Purge and Trap Troubleshooting: No Response, Carryover, and Contamination

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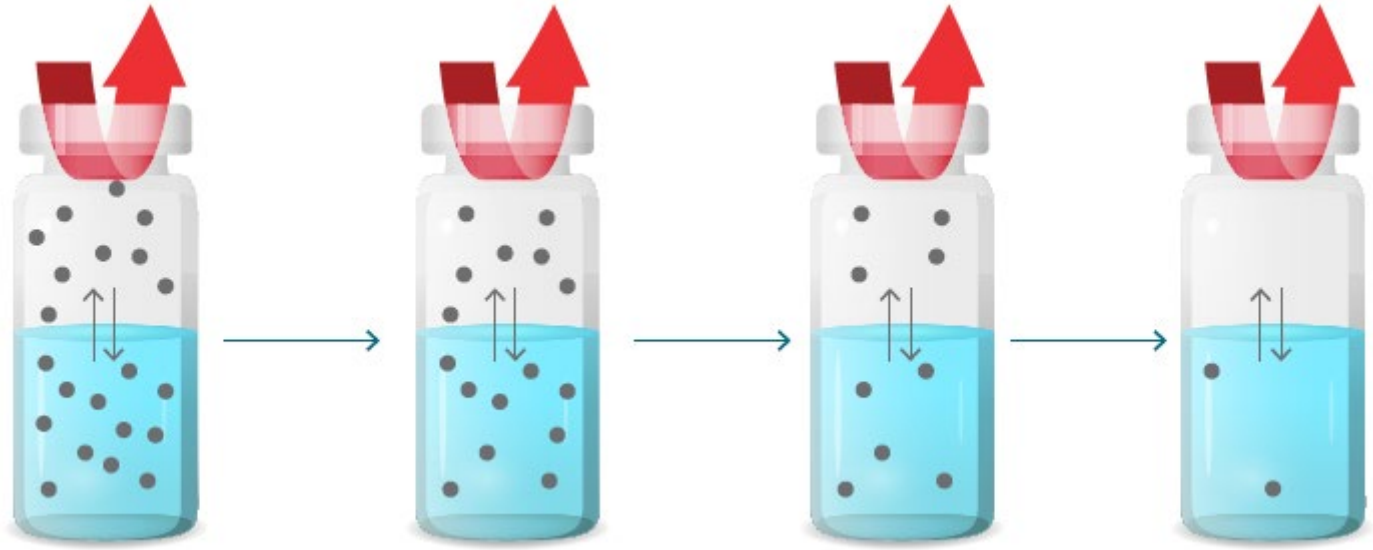
Outline of Topics

- Overview of Purge and Trap analysis
- Troubleshooting
  - No Response
  - Carryover
  - Contamination

