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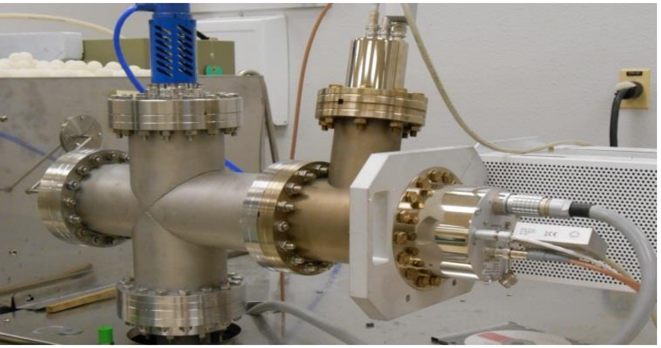

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Use of Modern Leak Detectors for the Calibration of Leak Standards

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U.S. DEPARTMENT OF ENERGY

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NNSA National Nuclear Security Administration

Outline

- **Motivation**
- Fundamentals of Leak Detection
- Calibration of Leak Standards
- Experimental Setup and Approach
- Leak Comparator Results
- Helium Background Study
- Conclusion

Motivation

- Few modern leak detectors are designed or marketed for metrology.
- The now obsolete Leybold-Heraeus ULTRATEST F* was originally designed for metrology applications.
  - Direct streaming, high sensitivity, high resolution signal output with option for all metal seals.
- PSL staff are exploring options to leverage commercial off-the-shelf (COTS) leak detectors for calibration of leak standards at very low leak rates.

*Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately describe the experimental procedure. Such identification does not imply recommendation or endorsement by the authors, Sandia National Laboratories, or NCSL International, nor does it imply that the materials or equipment identified are the only or best available for the purpose.

Motivation (cont.)

- Many modern leak detector designs are optimized for commercial product testing and industrial applications.
  - High throughput, reliability, and simplicity of operation.
  - Typically counterflow systems.
- We are exploring the leak rate sensitivity limits of the counterflow design found in modern leak detectors.
- An experimental program and results are presented, directly comparing performance of a direct streaming leak detector designed for metrology to a modern counterflow leak detector.

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What Are We Trying to Measure?

Leak Rates in Perspective

	Technically Tight	*Gas Tight*	*Virus Tight*	*Bacteria Tight*	*Water Tight*	*Dripping Faucet*
Leak Rate (atm-cc/sec)	10^{-11}	10^{-9}	10^{-7}	10^{-5}	10^{-3}	0.1
Leak Size	100 nm	800 nm	3 µm	10 µm	30 µm	0.1 mm
Escape Time for 1cc He-4 Bubble	**>1000 years!**	~30 years	~100 days	~1 day	~15 min	10 sec
Test Method					Pressure Decay, Bubble Test	
			Tracer Gas, Ambient Pressure			
	Tracer Gas in Vacuum					

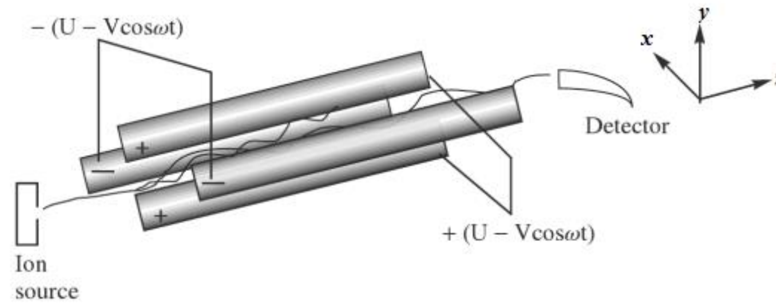
Adapted from *Pfeiffer Vacuum GmbH, Leak Detection Compendium, April 2013.*

Measurement of Low Leak Rates

- Use of a tracer gas (usually helium-4) in high vacuum is necessary for high sensitivity measurement of low leak rates.
- Mass spectrometers are used to differentiate tracer gas from other constituents.
  - Magnetic sector instruments
  - Quadrupole gas analyzers
- Natural helium background (~5.24 ppm at sea level) can contribute significantly to the signal in low leak rate measurements (<10^{-8} atm-cc/sec at STP).
- Electronic noise may also become significant.

