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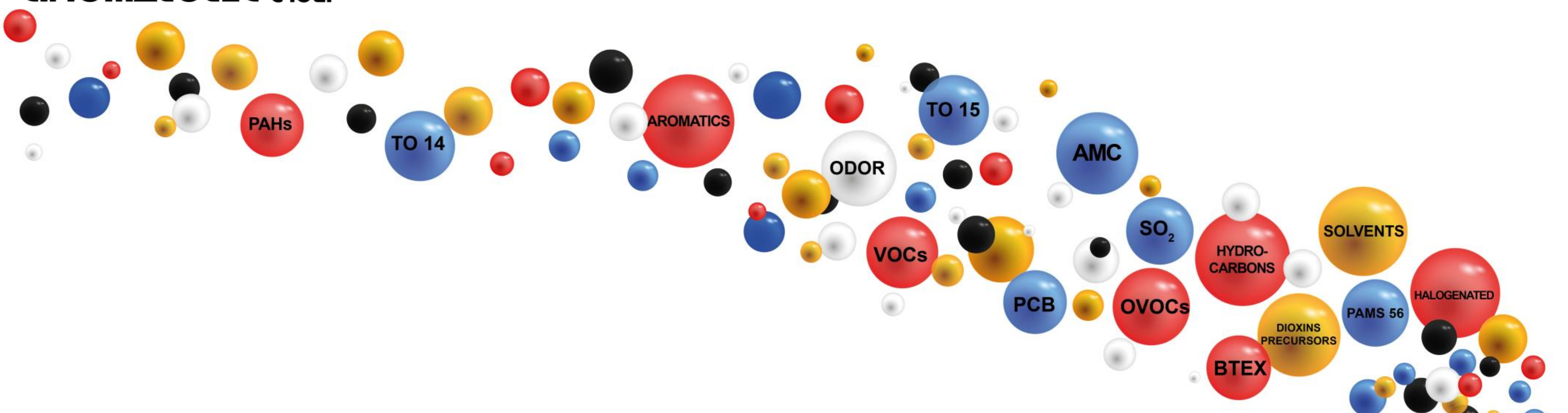

Calibration Using Permeation Tube Technology

Omar Guerra

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Calibration Using Permeation Tube Technology

- How permeation tubes work
  - Constant temperature
    - Tubes go in a vessel, which goes inside a heating block

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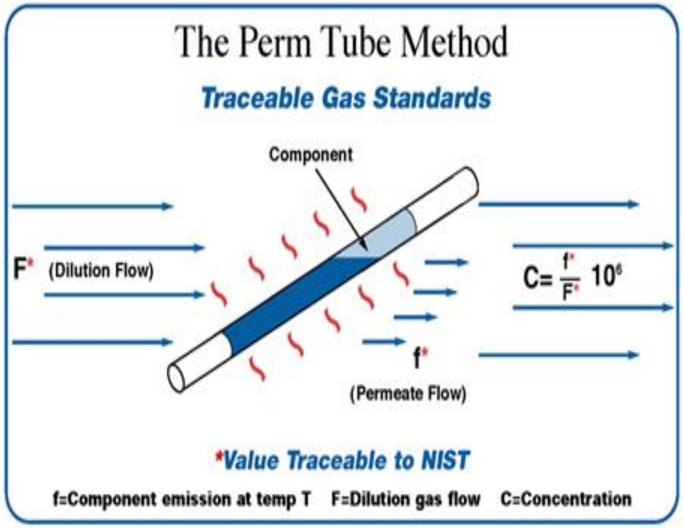
- Liquid-filled tube releases in a small but consistent emission of compound vapor
- Permeation membrane is made of Teflon



- Permeation tube emission rates and types
  - Factors effecting tube emission rate
    - Permeability of its chemical analyte through the membrane
      - Temperature
      - Molecular weight
      - Vapor pressure

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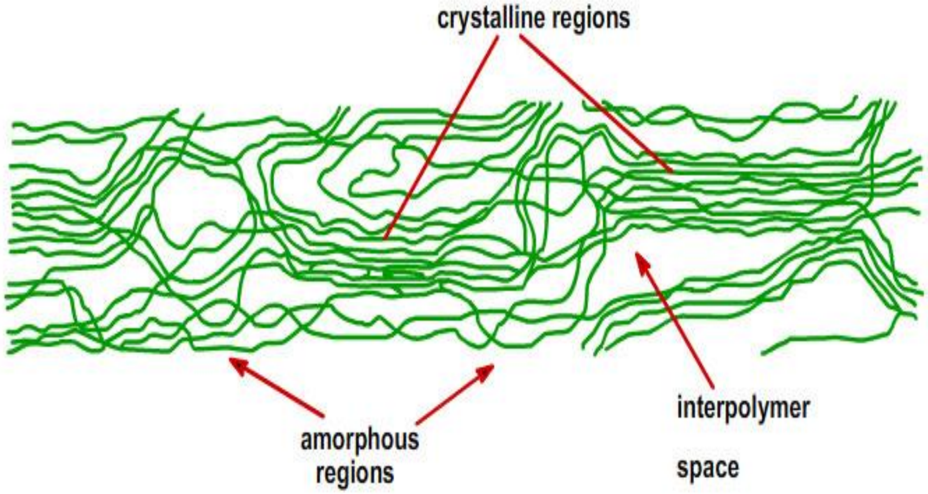
- Physical characteristics of its permeable membrane.