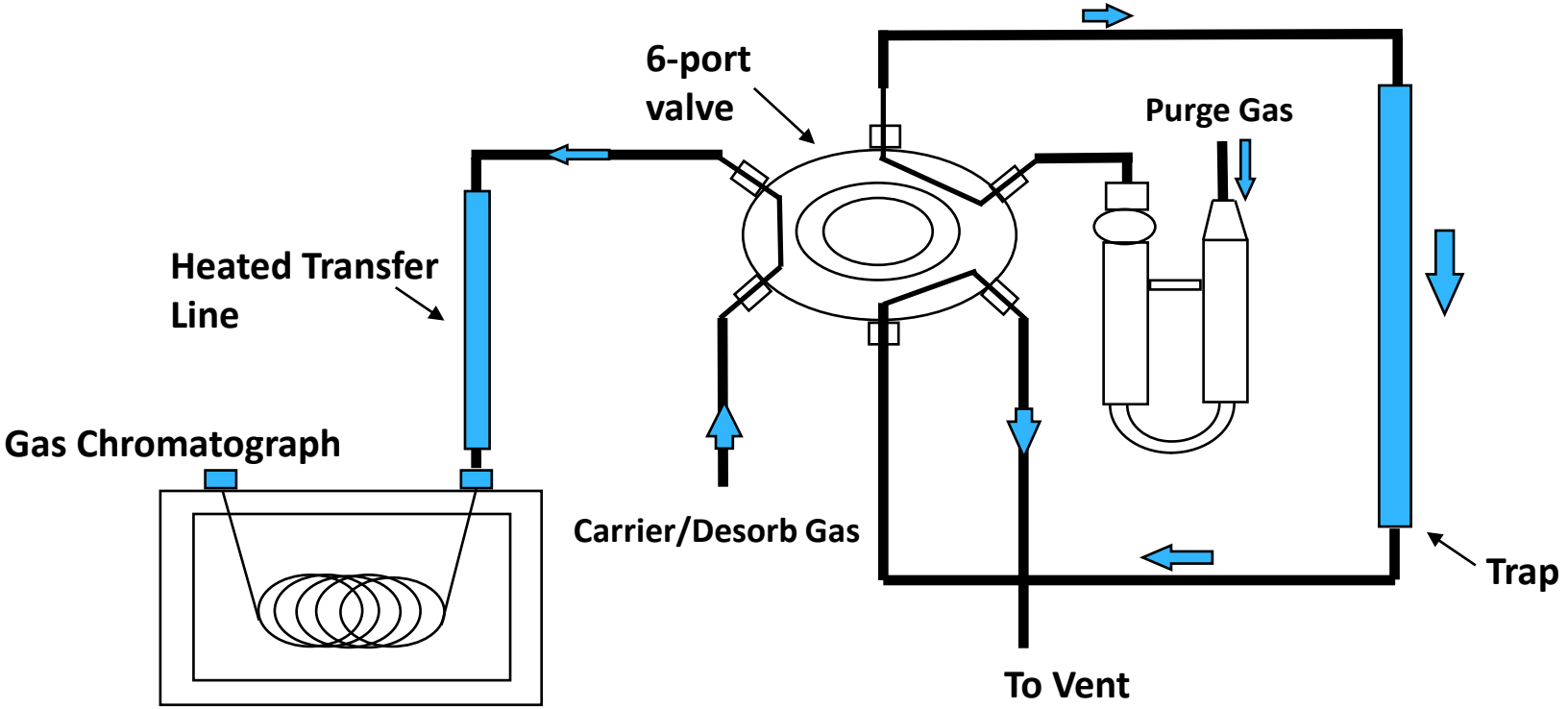
Teledyne Tekmar

- Created first commercial Purge and Trap (P&T) in 1975
- P&T Concentrators
  - Atomx XYZ
  - Lumin
  - AQUATek LVA

