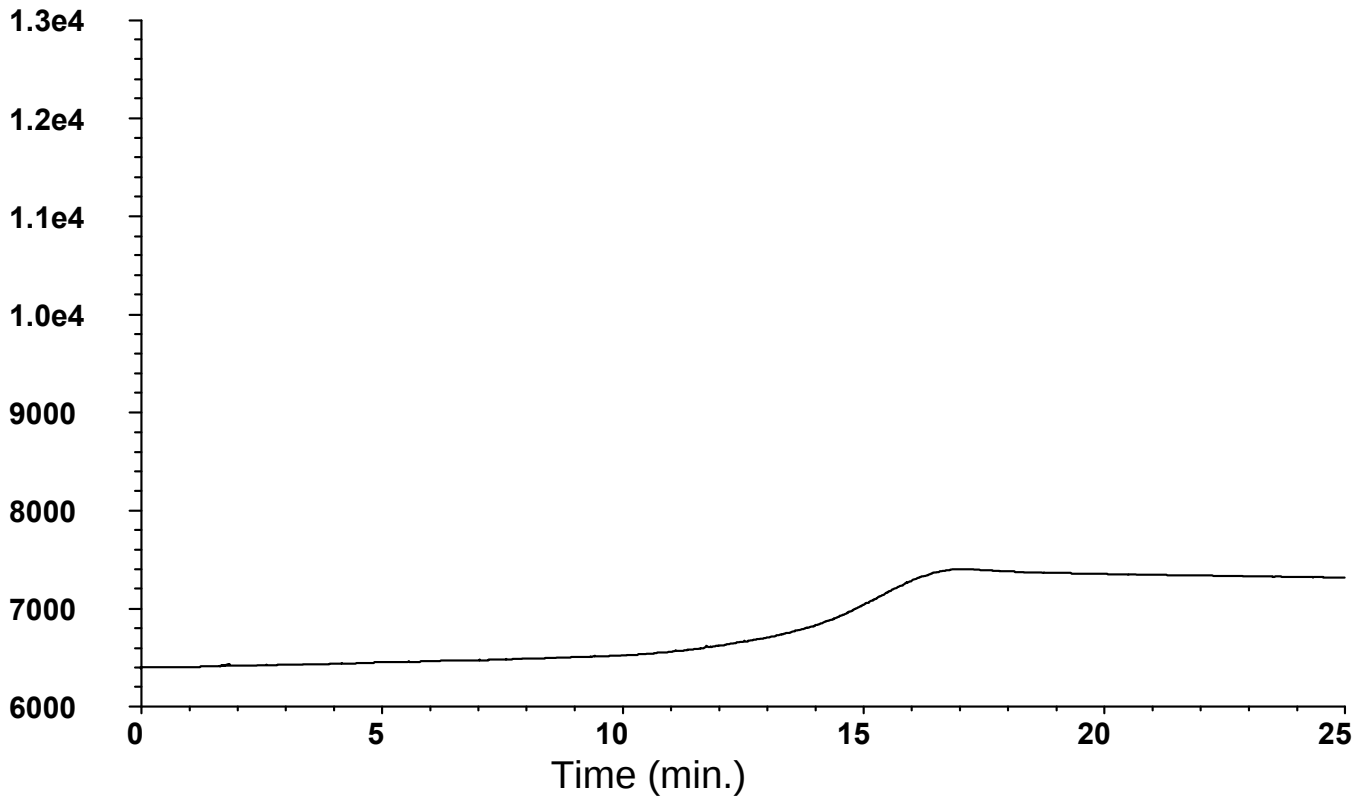
- Isothermal maximum temperature OR
- 20° to 30°C above highest operation temperature
- Temperature programming is not necessary

Stop conditioning when the stable baseline is obtained:

- 1 to 2 hours in most cases

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

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:		
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

