Optimizing the Analysis of Volatile Organic Compounds by Purge and Trap.

USEPA Method 8260

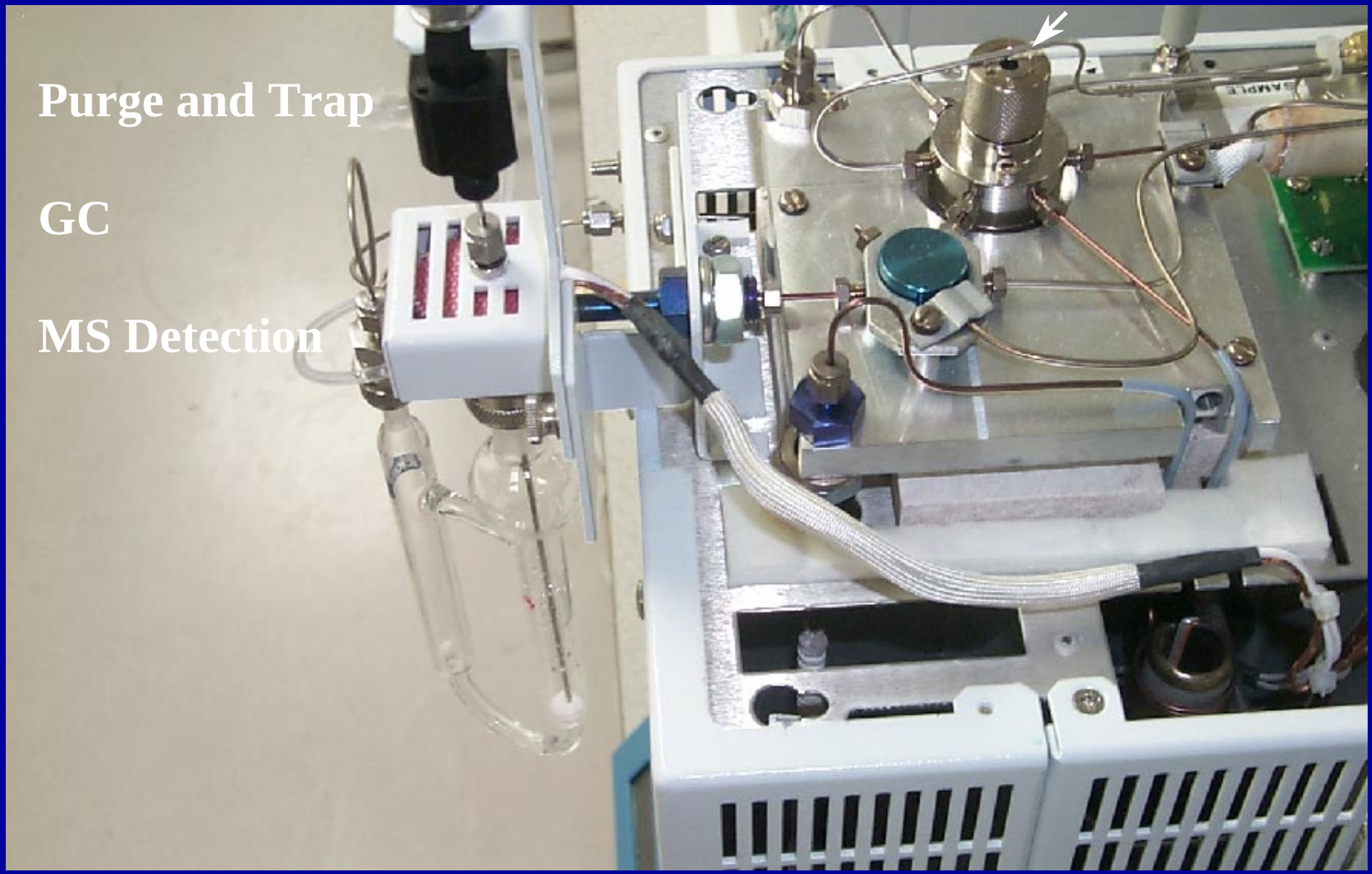
RESTEK

EPA Method 8260B

Purge and Trap

GC

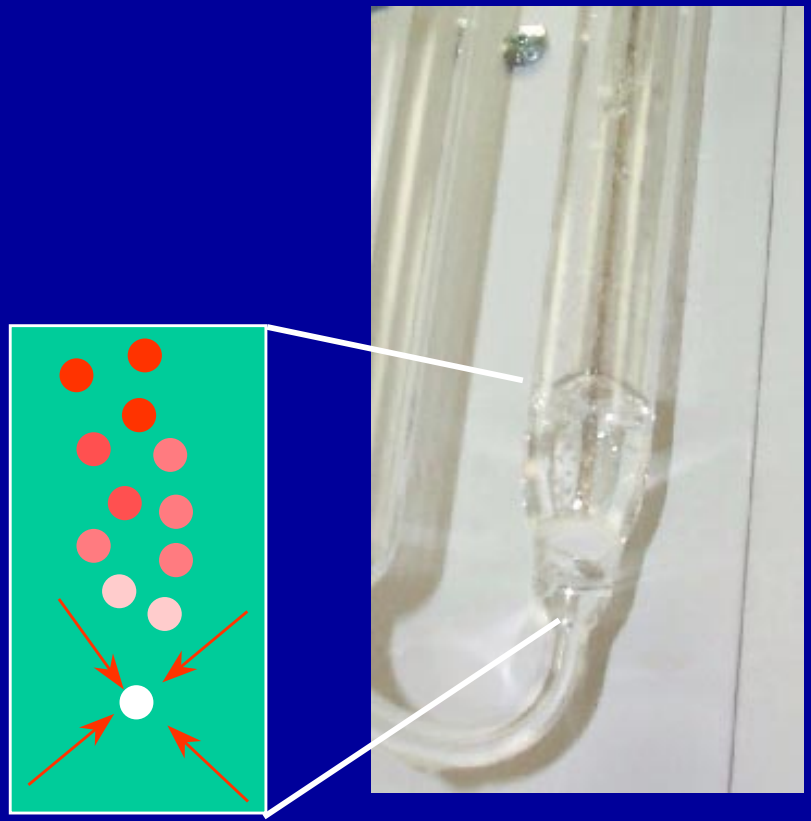
MS Detection



Optimizing P&T for 8260

- Purge
- Dry Purge
- Desorb Preheat
- Desorb
- Bake
- Traps

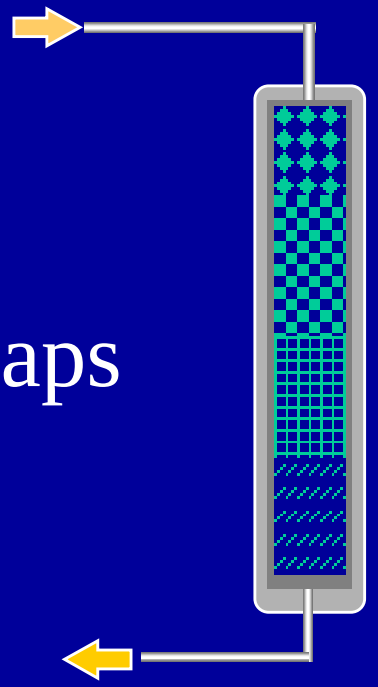
Purge



- EPA SW-846 Suggests 11 min. purge @ 40m/min
