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Quantitative Moisture Measurements for Limited Gas Volumes

Adam Pimentel, Curtis Mowry
Sandia National Laboratories

U.S. DEPARTMENT OF ENERGY | NNSA National Nuclear Security Administration

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Intro

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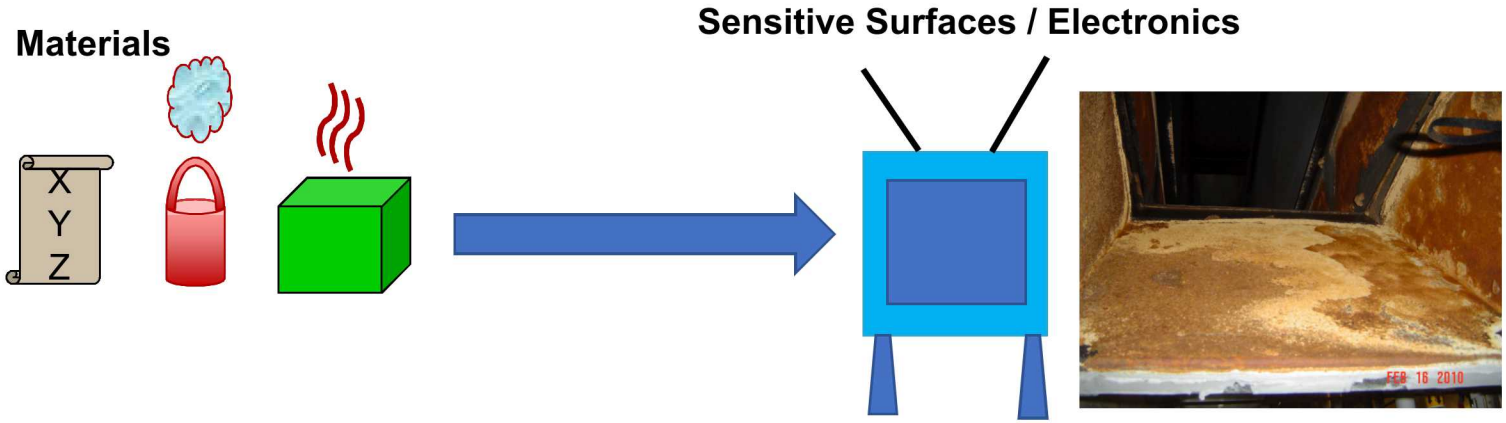
- Why measure moisture
  - Humidity bad for electronics
  - Material and surface incompatibility of water
  - Instrumentation incompatibility with water
- How we measure moisture
  - COTS GC/PDID
  - PLOT or Bonded PLOT phase columns to separate water from non-retained
  - Have observed on DB-5

Water is everywhere!

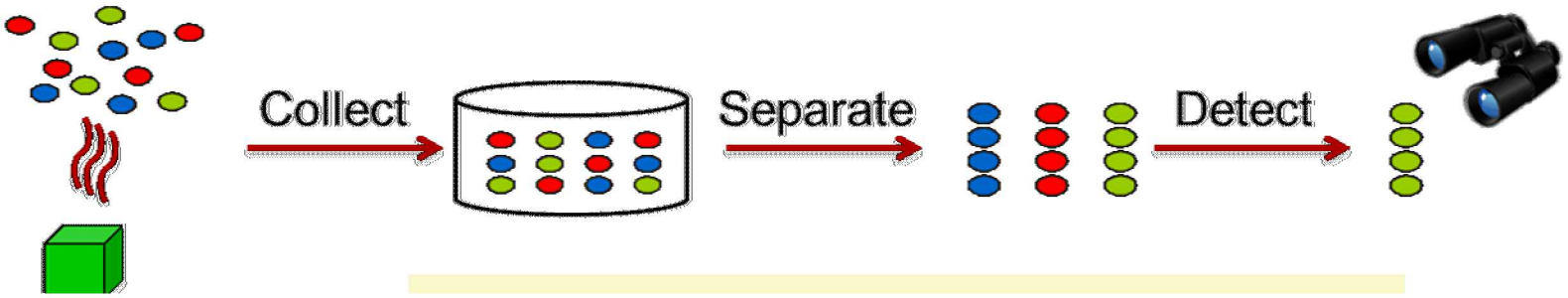


Unexpected, unmitigated water contamination can cause equipment failure.

