Purge and Trap Workflows: Tips to Overcome Challenges That Can Arise From Sample Collection to Sample Analysis

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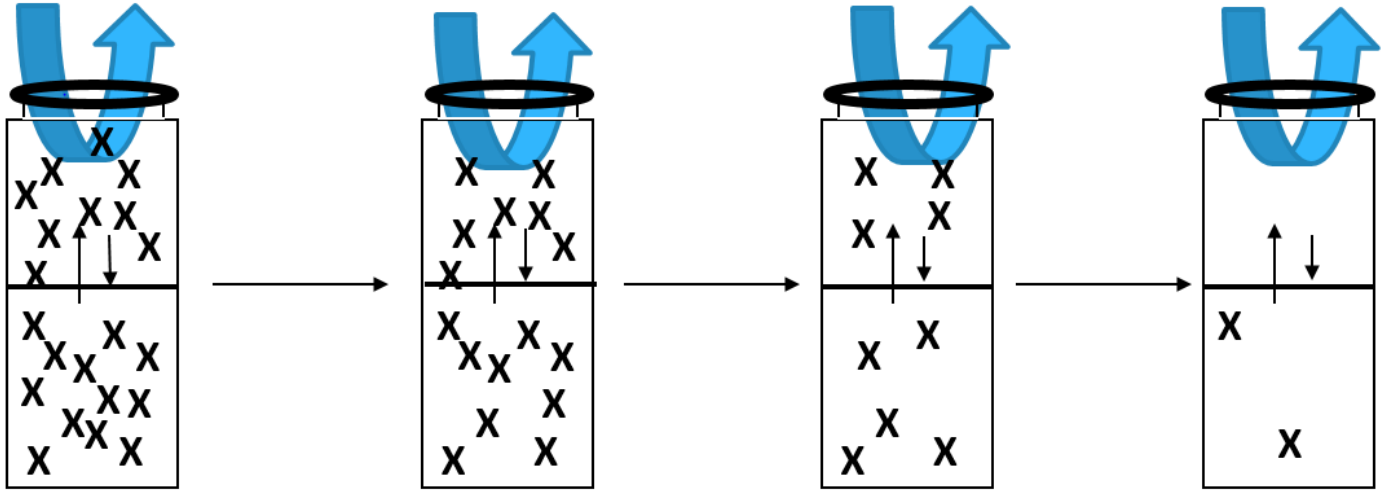
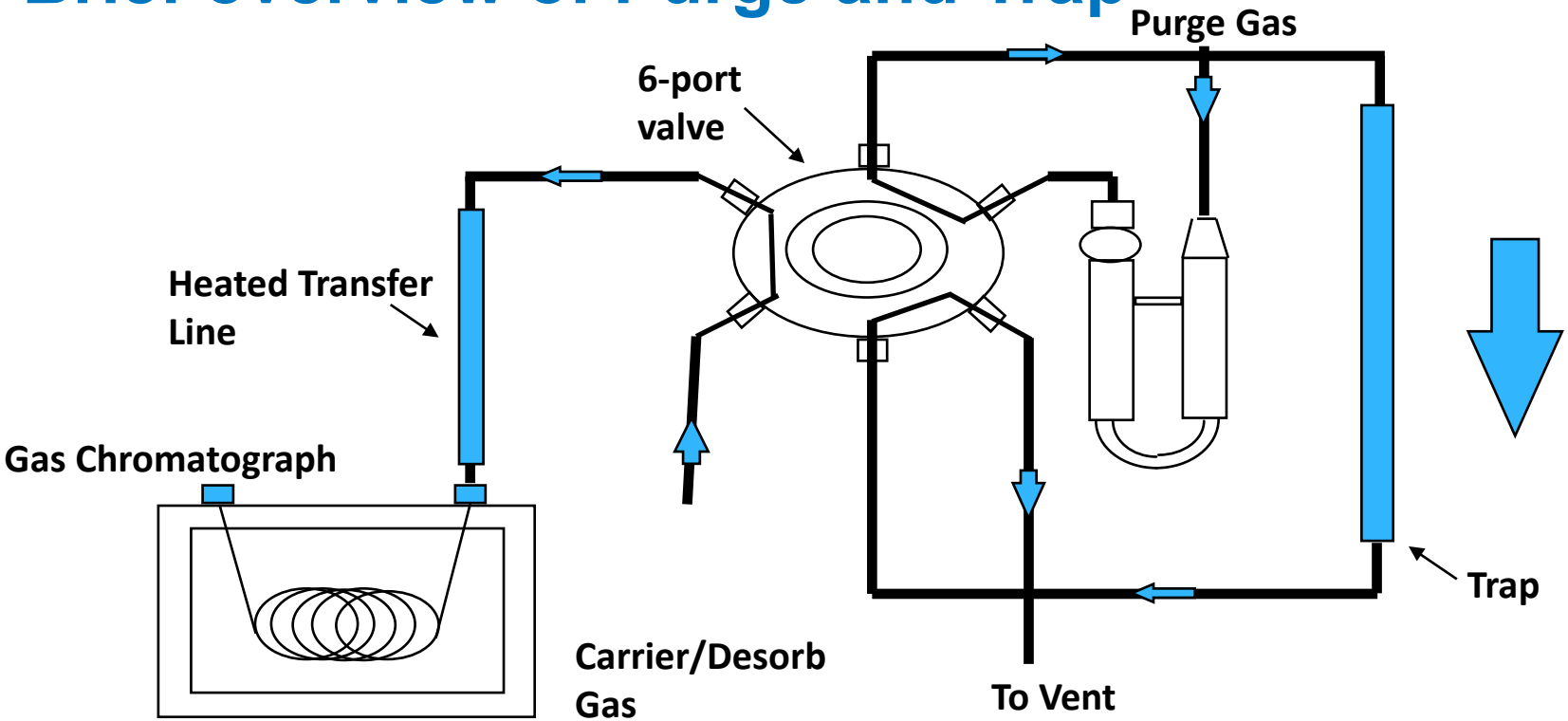
Outline of Topics