  - Heated purge allows less purge time
  - Always purge at/or below 40ml/min.

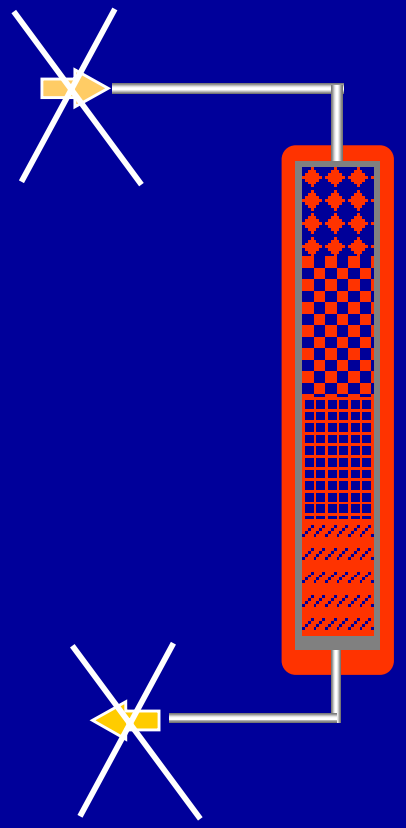
Dry Purge

- Avoid using if possible
  - Does not work with hydrophilic traps
  - Broadens the gases
  - Increases P&T Cycle Time



Desorb Preheat

- Trap is heated without flow.
- Typical temp: 5^{o} below desorb temp.
- Minimizes retention on trap.