outgassing

- Follow analytical chemistry trident

Sample preparation	Separation	Detection
Extraction Digestion Purge & trap Cryo-focus Preconcentration Thermal extraction Derivatization	Gas, Liquid or Ion chromatography thermal permeation	Electrochemical Spectroscopy (emission, UV/VIS) Mass spectrometry (QQQ, MS^n) FID, TCD, FPD, PDID, ECD

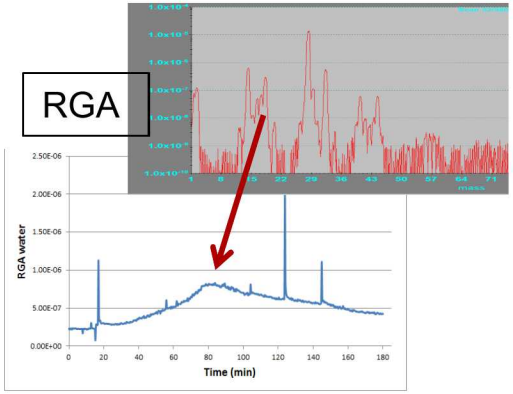


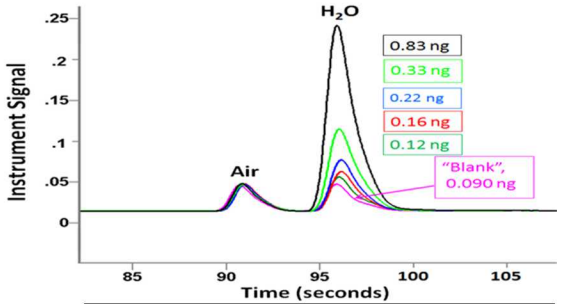
Current Tech for Water in Gases

- Applications: outgassing, controlled environments
- Residual Gas Analyzers (RGA) or RH solid state sensor(s)

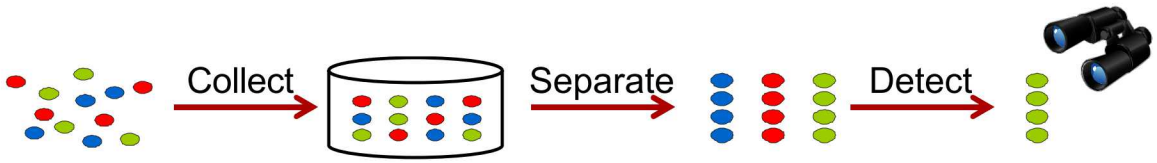
Chilled Mirror

Cons:
Lack of sensitivity
Adsorption
Background
Calibration time consuming
Affected by flow
Large gas volume
Long equilibration





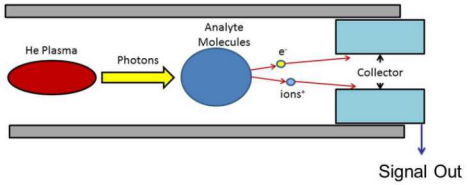
Hard to get "Blank" blanks.



Our hypothesis

- PDID is capable of sensitive H2O measurements (in gases)
  - Need to understand performance
  - Does data meet quality objectives?

Picture of PDID plasma



Plot of cal curve

Questions:

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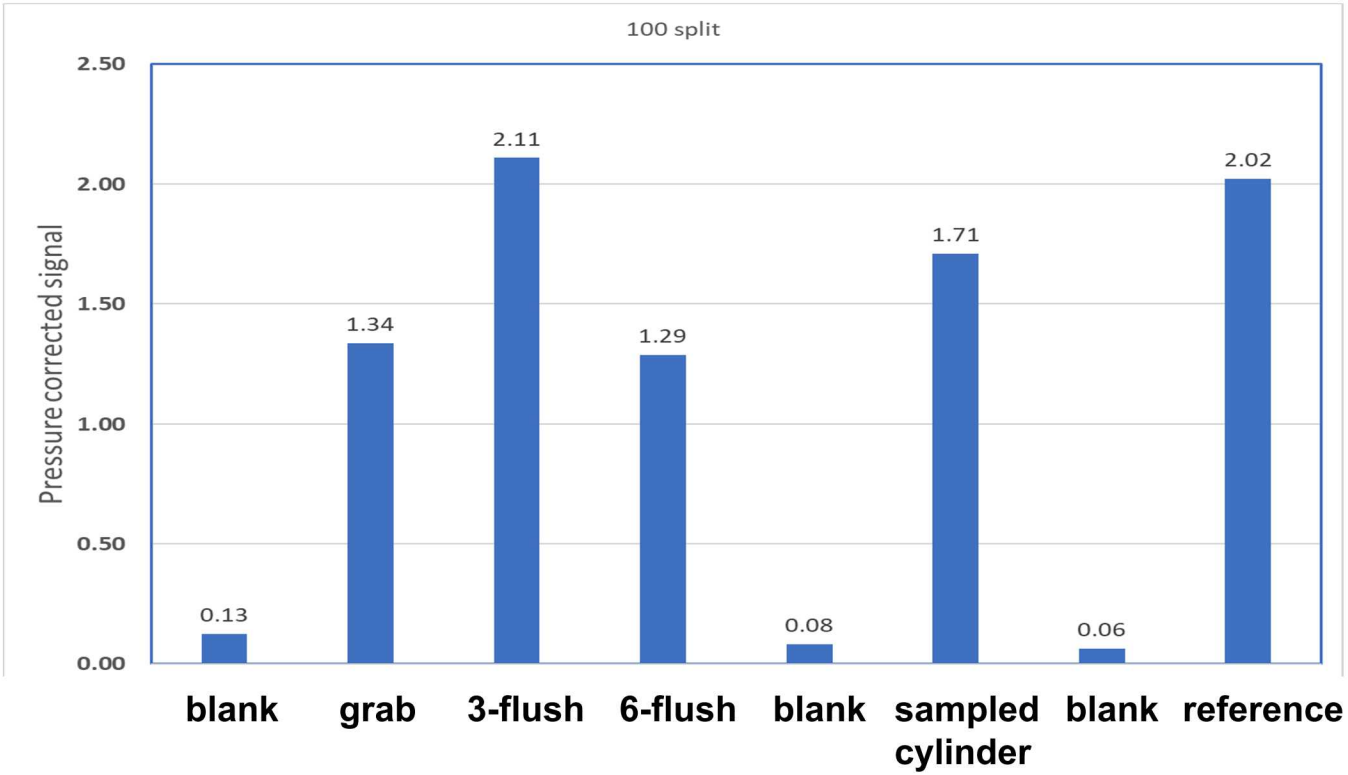
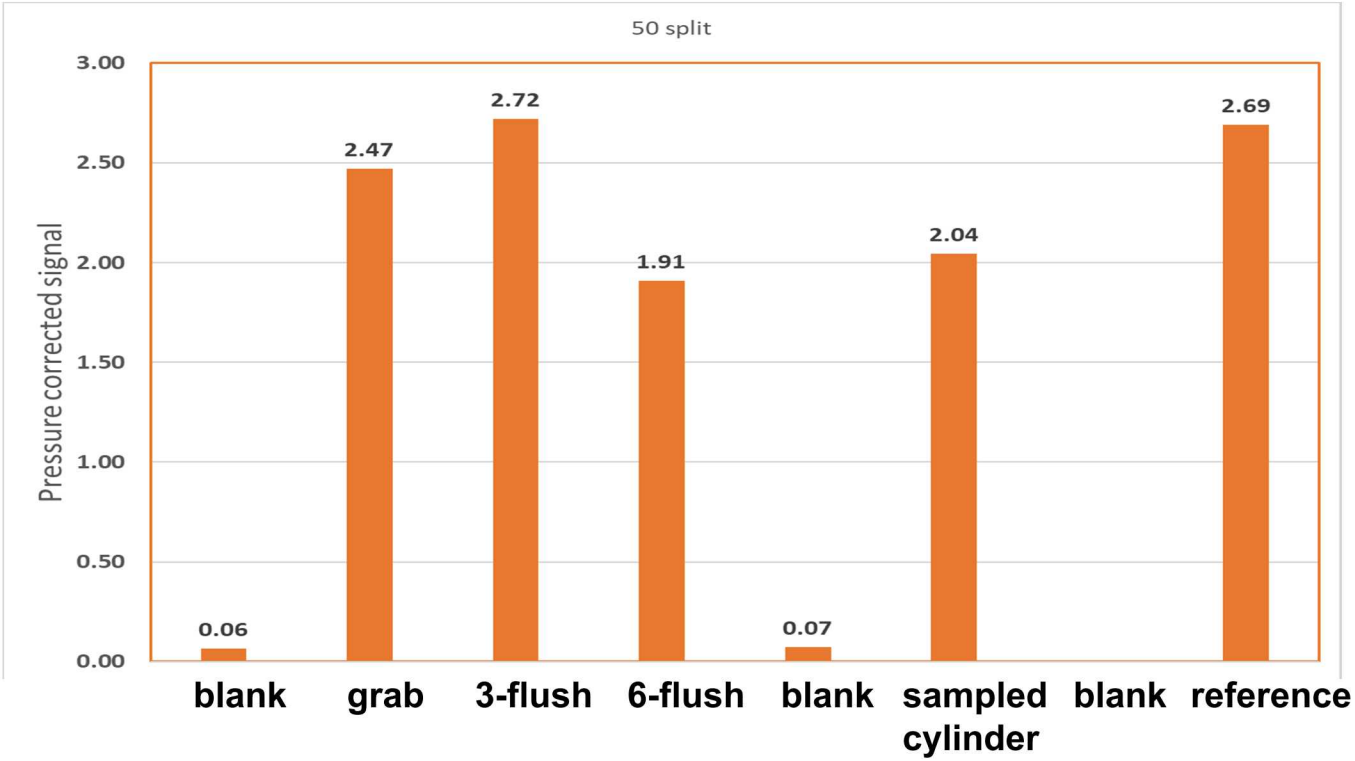
1. Are grab samples representative of a known standard?
2. How does surface adhesion of moisture affect sample signal?
    1. Single grab
    2. Multiple flushes
3. How do grab samples affect the moisture signal of the remaining (small) gas volume?

