Troubleshooting Any Purge and Trap

Amy Nutter
VOC Technical Product Specialist
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TELEDYNE TEKMAR
Everywhereyoulook™

Outline of Topics

- Overview of Teledyne Tekmar and Our Products
- Troubleshooting
- TekLink Troubleshooting Tools
- Questions?

Teledyne Tekmar

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

P&T Troubleshooting

- No Response
- Carryover/Contamination
- Elution issue- early, middle, late
- Water interference
- Internal Standard recovery variability
- Linearity/Reproducibility issues
- System leak check
- Benchmark test

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, Concentrator, and Autosampler
  - Split the system into these 4 separate entities

Eliminate the GCMS

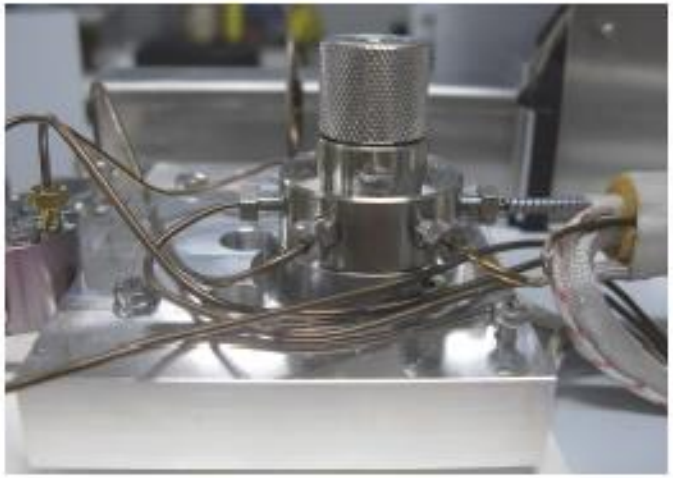
- Not necessary to disconnect P&T
- Confirm normal operation of the MS
  - Tune parameters/vacuum
  - Clean source
- Confirm normal operation of GC
  - Do direct inject of same standard used in P&T
- Poor chromatography/resolution/reduced response
  - Injection liner/septum
  - Clip column
- Once GCMS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

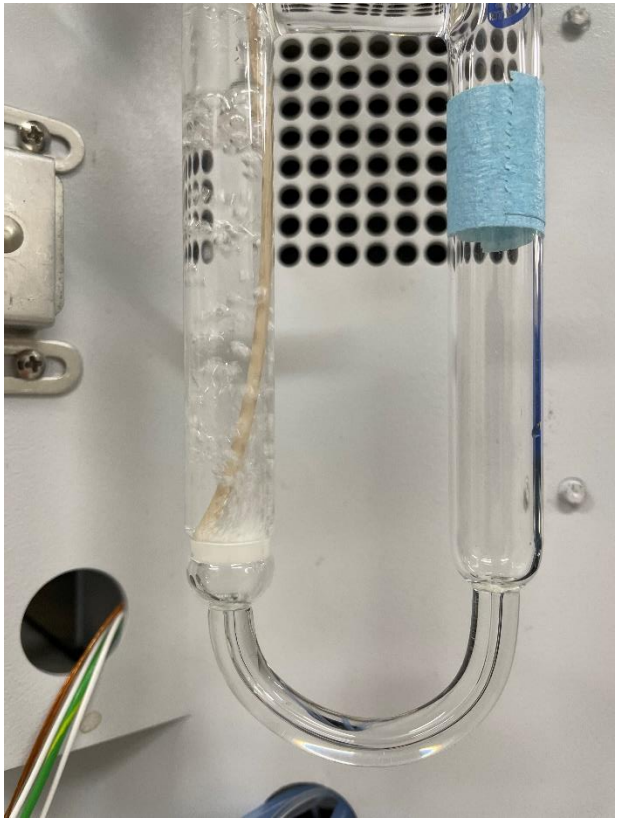
No Response

- 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

Elution Issues

- Tune
  - Auto-tune/BFB tune
- Direct inject
  - If missing peaks could be a bad EPC
  - Column
- Top of Trap
  - If missing peaks could be sample tubing/transfer line

Early Elution Groups

- GC
  - Split flow leak
  - Bad column
- P&T
  - Small leak
  - Water management
  - Too long of dry purge
  - Trap too hot (faulty electronics or heater)

Middle Elution Groups (Very Rare)

- GC
  - Column phase is bad
- P&T
  - Desorb preheat problems
    - Mass flow controller leaking
  - Bad Trap

Late Elution Groups

- GCMS
  - Source maintenance
  - Carrier flow too low
- P&T
  - Poor purge efficiency
    - Restrictive sample tubing
    - Dirty glassware
    - Sample not heated properly
    - Plugged vent valve
- Bad Trap

Water Interference

- Purge and Trap
  - Trap type
  - Sample purge temperature
  - Dry purge settings not optimized
  - Desorb time
  - Bake time and temperature

Water Interference

- GC
  - Split ratio
  - Might be time to replace column
- MS
  - Vacuum issues

Internal Standard Recoveries

- GCMS response increases as Target compound concentration increases
  - Dirty inlet liner or source
    - Validate by preparing 3 increasing target concentrations with Internal Standard concentration fixed in 1mL vials and then direct inject 1uL of the concentrations into the GC
    - If still increasing isolate between source and liner by performing cleaning and or replacement.