- Overview of Purge & Trap Technique and Tekmar Products
- Common Method Types and their Challenges
- How to Select Best Equipment for Method
- Consumables Considerations
- Tying it all Together

Brief overview of Purge and Trap



Brief overview of Purge and Trap



Introduction of Product Line

- Purge and Trap Concentrators
  - Atomx XYZ
  - Lumin
  - AQUATek LVA

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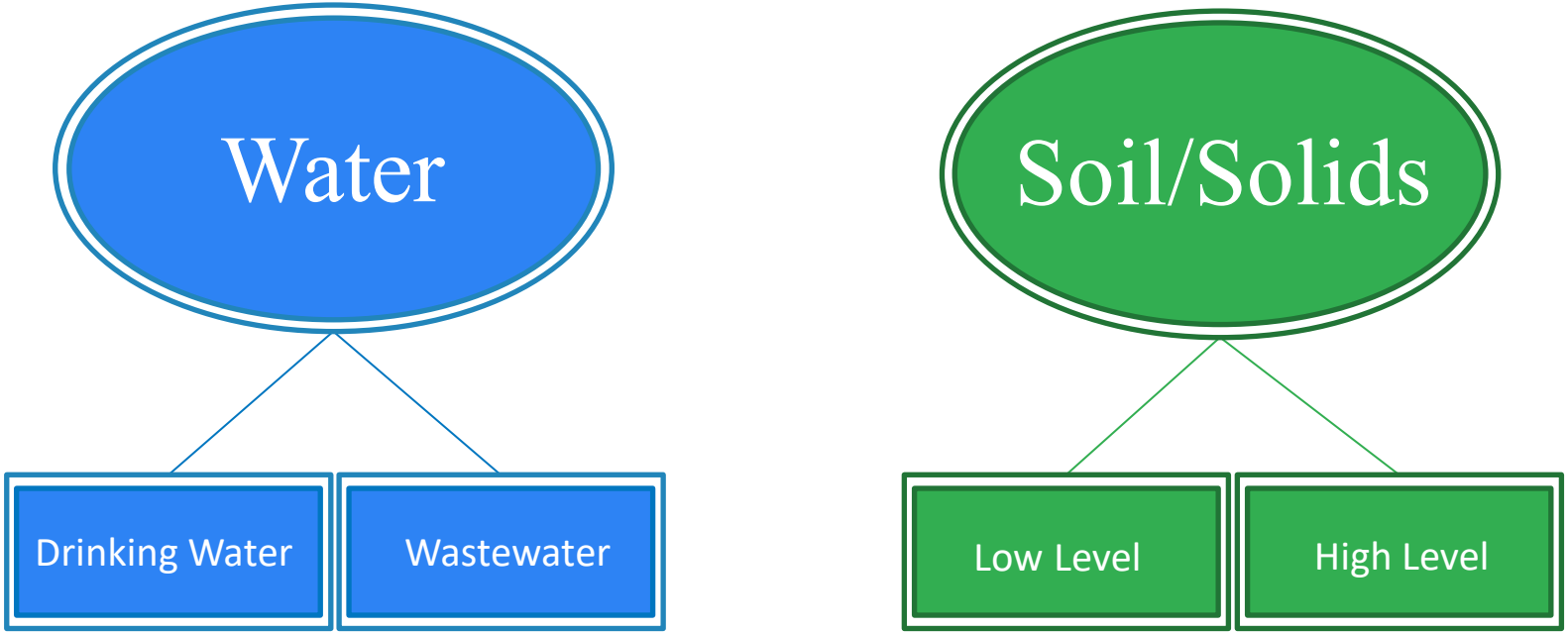
Basic Method Types