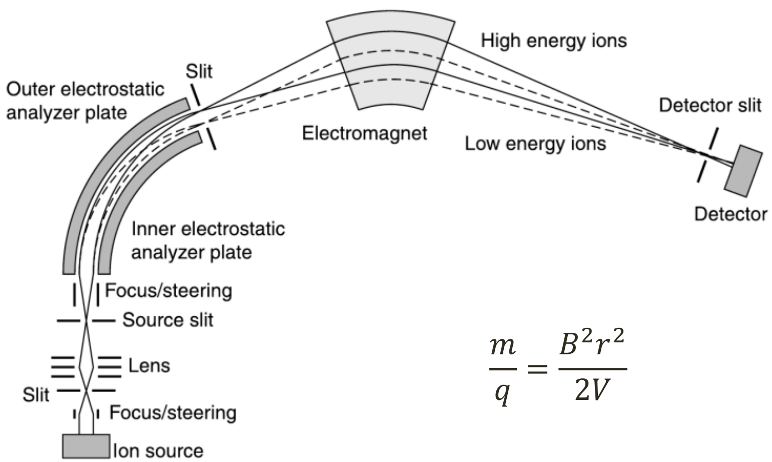
Mass Spectrometry Technology

- Quadrupole Gas Analyzer
  - Oscillating trajectory of ions in quadrupole field → Dynamic narrow bandpass filter.
  - Stability issues and lower accuracy.
- Magnetic Sector
  - Magnetic field deflects ions. Trajectory set by m/q; slit filters unwanted ions.
  - Highest accuracy and mass resolution.



Quadrupole Analyzer Principle of Operation.
Source:* Dass, *<u>Fundamentals of Contemporary Mass Spectrometry</u>

$$\frac{\partial^2 x}{\partial t^2} = -\left(\frac{e}{m}\right)\left[\frac{U + V\cos(\omega t)}{r_0^2}\right]x, \quad \frac{\partial^2 y}{\partial t^2} = \left(\frac{e}{m}\right)\left[\frac{U + V\cos(\omega t)}{r_0^2}\right]y$$



$$\frac{m}{q} = \frac{B^2 r^2}{2V}$$

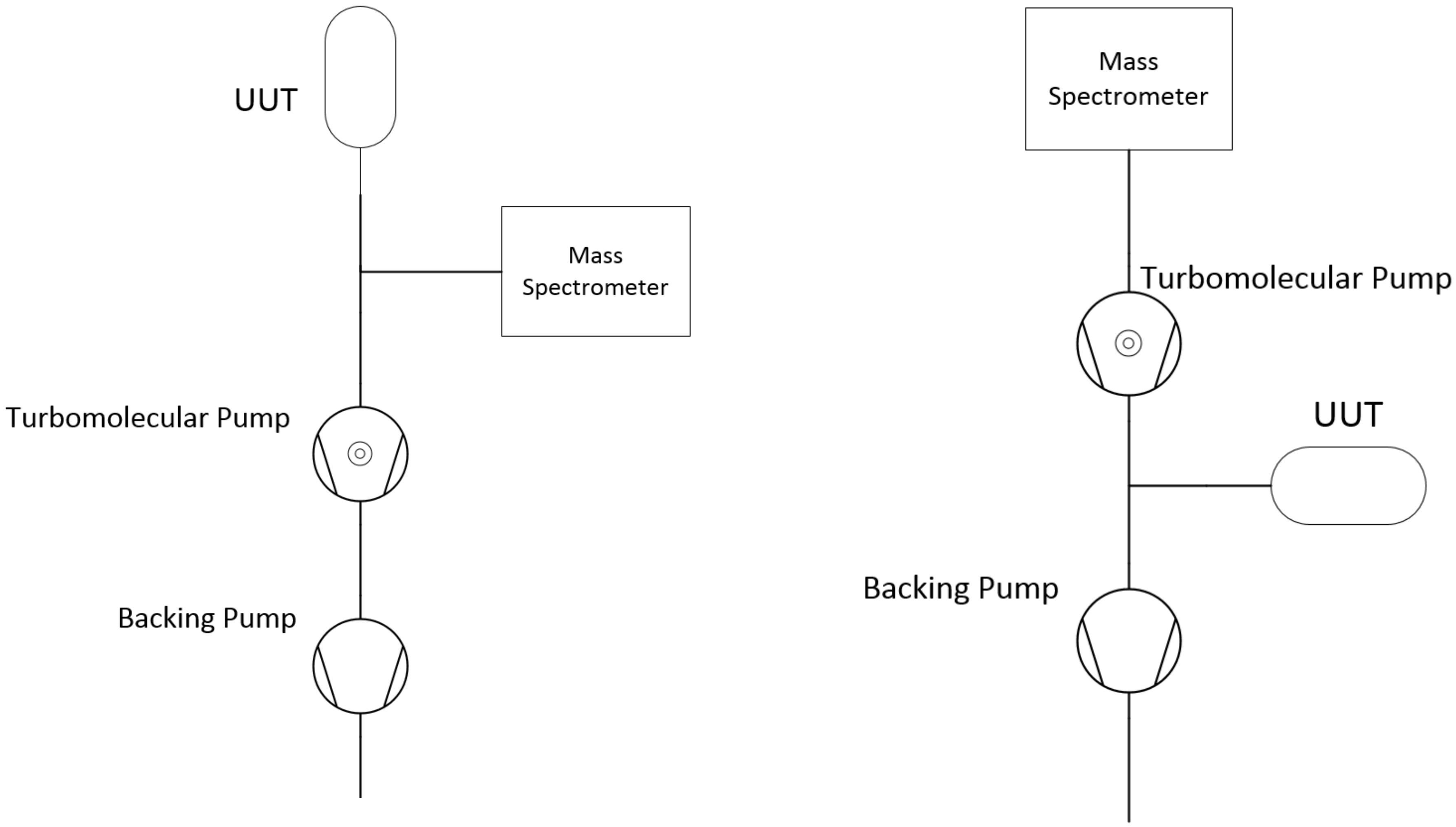
Dual Magnetic/Electric Sector Principle of Operation.
Source:* Kraj et al., *<u>Mass Spectrometry</u>