Internal Standard Recoveries

- P&T
  - Isolated to an active site
    - Sample tubing/analytical trap
- Autosampler
  - Hand spike vials first
  - Automated delivery system
    - Tubing restrictions
    - Valve malfunction
    - Pressure problems

Linearity and Reproducibility

- GCMS
  - Source maintenance
  - Vacuum problems
  - EPC failures
  - Bad column
  - Method not optimized
    - Oven temperature program
  - Multiplier going bad

Linearity and Reproducibility

- Purge and Trap
  - Method not optimized
  - Trap is failing
  - Drain valve leaking
  - Excess water in system
  - Temperatures not reaching set-points
  - Too much carry over
  - Sample preparation errors
    - Dirty standard syringe or glassware
    - Too much headspace
    - Analyst variability

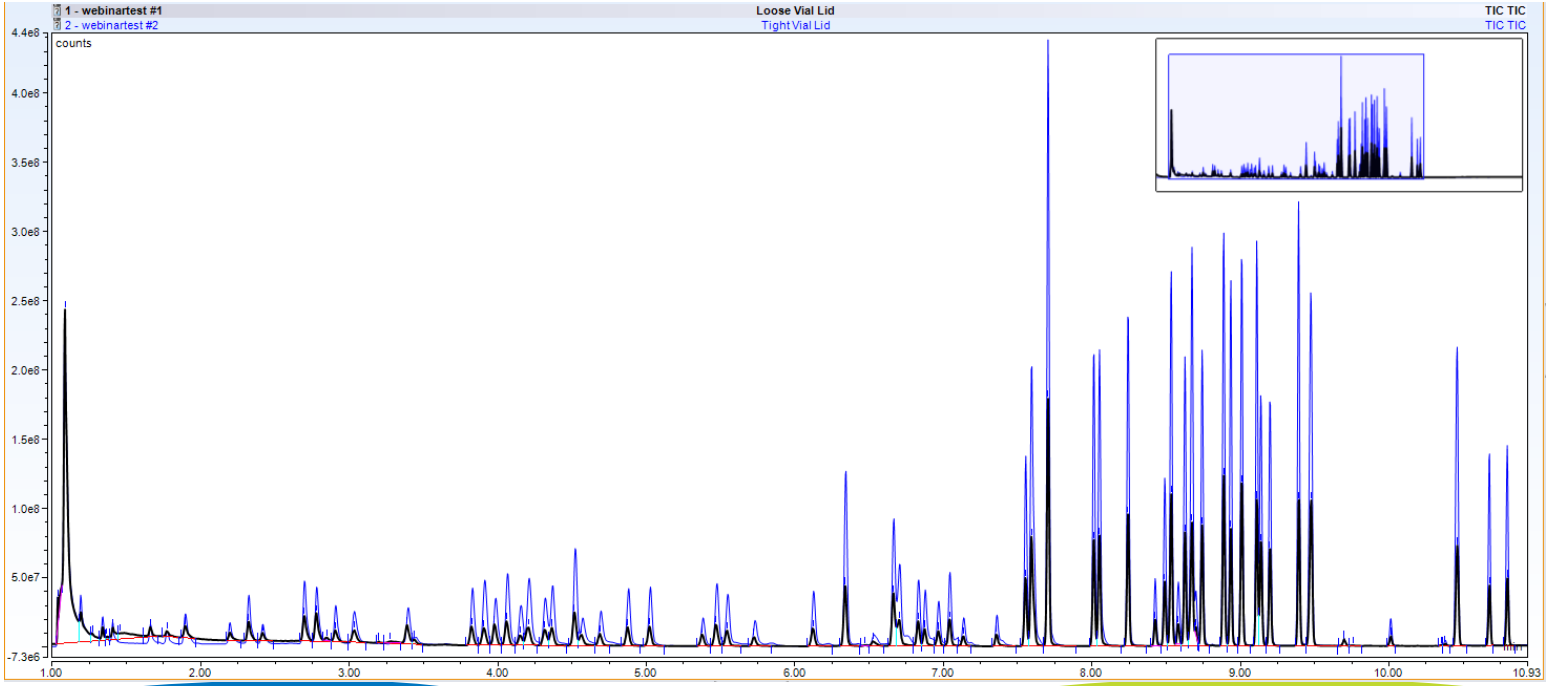
Linearity and Reproducibility

- Autosampler
  - Not pulling same amount of sample
  - Not transferring sample correctly
  - Improper rinsing between samples
  - Compare soil vs. water samples
  - Determine if IS is varying and causing everything else to look unstable