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- Permeation tube emission rate is determined by weight loss over time
  - Tube is weighed periodically and then assigned an emission rate
- Certification time frame determined by emission rate
  - The lower the emission rate, the longer it takes to get the tube

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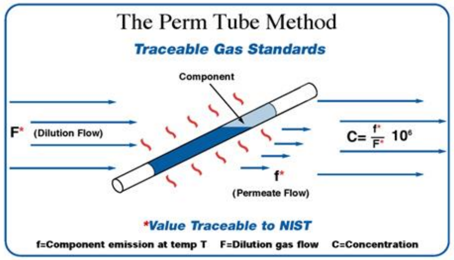
- How to generate a gas standard
  - Tube with a given emission rate
  - Must be heated to required certification temperature (varies by compound)
  - Inert dilution gas flow sweeps the compound out of the chamber
    - The lower the flow, the higher concentration and vice versa



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- Concentration calculation
  - **ppmv:** $C = (E * K_o) / F$
    - **Where:**
    - **C** = desired concentration in ppmv.
    - **E** = certified emission rate (ng/min) as reported on your tube certificate.
    - $\mathbf{K_o}$ = 22.4 (molar volume of gas)/compound molecular weight
      - value that corresponds to the compound-specific K_o on permeation tube certificates. Proportionality constant that is used to convert emission rates from a volume (nL/min) to a weight (ng/min).
    - **F** = rate of dilution flow you are using (cc/min)

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- Example: What is the concentration produced by an H2S tube that generates 1000 ng/min in a carrier gas flow of 500 cc/min?
  - **C** = ?
  - **E** = 1000 ng/min
  - **K_o** = 0.66
    - -value that corresponds to the compound-specific K_o on permeation tube certificates. Proportionality constant that is used to convert emission rates from a volume (nL/min) to a weight (ng/min).
    - **F** = 500
    - C = (1000 * 0.66) / 500
    - **C = 1.32 ppmv**

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- Calibrating an analyzer (based on procedures as set forth be KIN-TEK Analytical, Inc.)
  - Set up your gas calibration standards generator in close proximity to the gas analyzer
  - Connect carrier/diluton gas cylinder of N2 or Zero Air to the generator
    - Dilution gas in port
  - Connect the analyzer and the gas standards generator using appropriate hardware
    - Vent line to appropriate fume hood or other exhaust
    - Span gas out port of the generator tubing to the sample in port of the gas analyzer
    - Power supply

Calibration Using Permeation Tube Technology

- Calibrating an analyzer (based on procedures as set forth be KIN-TEK Analytical, Inc.)
  - Generate calibration gas
    - Power on the gas standards generator
    - Set a minimum flow before inserting the permeation tube
    - Using proper personal protective equipment, insert proper permeation tube (e.g. $H2S$) into the oven chamber of the generator
      - If the permeation tube is in the oven chamber, there must always be carrier gas flowing
        - » Compound may build up and damage critical components otherwise

Calibration Using Permeation Tube Technology

- Calibrating an analyzer (based on procedures as set forth be KIN-TEK Analytical, Inc.)
  - Generate calibration gas
    - Set the instrument to the proper operation temperature as indicated by the permeation tube certificate.
      - Equilibrate the oven chamber and tube to the certification temperature
        - » This typically takes 45 minutes, but can take 12 hours and in the case of a brand-new formaldehyde tube, 24-48 hours is recommended
    - Calculate the flow rate needed for the concentration needed to calibrate your analyzer

Calibration Using Permeation Tube Technology

- Calibrating an analyzer (based on procedures as set forth be KIN-TEK Analytical, Inc.)
  - Generate calibration gas
    - Set the instrument flow based on the concentration calculation results
      - Automated systems are available such as the FlexStream™ Base by KIN-TEK Analytical located in La Marque, TX, USA.
      - Manually-operated systems are available, as well
    - Place instrument in standby or other similar mode
      - This allows the instrument to remain ready for producing calibration gas with a minimum of carrier gas usage

Calibration Using Permeation Tube Technology

- Calibrating an analyzer (based on procedures as set forth be KIN-TEK Analytical, Inc.)
  - Set your analyzer to calibration mode
  - Set the gas standards generator to Span mode
    - This mode is used to create the calibration gas

Calibration Using Permeation Tube Technology

- Calibrating an analyzer (based on procedures as set forth be KIN-TEK Analytical, Inc.)
  - Feed the calibration gas to the analyzer and run the analyzer calibration procedure as per operating instructions
  - Many concentrations levels can be created by adjusting the flow rate of the gas standards generator

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- Advantages of Permeation Tube Technology over Gas Cylinders
  - Dynamic standards – Made fresh, not pre-made and allowed to sit over time

- Advantages of Permeation Tube Technology over Gas Cylinders
  - Safety:
    - Gas cylinders are heavy, cumbersome to move, and present a safety hazard because of the heavy weight and lack of mobility
    - Permeation tubes are the size of a pencil, contain only a small amount of toxic compound (<2 mL), cylinders have much more
    - Permeation tubes are rugged and can be run over by a car

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- Advantages of Permeation Tube Technology over Gas Cylinders
  - Cylinders are under an immense amount of pressure
  - Permeation tubes have no valves that can potentially fail or break off
  - No special equipment required for transportation
  - Most permeation tubes can be shipped world wide as non-hazardous goods because of the small amount of compound in the tube

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- Advantages of Permeation Tube Technology over Gas Cylinders
  - Cost effective:
    - One tube can often replace 10 cylinders
  - Permeation tubes can typically deliver concentrations over a range of 20:1 using primary dilution and up to 10,000:1 with secondary dilution

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- Advantages of Permeation Tube Technology over Gas Cylinders
  - Gas cylinders are often inaccurate or unreliable at low level concentrations (low ppm to ppb)
    - In low level concentration cylinders, trace components can adsorb to the walls of the cylinder
    - In low concentration cylinders, the trace components can also react with the metal walls of the cylinder causing inaccuracy and ultimately, unreliability