	Sector	Quadrupole
Mass Accuracy	1-5 ppm	100 ppm
Resolving Power	10^2-10^5	4000
Linear Dynamic Range	10^9	10^7
Abundance Sensitivity	10^6-10^9	10^4-10^6

Direct Streaming vs. Counterflow

- **Direct streaming configuration**
  - Higher sensitivity
  - Slower equilibration time
  - Not suitable for higher gas flows/pressure; greater risk of damaging filament or mass spectrometer.
- **Counterflow configuration**
  - Lower sensitivity (helium must backstream through turbopump)
  - Faster equilibration time
  - Allows for higher gas flows/pressure; simplicity of operation and less risk of damaging filament or mass spectrometer.

Direct Streaming vs. Counterflow (cont.)



Direct Streaming (Left) and Counterflow Configurations (Right)

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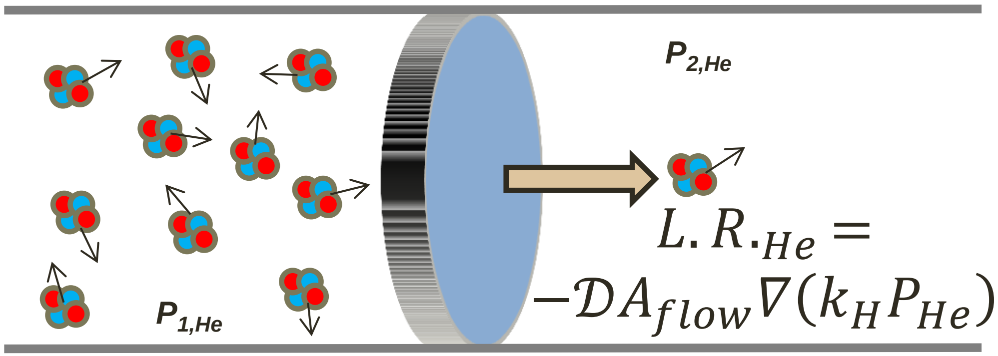
Construction of a Calibrated Leak

- Physical leak elements
  - Crimped tube, drawn glass capillary, sintered orifice
  - Prone to clogging and contamination
- Permeation elements
  - Glass, quartz, palladium
  - Lower leak rates achievable
  - Better stability
  - Limited gases and long equilibration time
  - Strong temperature dependence
    - ***≈3% per ˚C***

Cross-section of a Permeation Leak Element.
Image courtesy VTI.

Glass or Quartz Membrane



$P_{1,He} >> P_{2,He}$

Leak Calibration Techniques

- Accumulate Dump
  - Standard is an expansion of gas from known pressure, volume, and temperature dumped through restriction into mass spectrometer; unknown leak gas collected for time *t* dumped under identical conditions.
  - Uncertainty dominated by *P*, *V*.
  - Prohibitively long collection time for low-level leaks.
- Primary gas flow comparison with quadrupole gas analyzer (NIST).
- *PΔ*V (SNL) and V*ΔP*
  - $L.R._m = {}^{\partial n}/_{\partial t} = \left[P\left({}^{\partial V}/_{\partial t}\right) + V\left({}^{\partial P}/_{\partial t}\right)\right]/RT$
  - Outgassing of other species affects results.
- **Direct comparison methods**
  - $L.R._{m,unk} = L.R._{m,std}\,[S_{unk}(1+\alpha_{std}\Delta T_{std})]/[S_{std}(1+\alpha_{unk}\Delta T_{unk})]$
  - Limited by sensitivity of mass spectrometer.