Comparison of 100:1 split



Comparison of 50:1 split



Current humidity measurement techniques

- Chilled mirror
  - Large gas volumes, long equilibration times
  - Primary standard
  - Expensive investment
  - Must have flowing gas
- Panometrix
  - Large gas volumes
  - Time consuming calibration
  - Questionable robustness
  - Not designed for static gas

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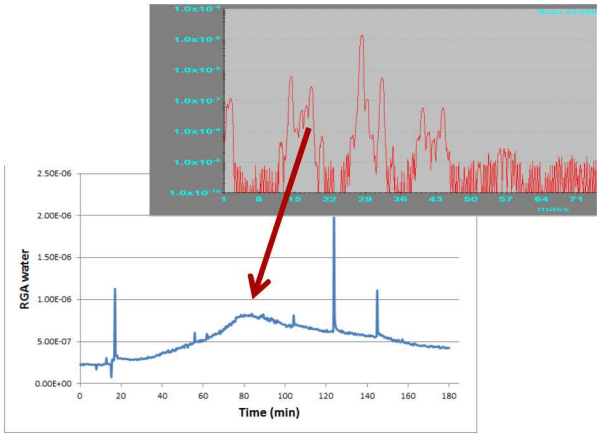

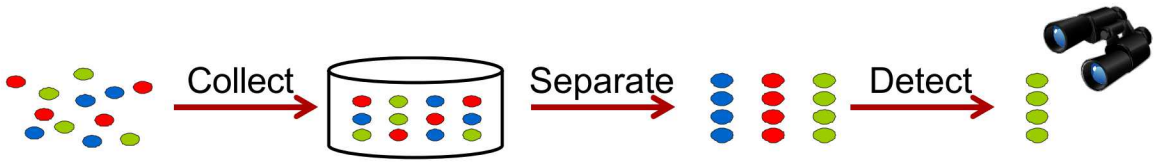

- Validating/technical questions
  - Surface passivation
  - Heating
  - Representative grab samples
  - Pressure changes
  - Standards lifetime

Conclusions

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- Moisture can be an important analyte
- Quantitative measurements are possible
- Careful method parameters are needed for consistent measurements
- Sampling parameters can effect the measurement of a fixed volume