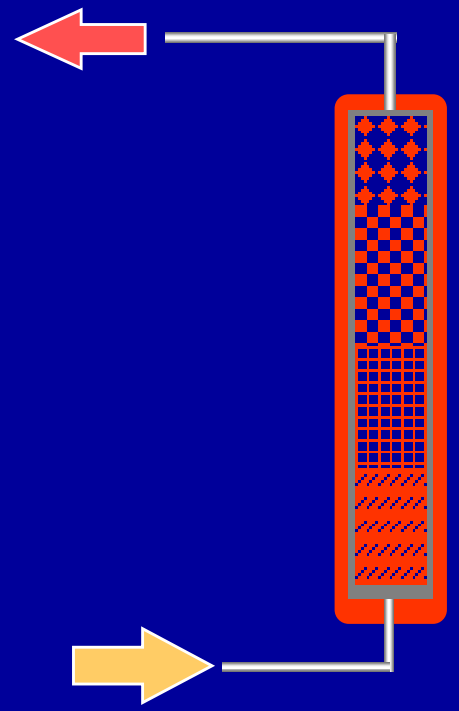
Desorb

- Trap is backflushed into column.
- Typical time: 2-4min.

  O.I. ~1min.

  Tekmar ~ 2 min.
- Typical flow: 10-80 mL/min.

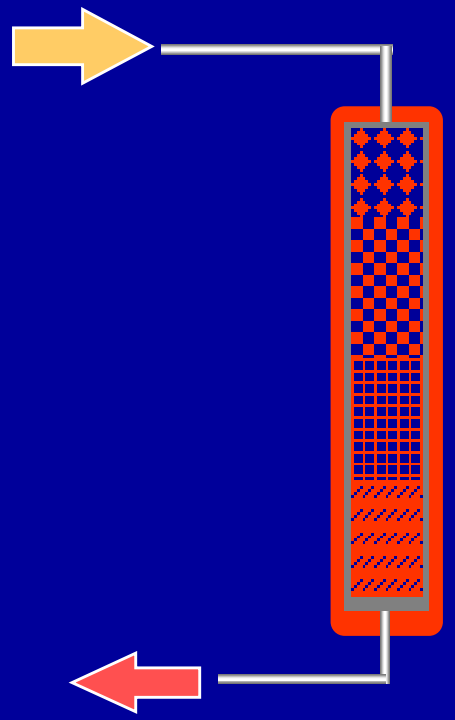
  > 20ml/min best performance
- Typical temp: 180^{o} - 250^{o}C