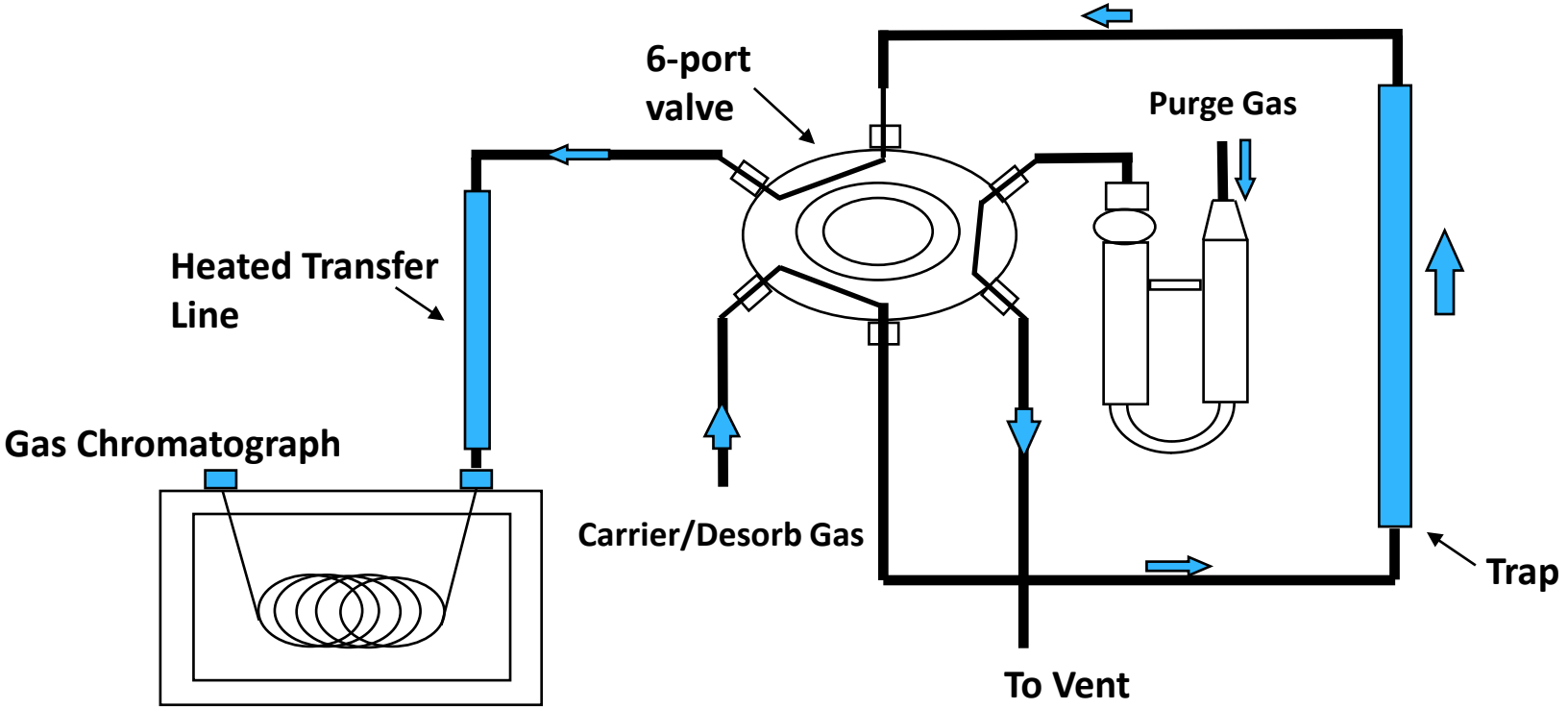
Brief Overview of Purge and Trap



Brief overview of Purge and Trap - Purge Mode



Brief Overview of Purge and Trap - Desorb Mode



Troubleshooting Tips

- Take a moment to review manuals
- Examine the facts and use valid reasoning
- Identify the “root cause” of the problem
- Avoid quick fixes and shotgun approach

BE PATIENT!

Troubleshooting – Finding the Root Cause

- MS, GC, Atomx XYZ (Concentrator and Autosampler)
  - Split the system into these 4 separate entities

Eliminate the GCMS

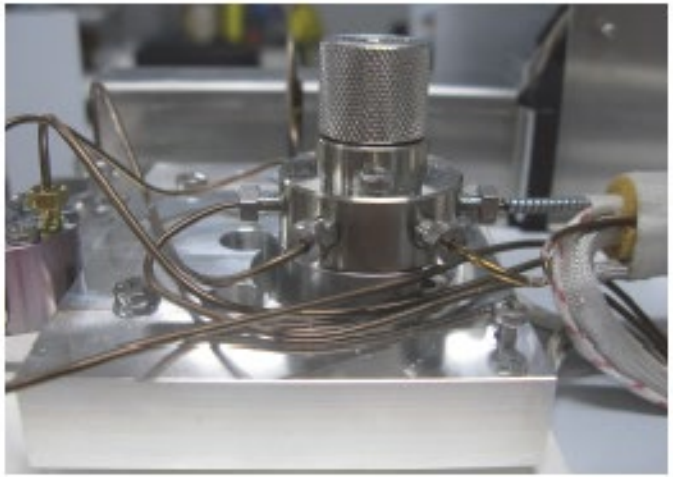
- Not necessary to disconnect P&T
- Confirm normal operation of the MS
  - Tune parameters/vacuum
  - Clean source
- Confirm normal operation of GC
  - Do direct inject of same standard used in P&T
- Poor chromatography/resolution/reduced response
  - Injection liner/septum
  - Clip column
- Once GCMS is eliminated as source of issue, proceed to P&T