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Experimental Setup

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- Leak comparison setup
  - Vacuum manifold enables valving between Standard and UUT, turbo pumping station and leak detector
  - Standard- Insulated Chamber
  - UUT- Temperature controlled chamber (to obtain temperature coefficient
- Modified setup to incorporate modern counterflow leak detector.
- Manual valving allows changing between leak detectors.

Std. Chamber (left) and UUT Chamber (right). Underneath, ULTRATEST F leak detector on right, modern counterflow leak detector and backing pump on left.

Experimental Method

- Data collected for following for each leak element on each leak detector:
  - Zero voltage (x4), Std. voltage (x3), UUT voltage (x3), Sum Std. + UUT voltage (x3), 60 seconds of data collection for each.
  - 10 runs performed at three temperatures for UUT.
  - UUT leak rate calculated for each temperature using leak comparison equation.
- Time of test uncertainty reported for UUT Leak Rate at k=2:
  - Includes repeatability of std. voltage, repeatability of UUT voltage, non-linearity, std. certification uncertainty, and std. temp. coefficient uncertainty.
- Note that for leak rates <1×10^{-9} atm-cc/sec at STP, a 10^{-9} leak standard was used against the UUT, increasing uncertainty due to nonlinearity.

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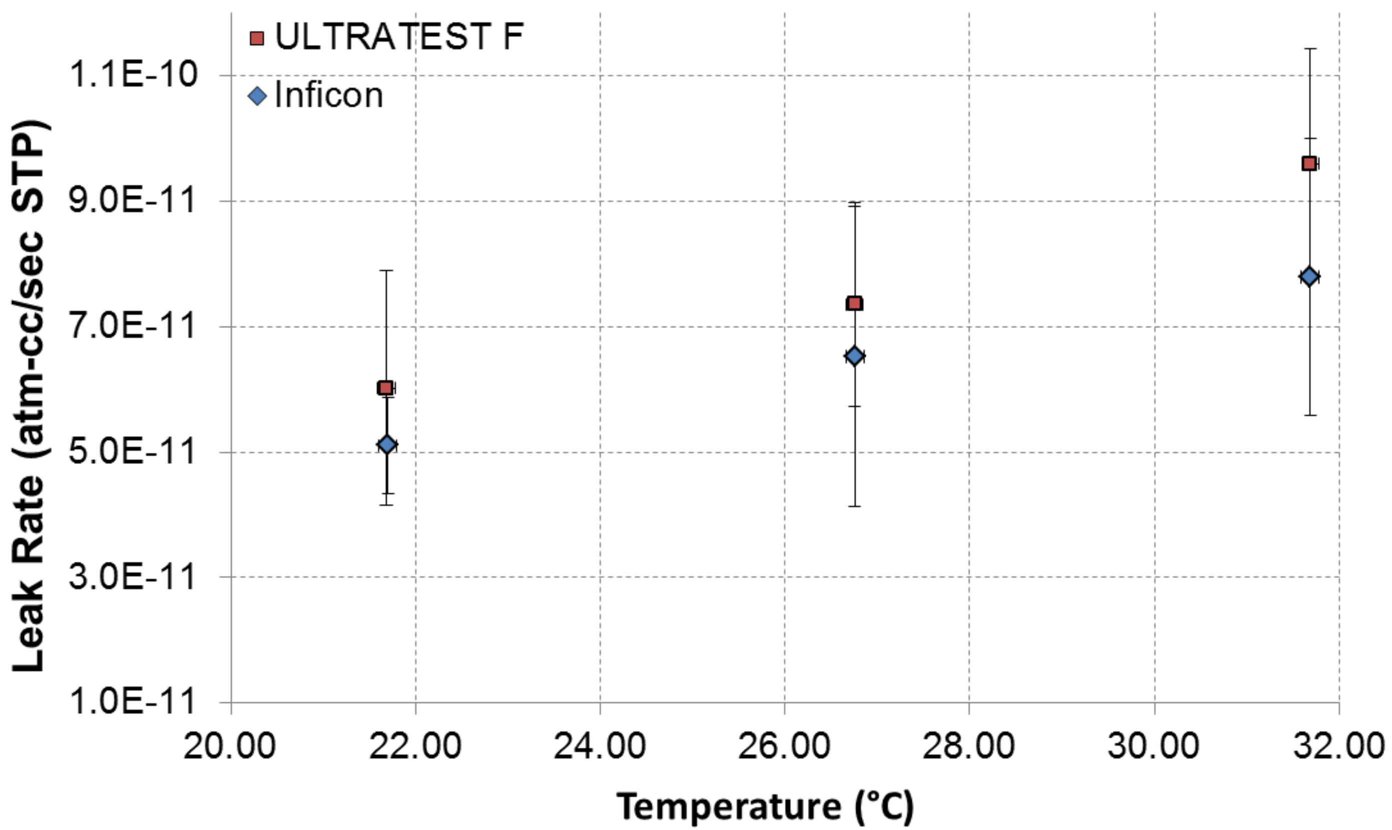
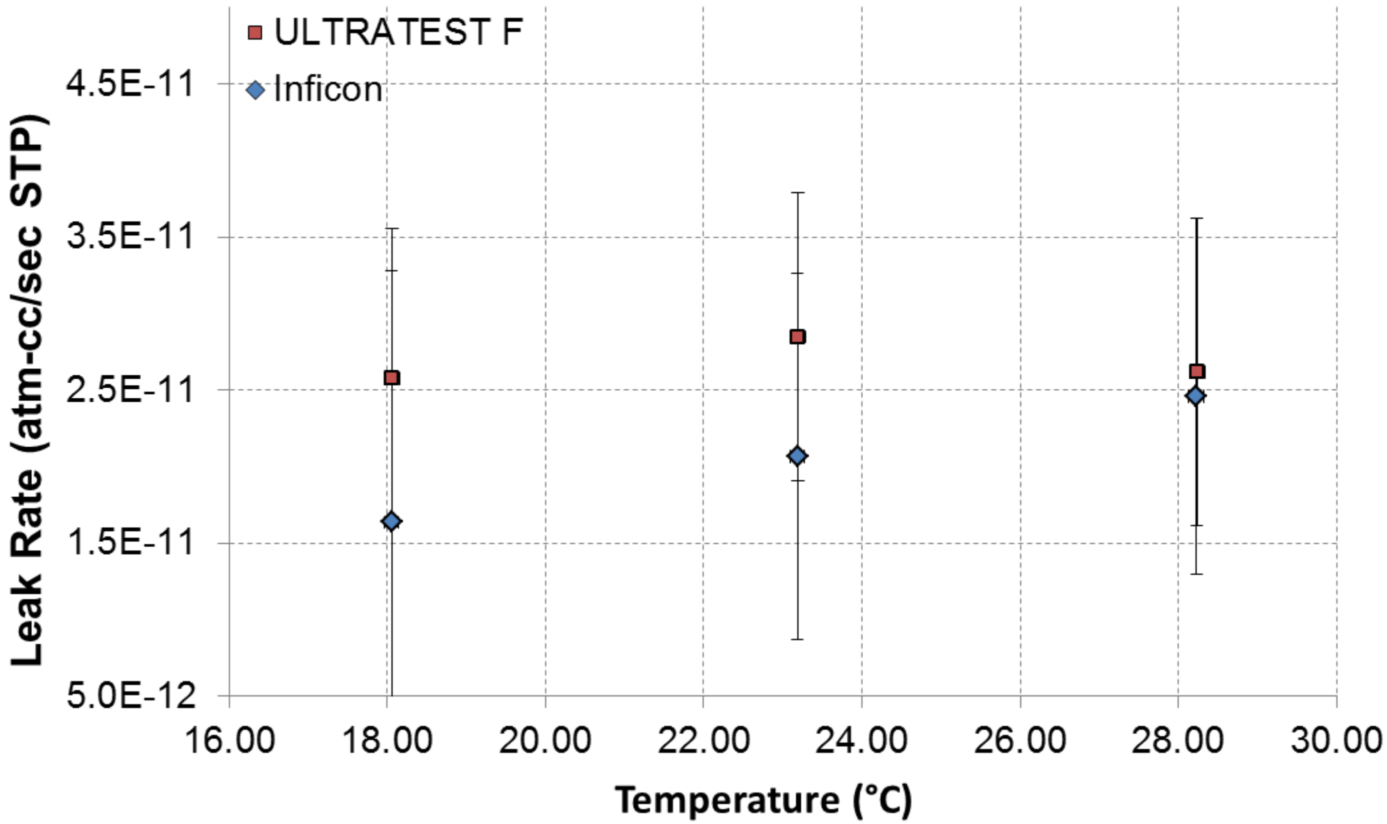
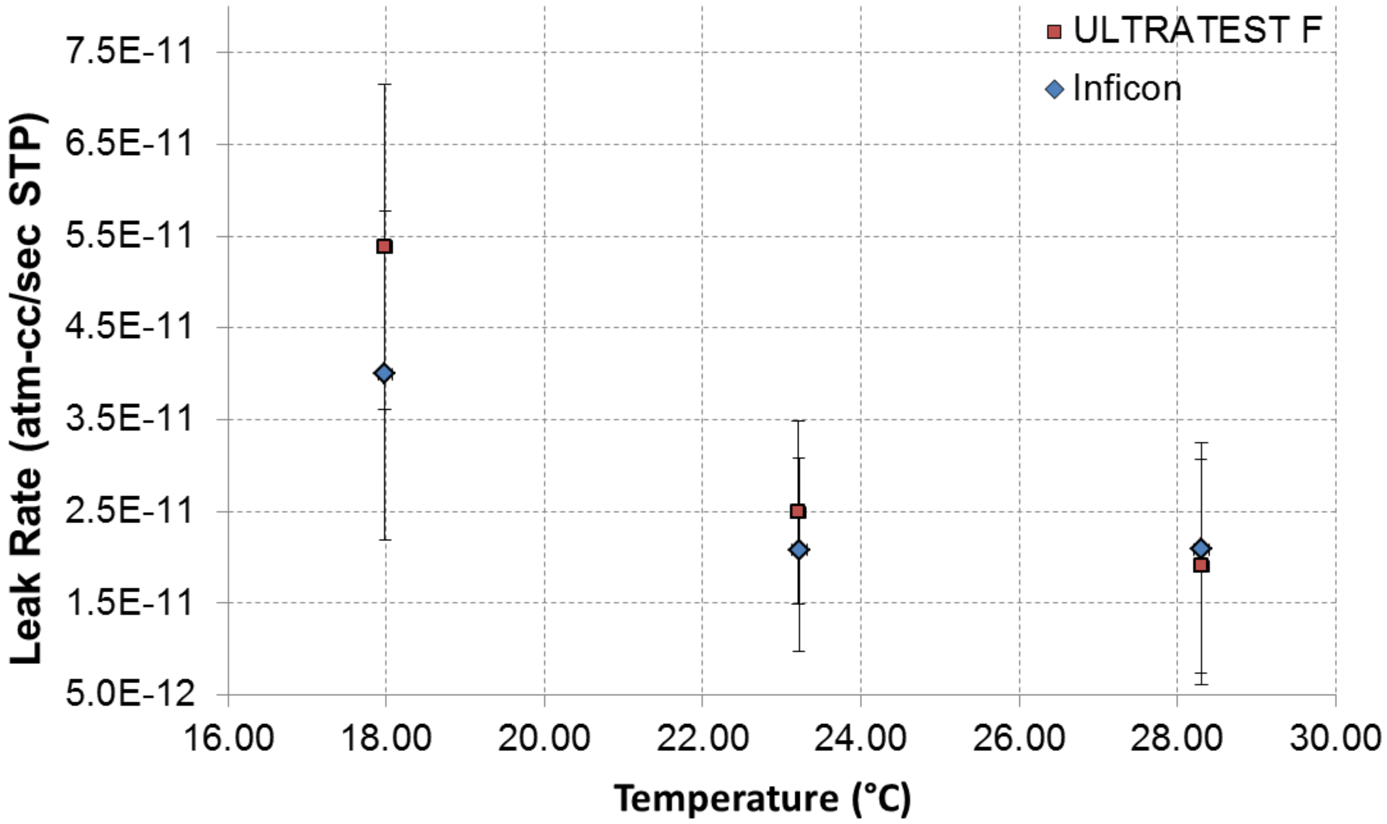
7.5×10^{-11} atm-cc/sec (STP) nominal He-4 Permeation Leak