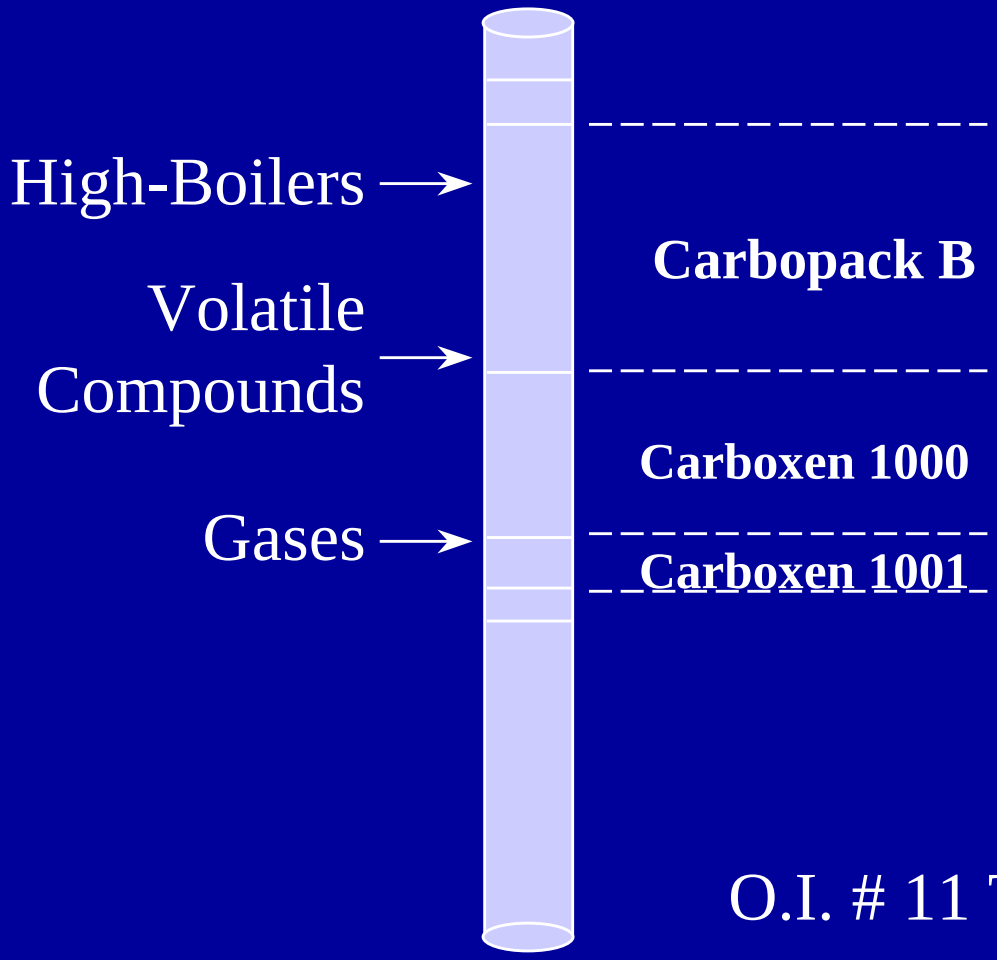
Bake

- Trap is baked clean with flow
- Typical time: 8 minutes

  Adjust bake using Naphthalene
- Typical temp: 5-10 above desorb
- Do not overhead adsorbents



Vocarb® 3000 Type “K”