Eliminate the P&T

- Confirm normal operation of the P&T
  - Run leak check
  - Make sure the correct analytical trap is in place
  - Manually load sample into P&T
    - Eliminates the autosampler

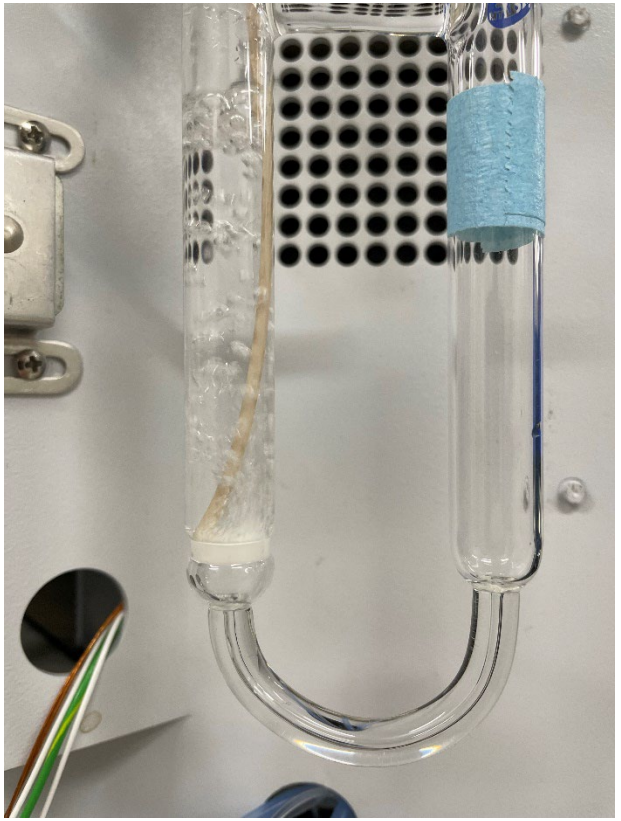
No Response

- Are the analytes getting to the GC/MS
  - Check the trap heater
    - Reaches the set temperature
  - Check 6-port valve
    - Hear the 6-port actuate
    - 6-port rotor installed correctly



No Response

- Are the analytes getting to the trap?
  - Is there a sample in the sparger and is it bubbling during purge?
    - Correct flow set in method
    - Mass flow controller working
    - System leak tight
    - Gas tank still have pressure



Carryover/Contamination

- Carryover for full target list/late eluters and contamination
- Run GC only
- Desorb only
- Check temperature
  - Desorb/Bake
- Check hot water/MeOH rinse

Carryover of Full Target List

- GC/MS
  - Faulty EPC
- P&T
  - Solenoid valve leaking cross port
  - Faulty trap heater
  - Sample not draining entirely
  - Dirty glassware
- Autosampler
  - Check soil vs. water
  - System not rinsing correctly
  - Internal standard valve leaking(allows standard to enter vessel)
  - Not transferring entire aliquot to P&T

Carryover of Late Eluters

- GC
  - Inlet too cold
- P&T
  - Not enough bake
  - Bad trap
  - Cold spots
  - Transfer line connection to GC inlet
  - Faulty condensate or trap heater
- Autosampler
  - Check soils vs. waters
  - Hot water heater/rinse failure
  - Not enough bake rinses
  - Sample needle not cleaning up
  - Not transferring bake rinse to P&T

How the Atomx XYZ Removes Contamination

- Able to rinse sample pathway with 90˚C water
- Atomx XYZ uses methanol to rinse entire pathway
  - Includes syringe, associated transfer lines and valves, sparger and 3-stage needle.
  - User can vary volume and number of rinse times user settable within the software method

Thank You!

For more information:

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