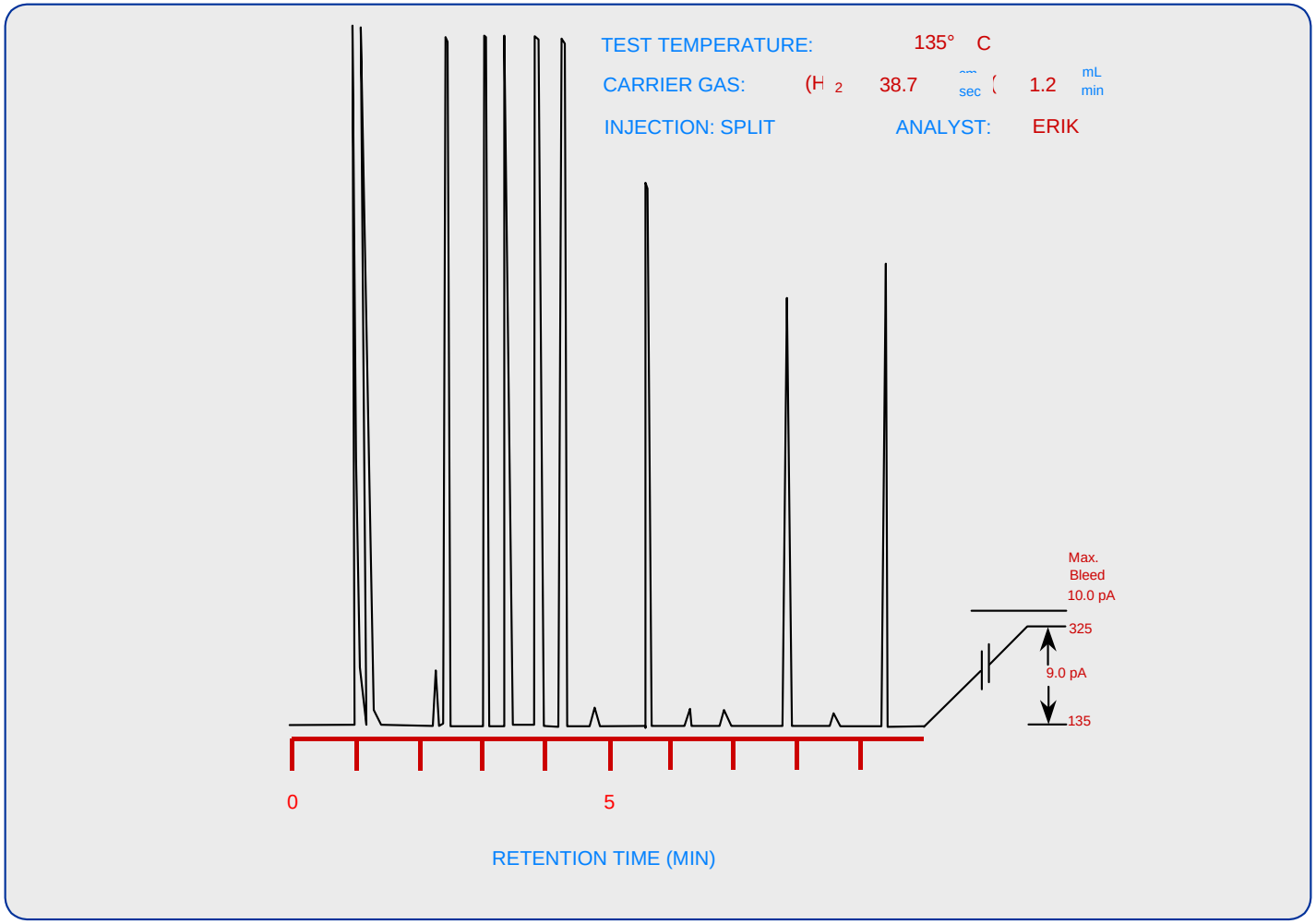
COMPOUND IDENTIFICATION	RETENTION TIME (R	PARTITION RATIO (k)	PEAK WIDTH (W 1/2)
1,6-HEXANEDIOL	2.51	0.9	0.019
4-CHLOROPHENOL	2.95	1.3	0.022
METHYL NONANOATE	3.21	1.5	0.022
4-PROPYLANILINE	3.81	1.9	0.026
TRIDECANE	4.20	2.2	0.027
1-UNDECANOL	5.52	3.3	0.036
ACENAPHTHYLENE	8.00	5.2	0.053
PENTADECANE	9.58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

Approximately 5-10 ng on column

1.29

Agilent Technologies

Chromatographic Performance



Test Mixture Components

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

Own Test Mixture

- More specific
- Selective detectors
- Actual concentrations
- No conditions or instrument changes

Break Number 1

- For Questions and Answers
- Press *1 on Your Phone to
- Ask a Question

An Ounce of Prevention.....

Common Causes of Column Performance Degradation

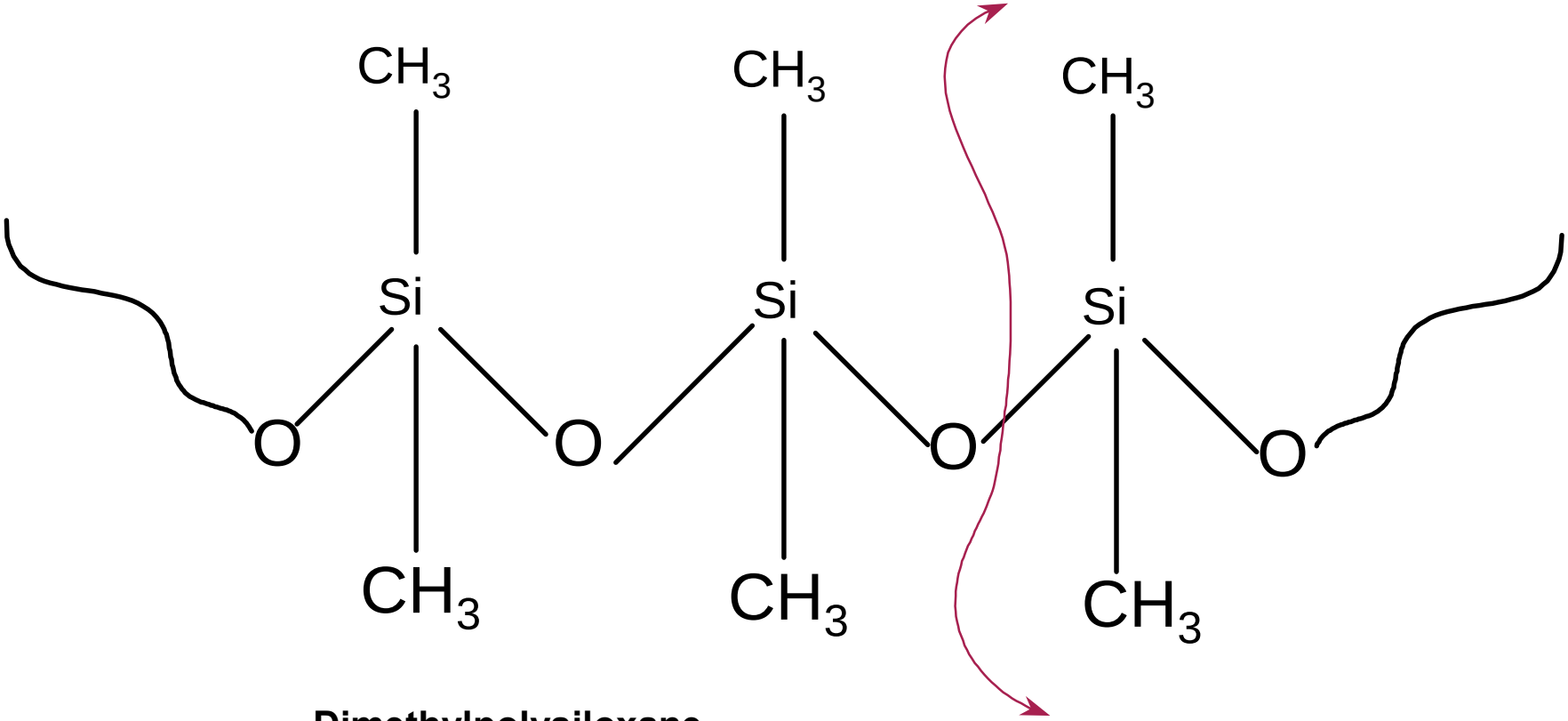
- Physical damage to the polyimide coating
- Thermal damage
- Oxidation (O_2 damage)
- Chemical damage by samples
- Contamination