Regulated Methods

- Globally, P&T usage is largely driven by regulated methods, all of which present unique challenges to end users
  - Sampling techniques and container considerations
  - Often, instrument conditions tightly controlled
    - Purge rates
    - Tuning requirements

What are the Challenges?

- Vial cleanliness
  - Contamination from vials can be frustrating to identify
    - All septa contribute siloxane peaks, worse in heated, in-vial purging
    - Target compounds are of a greater concern, can lead to failure of calibration and/or false positives
  - Solution
    - Clean vials with soap and water, then bake before using
    - **Purchase certified pre-cleaned vials**
    - Keep sample heating to the minimum needed for low-level sensitivity

What are the Challenges?

- Cold preservation of samples
  - Once collected, must be kept on ice – makes shipments heavy
  - Once received into lab, must be kept in refrigerator until time of analysis
    - Some methods (EPA 524.3) require sample to be chilled on the autosampler as well.

What are the Challenges?

- Regulated parameters may not be the most efficient
  - USEPA (and China) method parameters are notorious for long run times and introducing significant amounts of water to GC
  - USEPA also regulates MSD parameters, often disallowing improvements in sensitivity by requiring BFB tune, not allowing SIM

What are the Challenges?

EPA 524.2

Parameter	Time (min)
Purge	11
desorb	4
Bake	4
Total	**19!**

EPA 8260

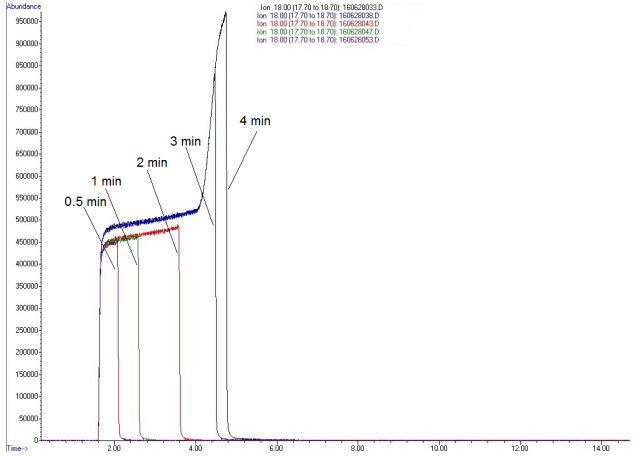
Parameter	Time (min)
Purge	11
desorb	2
Bake	4
Total	**17!**

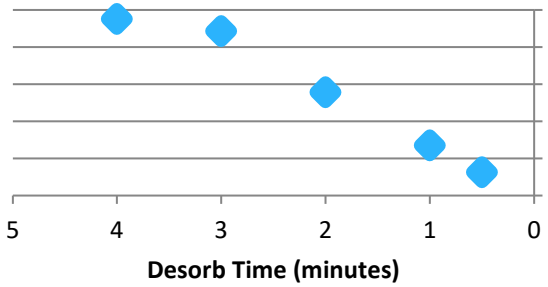
EPA 524.3

Parameter	Time (min)
Purge	5 to 11
desorb	0.5 to 2
Bake	2
Total	**7.5 to 15!**

What are the Challenges?