Tips to Identify Leaks Before They Become a Problem: Warning Signs

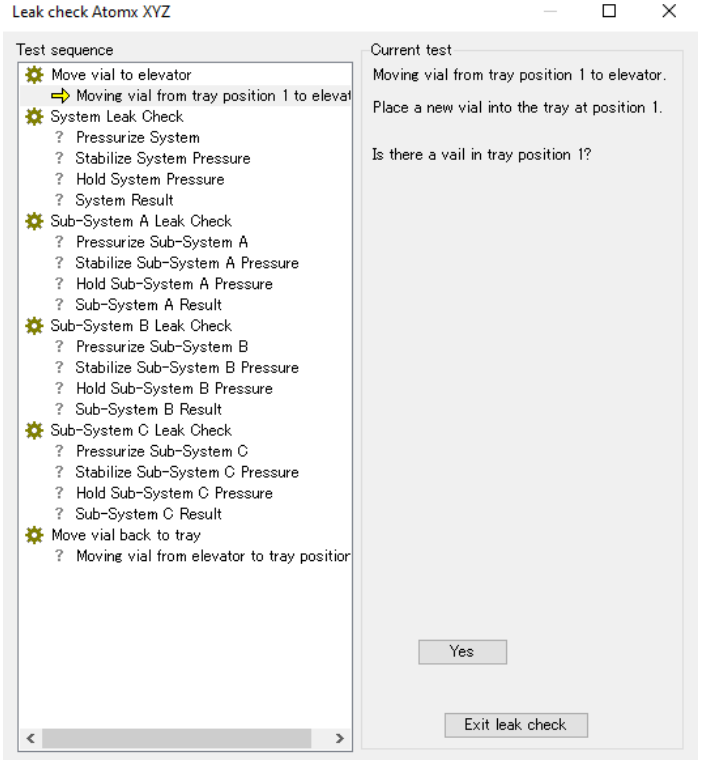
- Check History Log in Tools and review purge and bake pressures
- Front end compounds are missing/low response
- Linearity and reproducibility
- Slow draining of the sparger
- Low/No sparge bubbles
- GC beeping/alarming during desorb

Tips to Identify Leaks Before They Become a Problem: Warning Signs



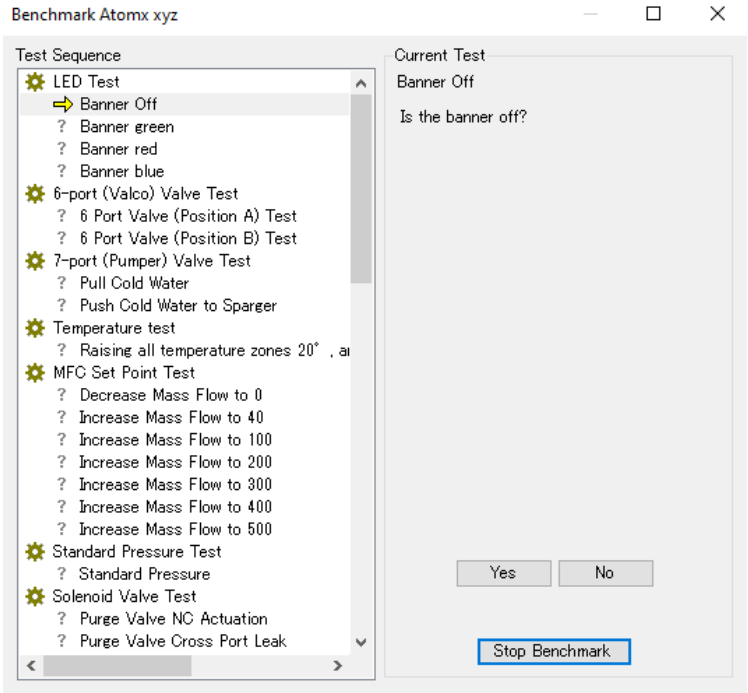
TekLink™ Leak Check Diagnostic Tool

- Perform a Leak check:
  - after changing the IS, foam eliminator, any glassware, trap, and/or any fittings
  - after turning the system back on after being off for a while
  - before performing a benchmark test



Benchmark

- Make sure system is leak tight first
- Checks all system controls including solenoid valves, heaters, flows, and 6-port valve
- Helps isolate a variety of potential issues



Thank You!

For more information:

Website: **www.teledynetekmar.com**
Phone: 513-229-7017 or 513-896-0702
Email: Amy.Nutter@Teledyne.com

Check out our website for all new applications!
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