Physical Damage to The Polyimide Coating

- Bending radius decreases with increasing tubing diameter
- Avoid scratches and abrasions
- Immediate breakage does not always occur upon physical damage

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

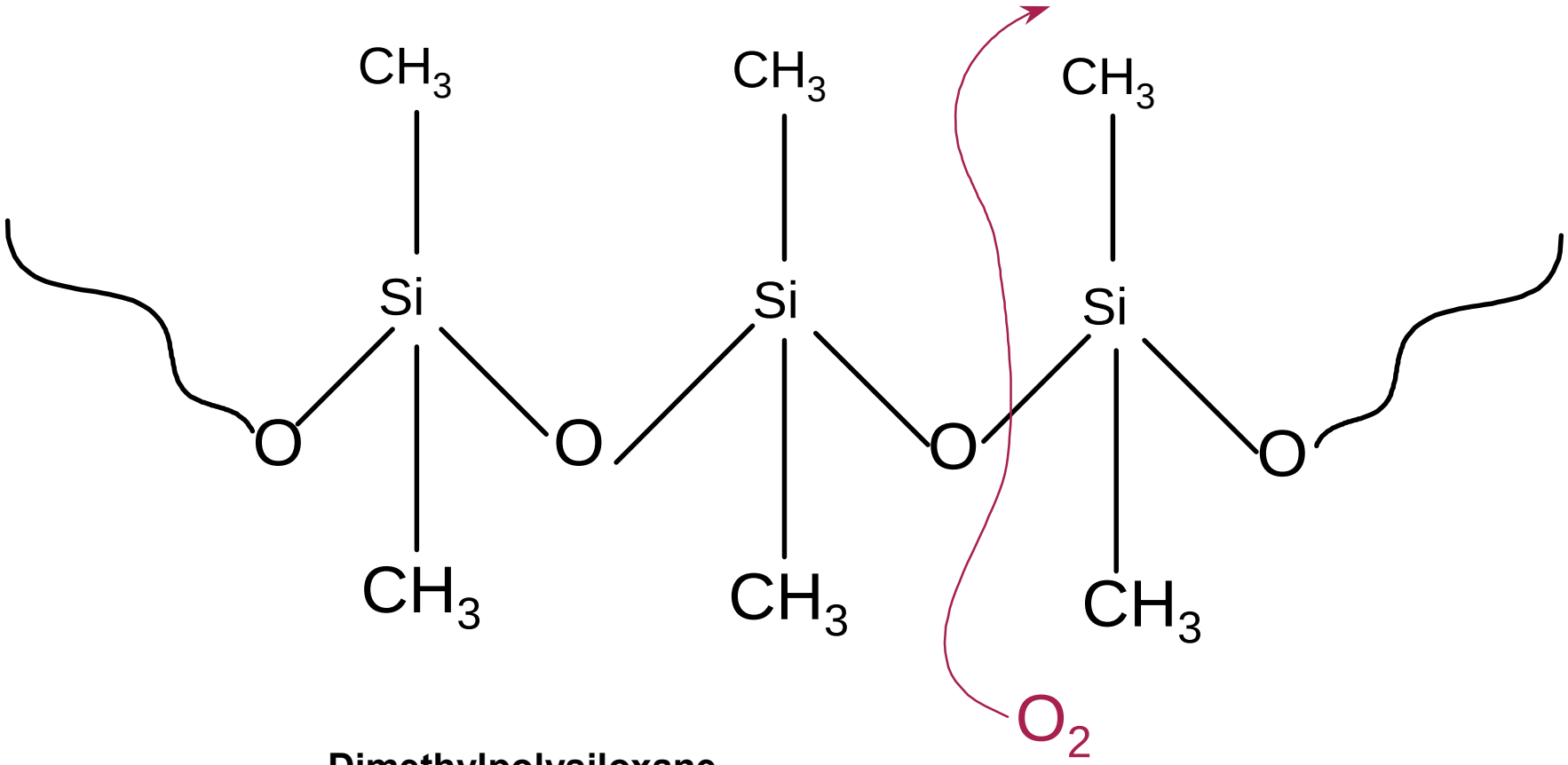
Isothermal limit = Indefinite time

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

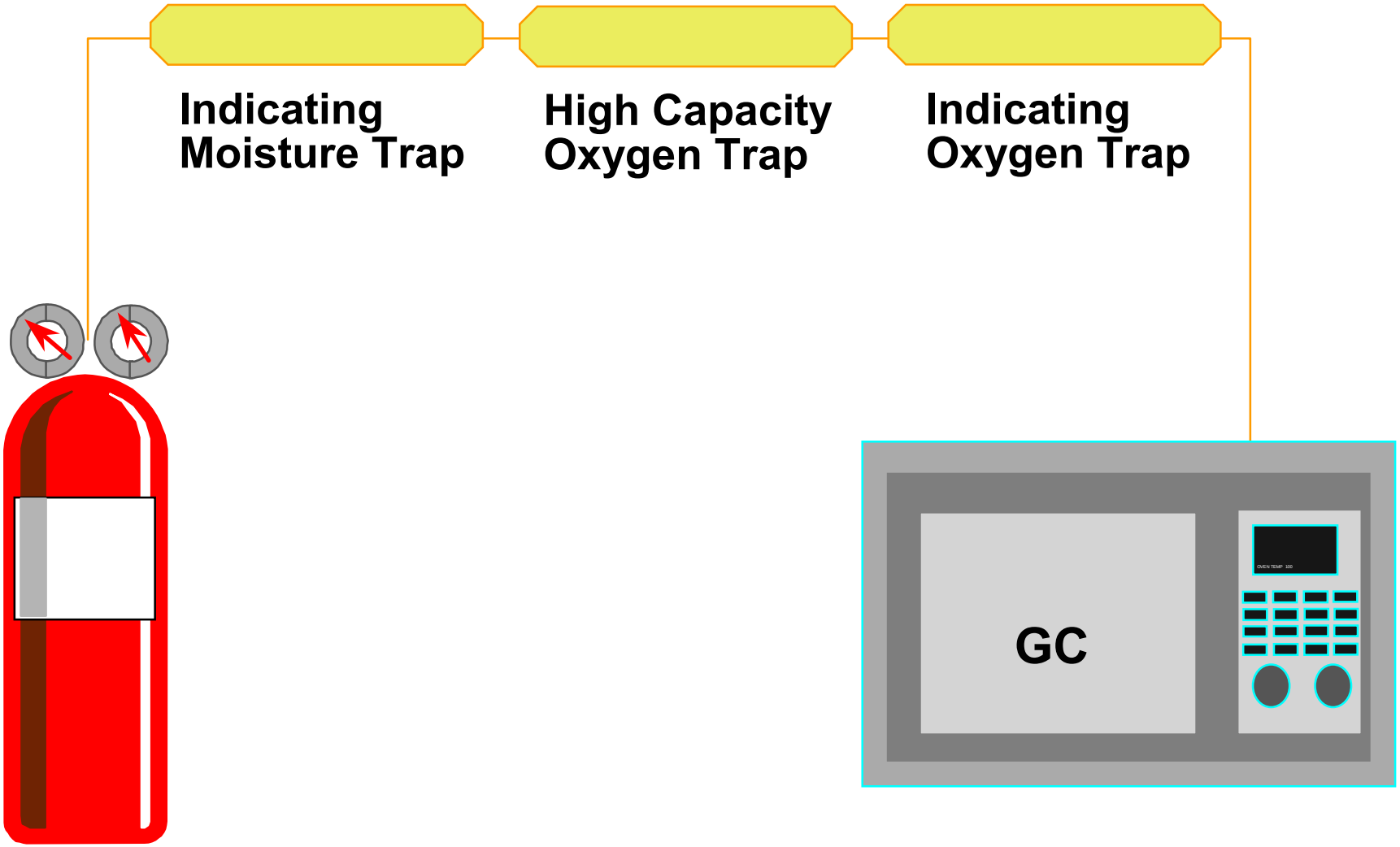
High quality carrier gas (4 nine's or greater)