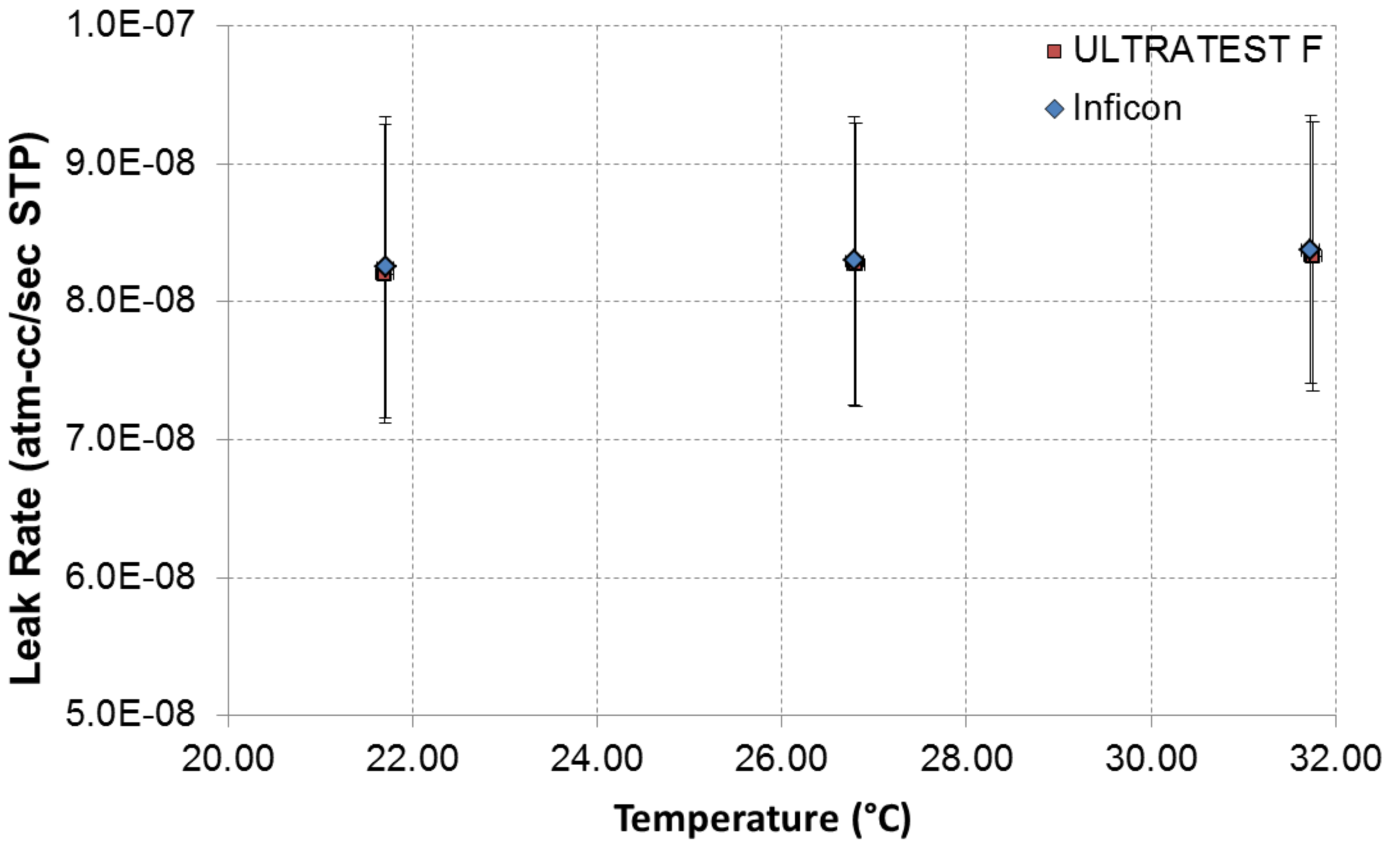
2.5×10^{-11} atm-cc/sec (STP) nominal He-4 Permeation Leak