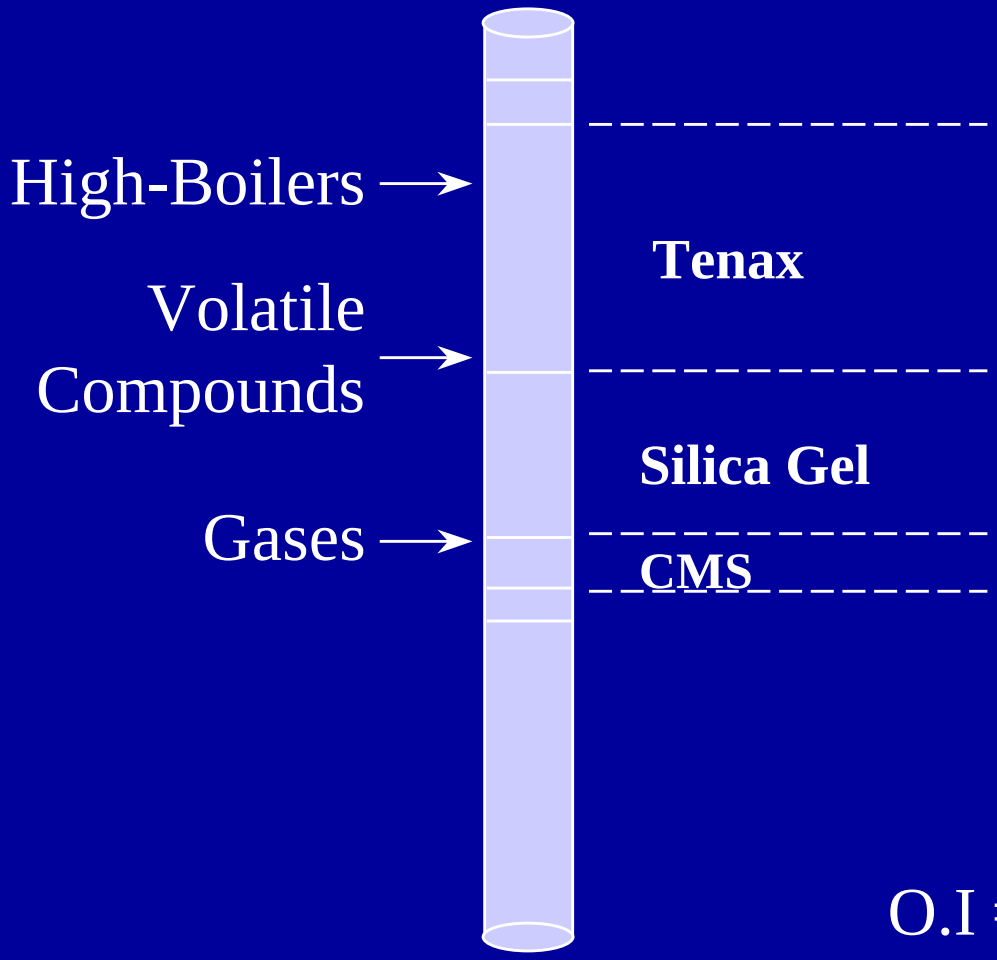
O.I. # 11 Trap

Vocarb® 3000 Type “K”

- Pros
  - Low H_20, Meoh & CO_2 retention
  - Excellent gas retention
  - Polar/Non-polars
- Cons
  - Variability from trap to trap
  - Trap breakdown/contamination

* Suggested for Tekmar Purge & Traps

Tenax®/silica gel/CMS



O.I #10 Trap

Tenax®/silica gel/CMS

- Pros
  - Excellent recoveries of polars
  - Broad range of analytes
  - Very consistent
- Cons
  - $C0_2$ retention
  - Requires use of moisture control system

* Suggested for O.I. Purge and Traps.