Leak free injector and carrier lines

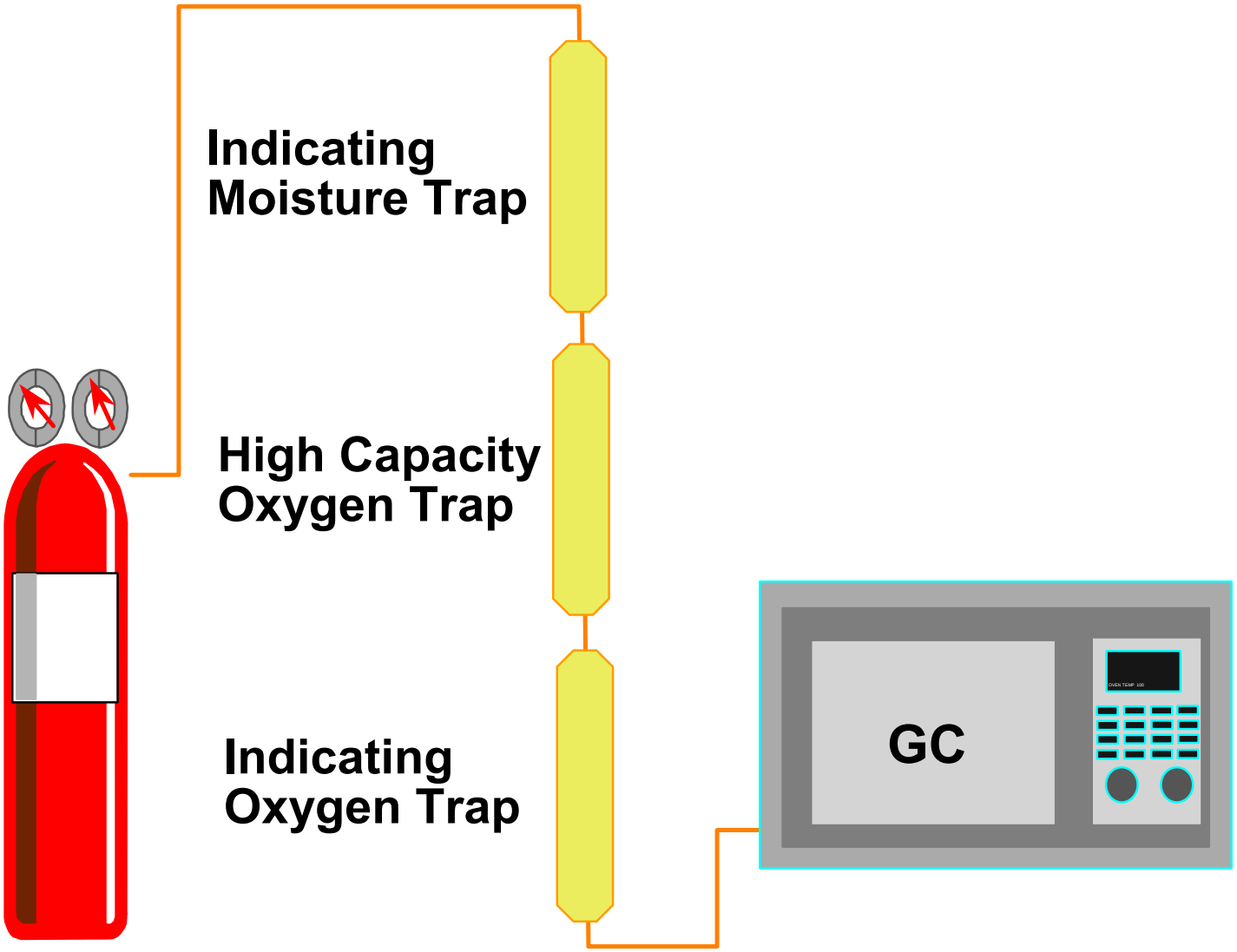
- Change septa
- Maintain gas regulator fittings

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/retentionlack of inertness or loss of resolution/retention