$7.5x10^{-12}$ atm-cc/sec (STP) nominal He-4 Permeation Leak



$7.72x10^{-8}$ atm-cc/sec (STP) nominal Deuterium Capillary Leak



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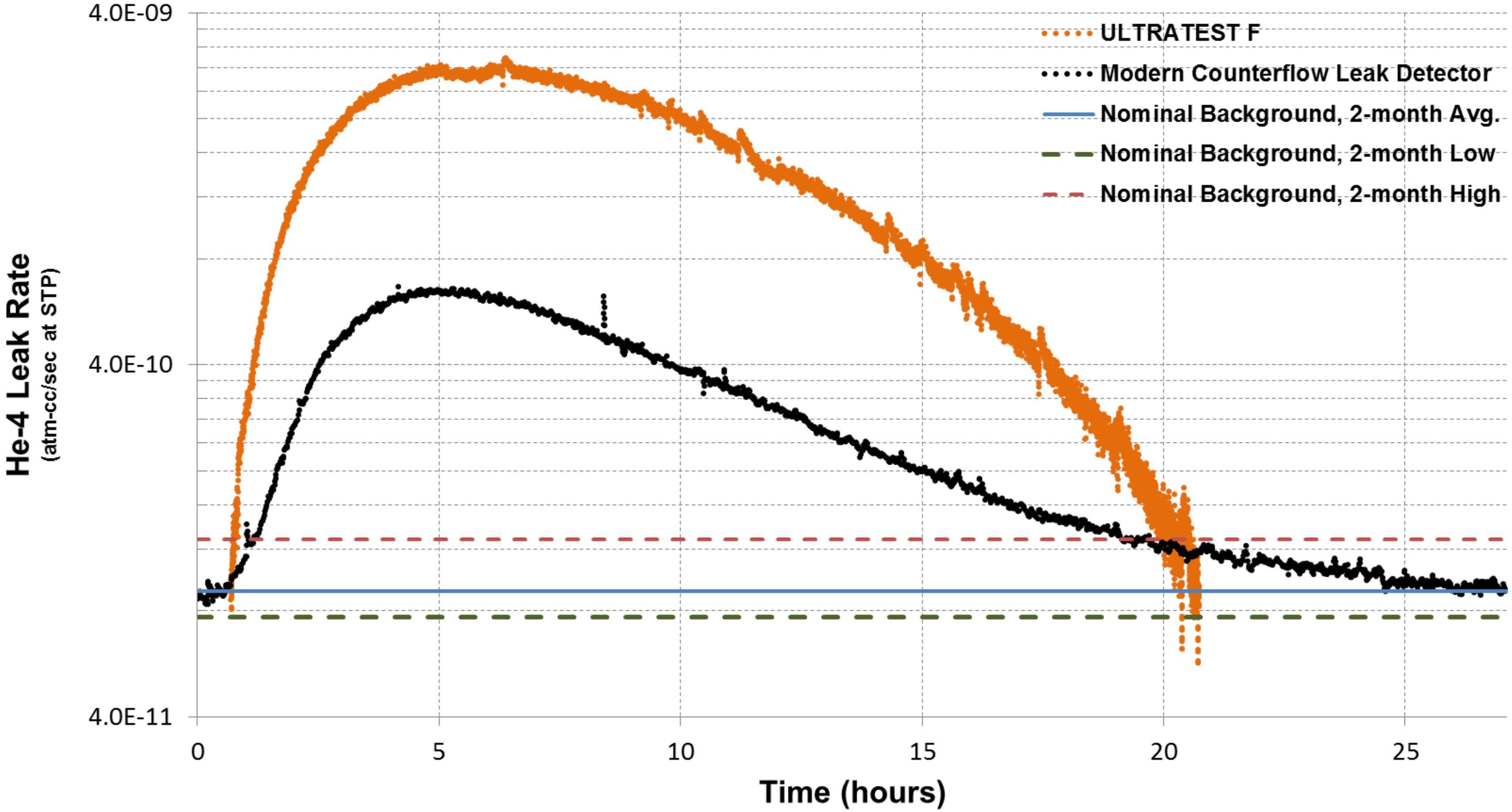
Helium Background Study

- Helium background appears to be a major factor limiting accuracy at very low leak rates.
- Backstreaming through turbo pump, along with helium diffusion through non-metal seals is possible.
- Equilibration time longer for the modern counterflow detector, likely due to use of elastomer seals.
- Experiment performed during liquid helium transfer in adjacent laboratory to evaluate each leak detector's response to background spike.

Helium Background Study (cont.)



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Conclusion

- Possible to build a leak comparison system capable of calibrating leaks down to at least 10^{-11} atm-cc/sec by leveraging commercial technology with modern counterflow design.
  - Precise control of leak environmental parameters is required (i.e., temperature).
  - Larger uncertainties due to background contribution to helium signal, which is unavoidable with elastomer seals.
- Modern counterflow leak detector performs comparably to direct streaming system for very low leak rates as long as zero hold times increased.
  - Longer equilibration at very low leak rates likely due to virtual leaks from elastomer seals in counterflow detector.

Conclusion (cont.)

- However, counterflow detector less susceptible to backstreaming during helium background spike, likely due to much higher compression ratio turbomolecular pump (~1.3x10^7 He vs. ~3000 He for replacement pump on ULTRATEST F).
- Replacement of elastomer seals on counterflow detector with all metal seals may reduce background signal, improve signal to noise ratio, and reduce uncertainty for very low leak rate measurements.