Common Types Of Traps

Type of Trap	Dry Purge	Temp: MAX
Tenax	Yes	220°C
Tenax\Silica Gel	No	220°C
Tenax\Silica\Charcoal	No	220°C
OV-1\Tenax\ Silica\Charcoal	No	230°C
OV-1\Tenax\Silica	No	220°C
OV-1\Tenax	Yes	220°C
Carbopack B\ Carbosieve S-III	Yes	260°C
Vocarb 4000 (4 beds)	Yes	270°C
Vocarb 3000 (3 beds)	Yes	270°C

Purge & Trap Connection Though the Injection Port

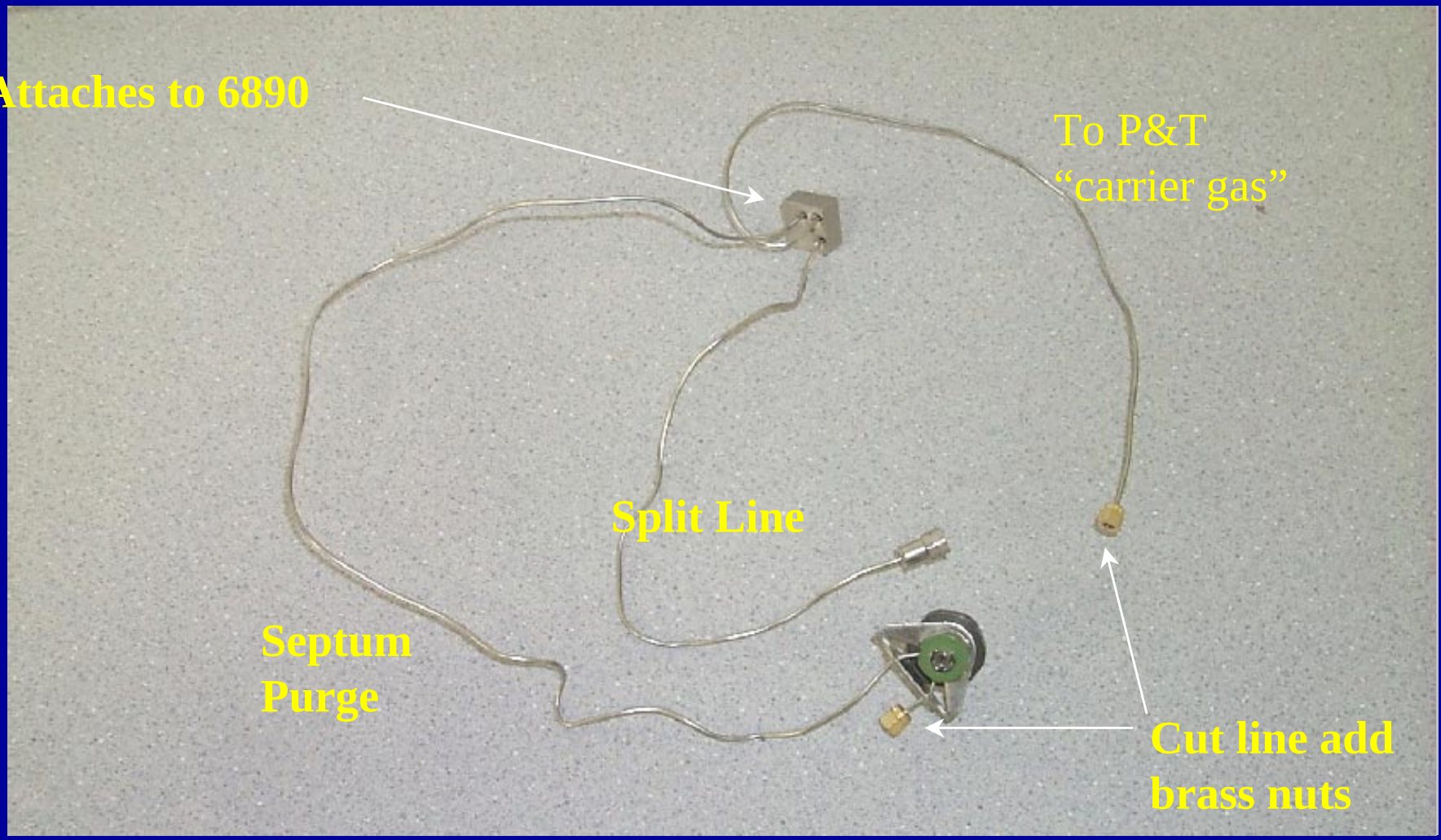
- Connect transfer line to carrier gas line on GC
- Pro: manual injections
- Con: dead volume (overcome with high flows)
- Use 1mm sleeve