Chemical Damage

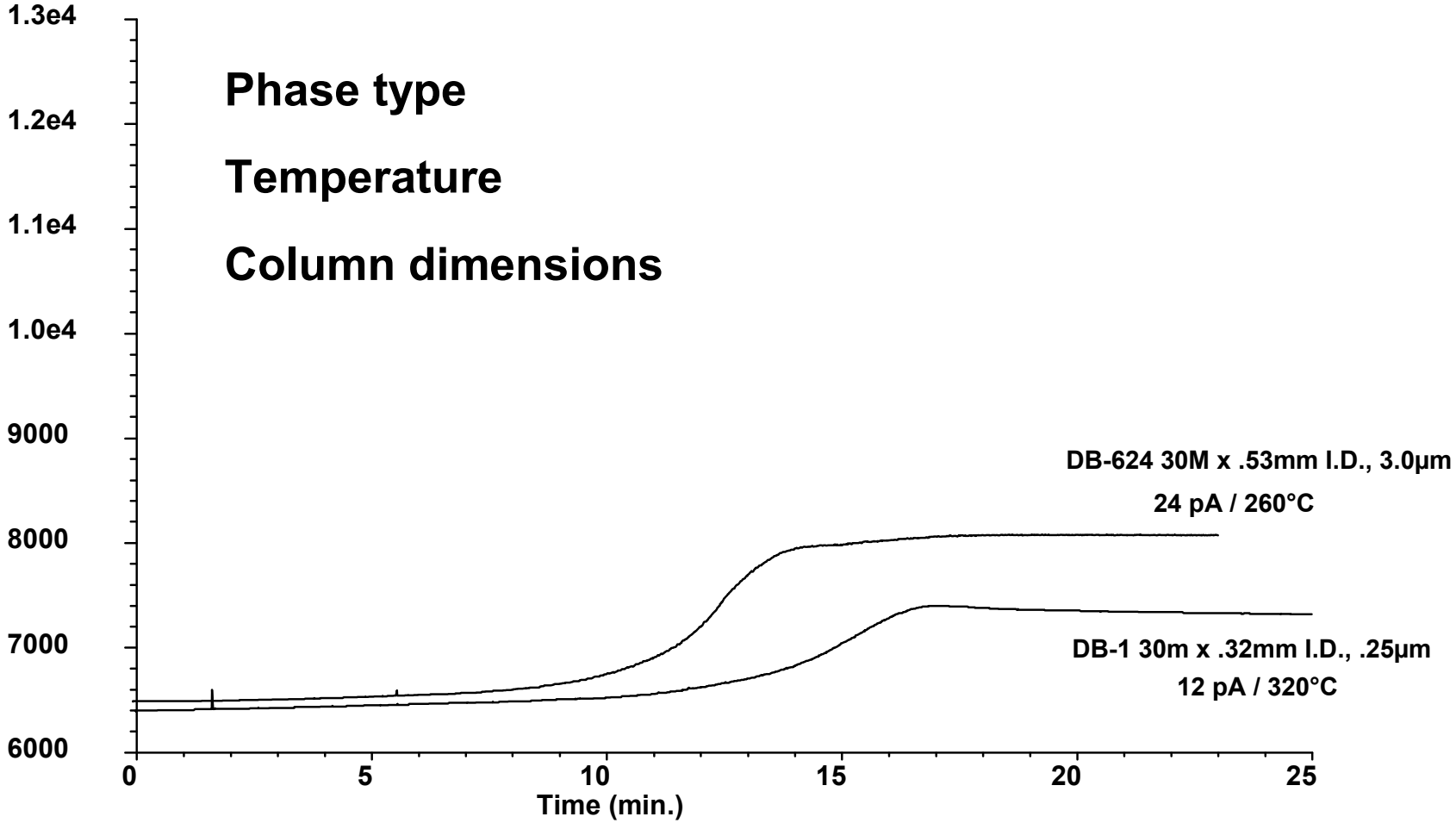
What To Do If It Happens

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

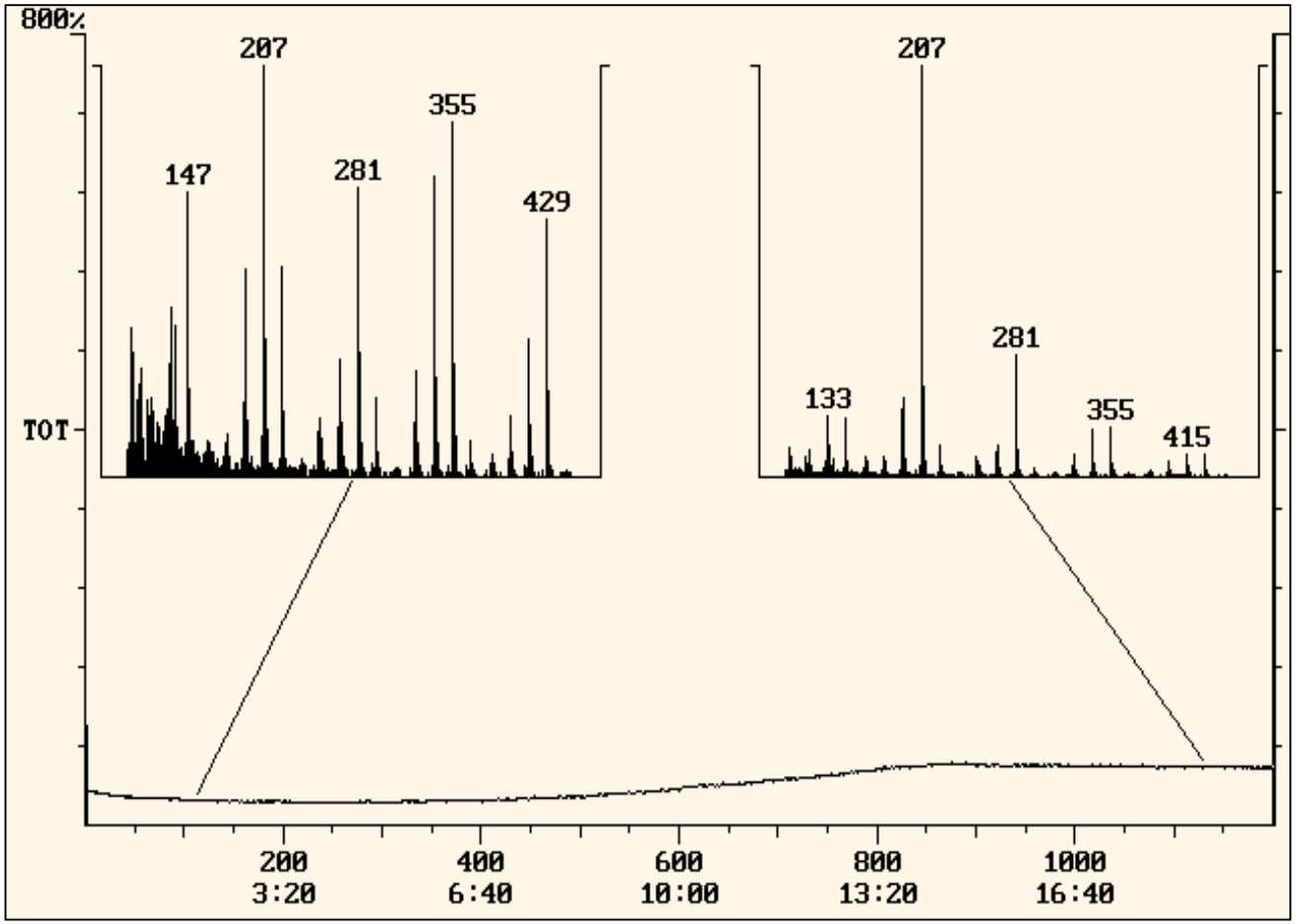
What is Normal Column Bleed

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 <u>NOT</u>

A high baseline at low temperature

Wandering or drifting baseline at any temperature

Discrete peaks

Break Number 2

- For Questions and Answers
- Press *1 on Your Phone to
- Ask a Question

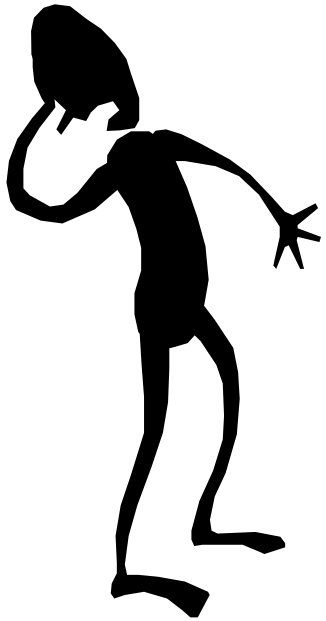
Column Contamination

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!!