- Methods introduce large amounts of water to GC





Desorb Time (min)	% of 4 min
4	100
3	93
2	58
1	28
0.5	13

Method-specific Challenges

- Each method, or group of methods has their own collection of challenges.
  - Drinking water – low level sensitivity
  - Wastewater – carryover and dynamic range
  - Specialty methods
    - Mold/Odor analysis – purge efficiency
    - 1,4-dioxane – low level sensitivity

Drinking Water Method Challenges

- Low level sensitivity
  - As source, column, trap and sparger become dirty, low-end sensitivity becomes an issue
  - Moisture control is particularly important to controlling this
  - Cleanliness of purge gas, rinse water and glassware associated with standards prep
  - Sparger and trap maintenance

Wastewater Method Challenges

- Carryover can cause false positives or require additional rinses or blanks – increase run time
- Source, column, trap and sparger maintenance
- Active sites in gas pathway
  - Activity in tubing, trap and MCS can all contribute to poor calibrations and loss of compounds
  - Clean and/or replace pathway

Specialty Methods Challenges

- Mold/Odor
  - Method parameters often require optimization
    - Application note is a good starting point, not always workable solution
    - Can benefit from use of a sparge vessel heater
  - Compounds are large molecules, tend to foul traps and cause backpressure issues
    - Recommend #1A trap, large mesh tenax offers decent sensitivity while lasting longer than other options

Specialty Methods Challenges

- 1,4-dioxane
  - Not very volatile, shows poor purging characteristics
  - Often requires large sample volumes (25ml sparger)
  - SIM scanning can be helpful in improving sensitivity
  - Application can benefit from use of sparge vessel heater
  - K or #9 trap are sufficient for this analysis

What are the Solutions?

- Improvements in Tekmar hardware have allowed some improvements in time
  - Faster trap cool down from bake to purge ready
  - Faster trap heating shortens time to reach desorb temp.
  - Improvements in moisture management reduce or eliminate need for dry purge

What are the Solutions?

- Moisture management is controlled through hardware and application parameters
  - Improved moisture control fitting in Lumin and Atomx XYZ
  - Shorter, narrow bore GC columns allow for higher split ratios, reducing moisture introduced
  - Shorter desorb times reduce water introduced

Packages can be Broken into Key Methods

- Drinking Water
  - Sparger purge
    - Need chilling
    - Don’t need chilling
  - In-vial purge
- Wastewater/soils
  - Need in-vial purge capabilities
  - Do not need in-vial purge
    - Need dilutions or MeOH extraction
    - With or without antifoam
    - No need for dilutions or MeOH extraction
      - With or without antifoam

Drinking Water Packages

- Lumin and AQUATek LVA
  - Offers chiller standard, 84 vial positions, fixed sample loop (available in multiple sizes), same concentrator tech found in Atomx XYZ
  - K (vocarb 3000) or #9 trap

Drinking Water Packages

- Atomx XYZ
  - Typically, these users are not interested in dilutions, MeOH rinse or extractions
  - K (vocarb 3000) or #9 trap

Wastewater Packages

- Atomx XYZ
  - Offers dilutions, soil purge, MeOH extraction and rinsing all in base model
  - Top model add foam eliminator and chiller
  - K (vocarb 3000) or #9 trap
  - Consider adding **fritless sparger** if foam is a concern

Wastewater Packages

- Sparger Purge
  - Lumin and AQUATek LVA
    - Good for customers who do not analyze soils, and do not need dilutions capability.
    - If foam is a concern, foam eliminator model of Lumin and/or **fritless sparger**
    - K (vocarb 3000) or #9 trap

Thank You!

For more information:

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