Narrow Bore GC Systems

- Desorb at 10-80mL/min. and split desorb flow to decrease column flow
- Injection port splitting lowers amount of sample on column
- Purge larger volume (25mL) to increase sensitivity
- 0.18 to 0.25mm ID columns
- Improved resolution

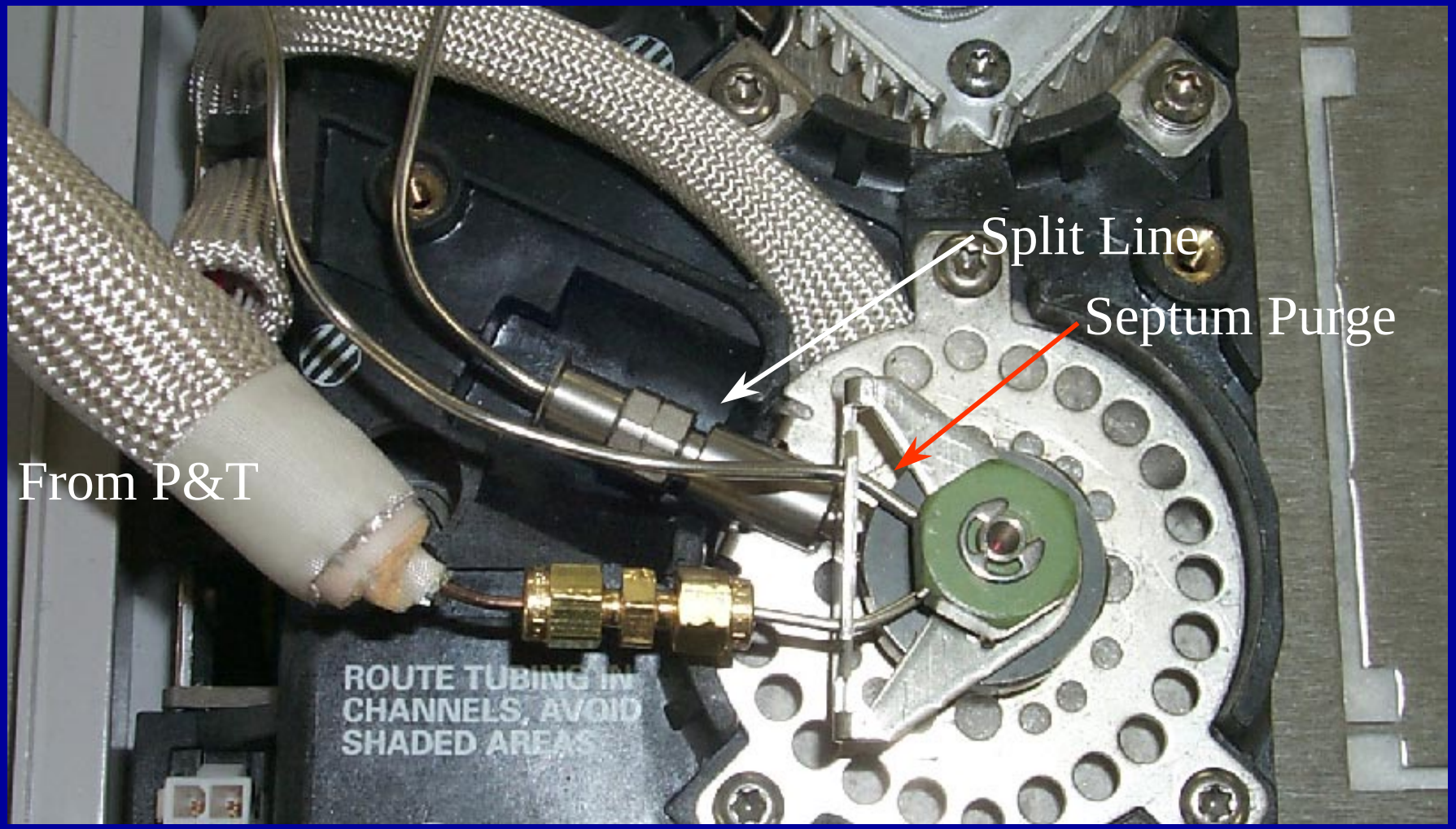
Injection Port Connection Setup

Step #1 -- cut injection port lines.



Injection Port Connection Setup

Step #2 - install lines



Injection Port Connection Setup

Step #3 -gas lines to P&T

GC/MS Detectors

- Quadrapole
- Ion Trap

Tuning GC/MS System

- Tune with PFTBA (FC 43)
- Check tune with BFB 50ng solution
- Must pass criteria

Tuning Objectives

- Maximize all abundances
- Optimize high mass
- Adjust peak width
- Adjust mass assignments

Ion Abundance Criteria:
For 4-Bromofluorobenzene (BFB)

Mass	Relative Abundance Criteria
50	15-40% of mass 95
75	30-80% of mass 95
95	Base Peak, 100% Relative Abundance
96	5-9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5-9% of mass 174
176	>95% but <101% of mass 174
177	5-9% of mass 176

GC/MS
Electron Multiplier

- After Market K&M, ETP 10X more sensitive
- Increase Signal & Noise
- Increase voltage decreases lifetime
- Use at lowest sens. to achieve DL

Compound Class & Fragmentation Ions

- Aldehydes, Amides, Amines 44,58,72,59,30
- Aliphatic Hydrocarbons 43, 57, 71, 85, 99
- Alkylbenzenes 104, 91
- Aromatics (HC) 39, 50, 51, 52, 63, 65, 76, 77, 91
- Ethers 31,45, 59, 73
- Fluorine Containing 50, 69
- Methacrylates 41, 69
- Methyl Ketones 43, 58
- Oxygen Containing 31,45,59,73
- Sulfur Containing 47,61
- Unsaturated Hydrocarbons 41, 55, 69