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

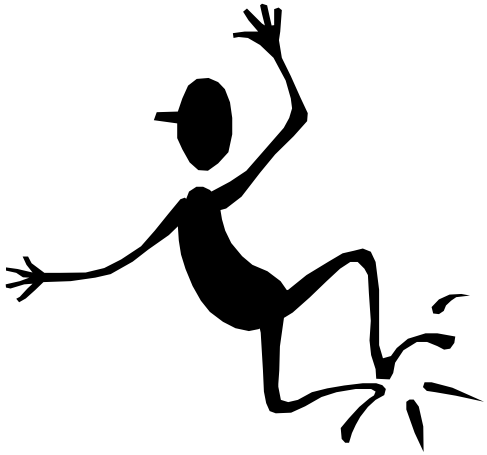
Any portion of the sample that elutes from the column after the current chromatographic run.

Methods to Minimize Non-Volatile Residue Problems

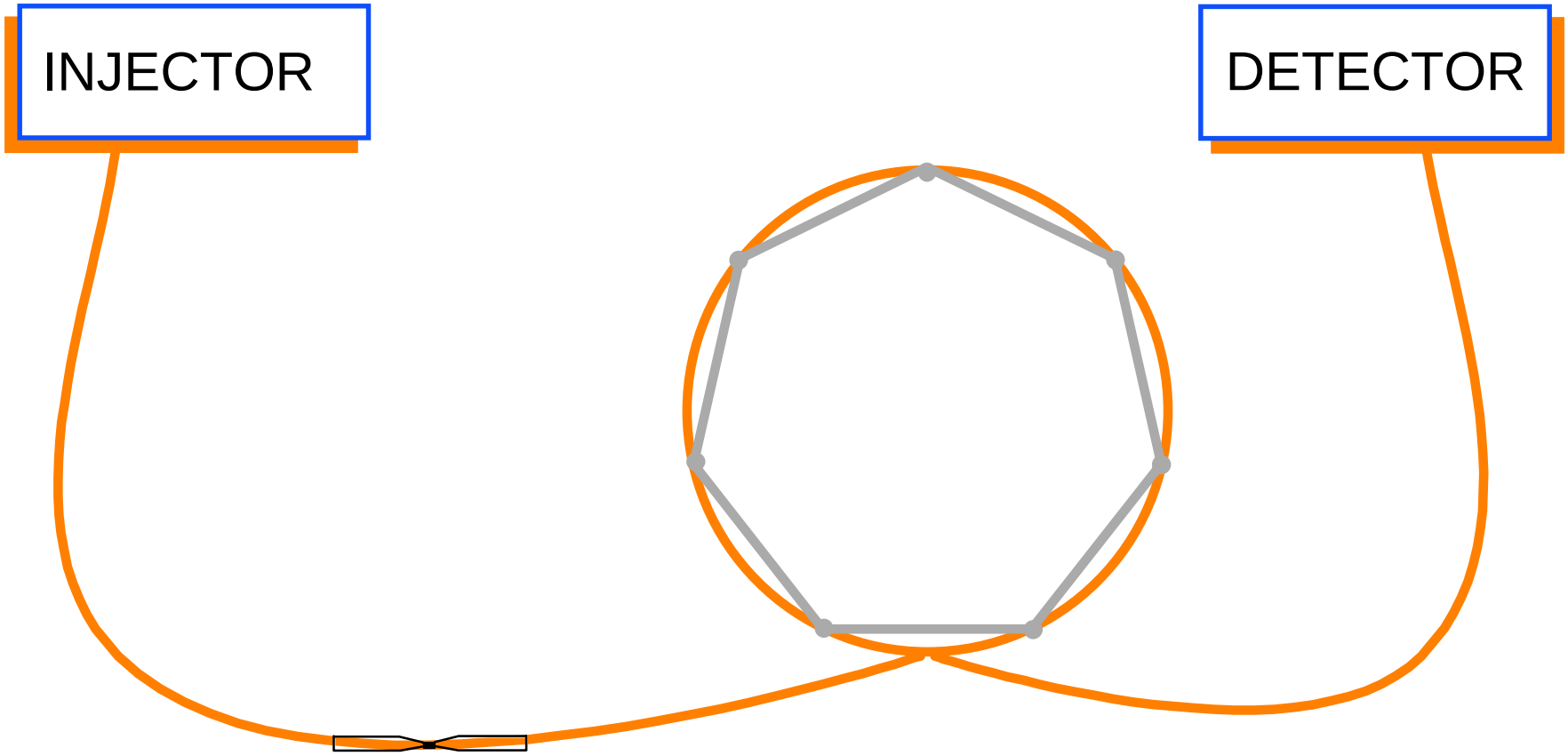
Sample cleanup

Packed injection port liners

Guard columns



Guard Column



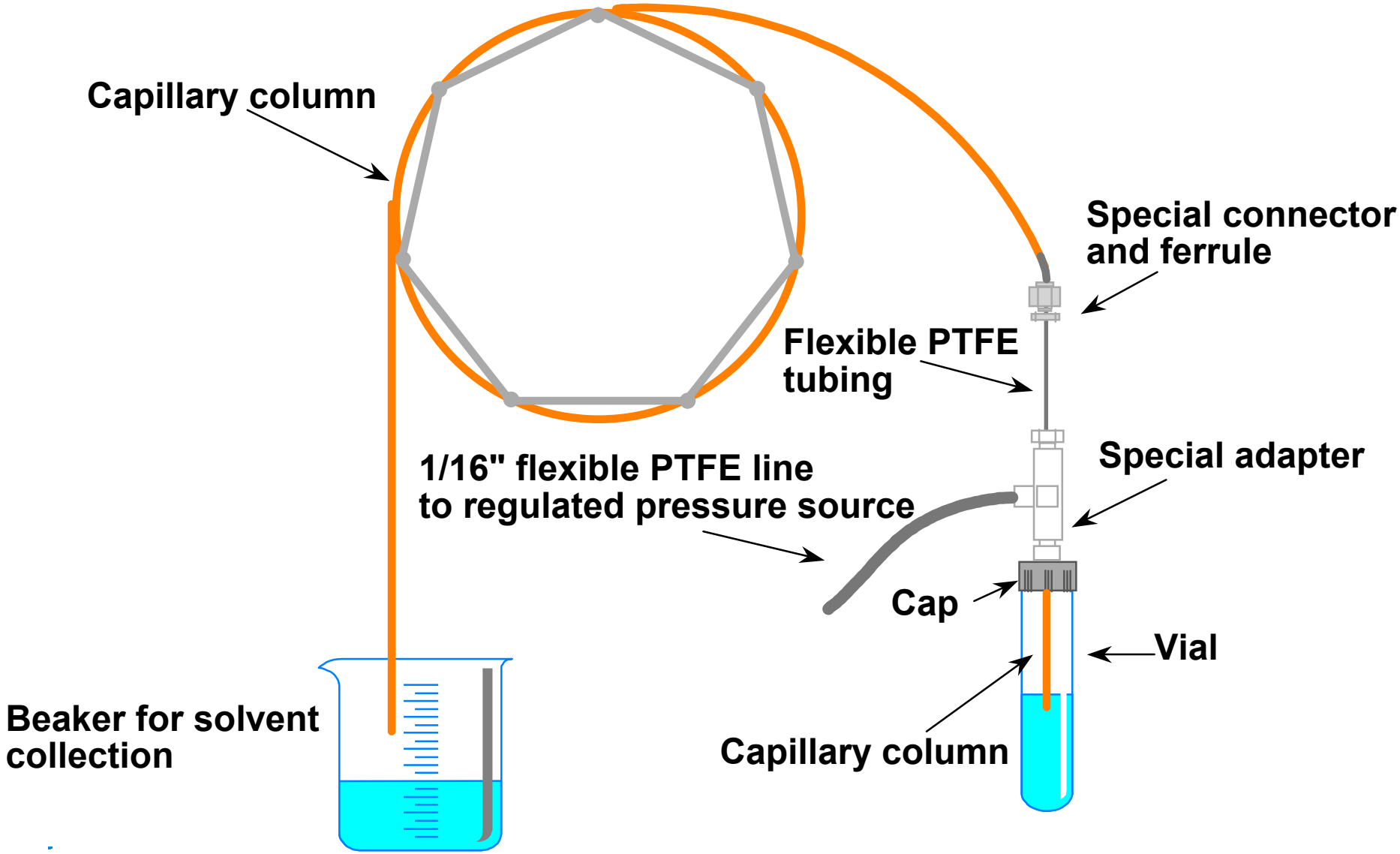
The guard column is 0.5 - 10 meters of deactivated fused silica tubing with the same diameter as the analytical column. It is connected with a zero dead volume union.

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


Capillary column
Special connector and ferrule
Flexible PTFE tubing
Special adapter
1/16" flexible PTFE line to regulated pressure source
Cap
Vial
Beaker for solvent collection
Capillary column

Semi-Volatile Contamination

What To Do If It Happens

- "bake out" the column
  - Limit to 1-2 hours
  - May polymerize some contamination
  - Reduces column life
- Solvent rinse the column

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

J&W Scientific Technical Support

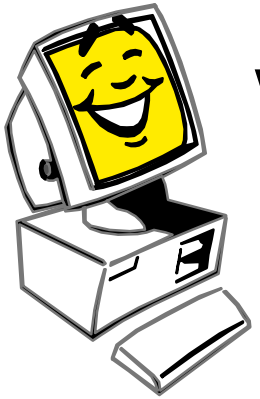
800-227-9770 (phone: US & Canada)*

302-993-5304 (phone)*

** Select option 4, then option 1.*

916-608-1964 (fax)

www.agilent.com/chem



Final Wrap-up e-Seminar Questions Reference Information

For your reference, the following products described during this e-Seminar are available from Agilent Technologies. Visit our web site at *www.agilent.com/chem* or contact your local sales office.

• Column Cutter	5183-4620
• Flow Tracker 2000	5183-4780
• Electronic Leak Detector	5182-9646
• Rinse Kit	9301-0982
• Gas Purification Systems	Multiple Options
• Gas Purifiers	Multiple Options