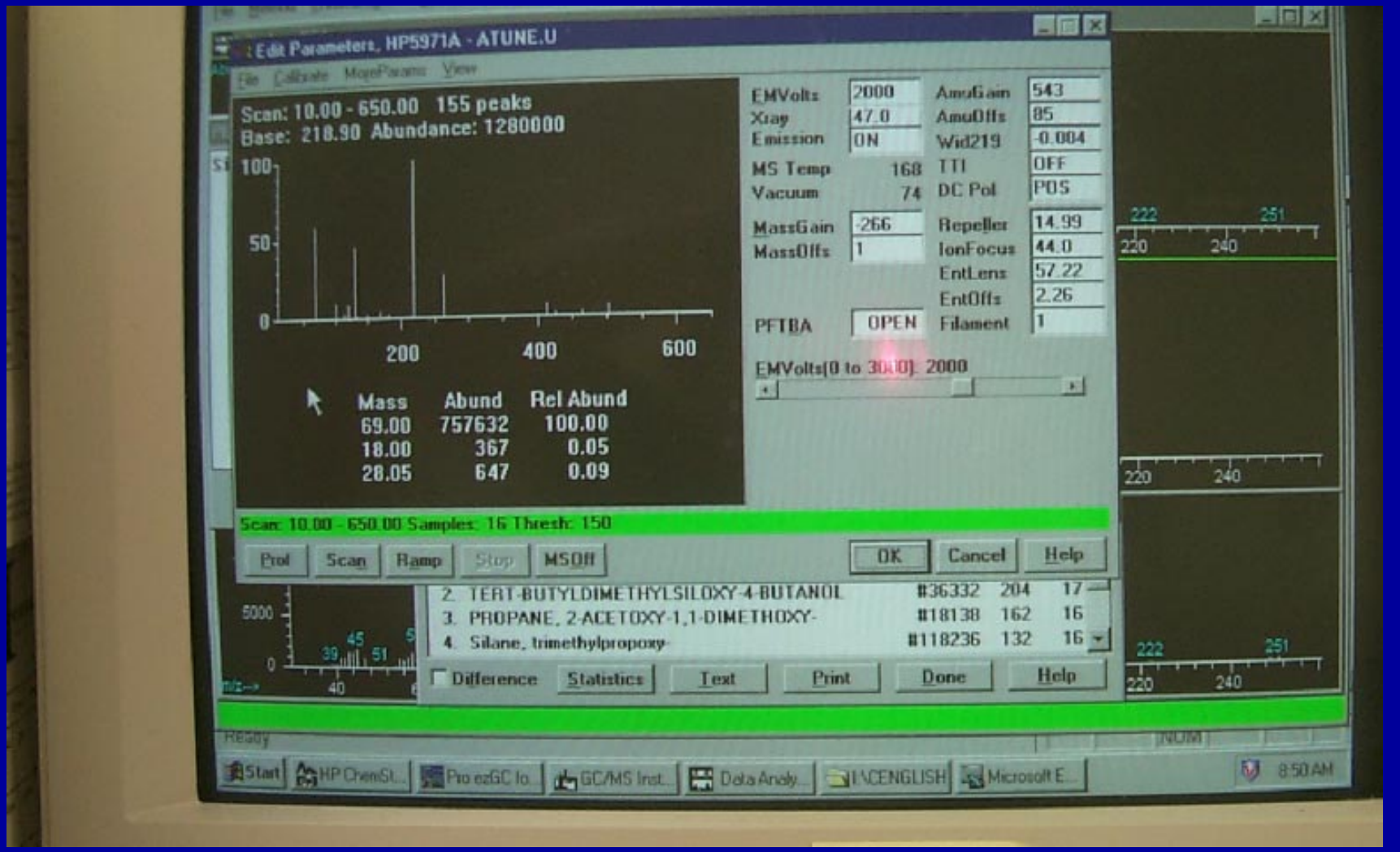
Contamination and Its Ions

- Silcon 73, 147, 207, 221, 281, 355, 429, 503
- Rough Vacuum Pump Oil 55-57, 61-67, 81-85, 95-99
- Diffusion Pump Oil 77, 115, 141, 168, 223, 260, 446
- Plasticizers 149, 223, 278

GC/MS Tips

- Use air/water scan for leaks in system
- Monitor 69 area from tune to tune
- Watch source pressure –
  - Leaks
  - Flows
  - Troubleshoot vacuum pump

Air Water Checks



Mass Spectroscopy Troubleshooting

- Leaks-
  - air/water
  - methanol & scan for mass 31
  - check source pressure

Lit. #59887 — VOA Guide

Compound-Specific Sensitivity Problems and Their Causes

- Leaks
  - Dichlorodifluoromethane
  - Chloromethane
  - Vinyl Chloride
  - Bromomethane
  - Chloroethane

Compound-Specific Sensitivity Problems and Their Causes

- Active Sites
  - Bromoform, Bromomethane
  - 2-Chloroethyl Vinyl Ether
  - Chloroethane
  - 1,1,1-Trichloroethane
  - 1,1,2,2-Tetrachloroethane
  - Ketones

Causes of Ghost Peaks

- Carryover
- Impurities in the gas supply
- Contamination
- Trap breakdown

Calibration

- Multipoint calibrations diluted in purge & trap grade methanol
- Careful with volatile loss (store standards in freezer)
- Monitor response of standards (especially gases)

Common Problems

- Water
- Reduced sensitivity
- Sample contamination (ghost peaks)
- Broad peaks and/or tailing peaks