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Precision Volume Measurements: Challenges to Reducing Uncertainties

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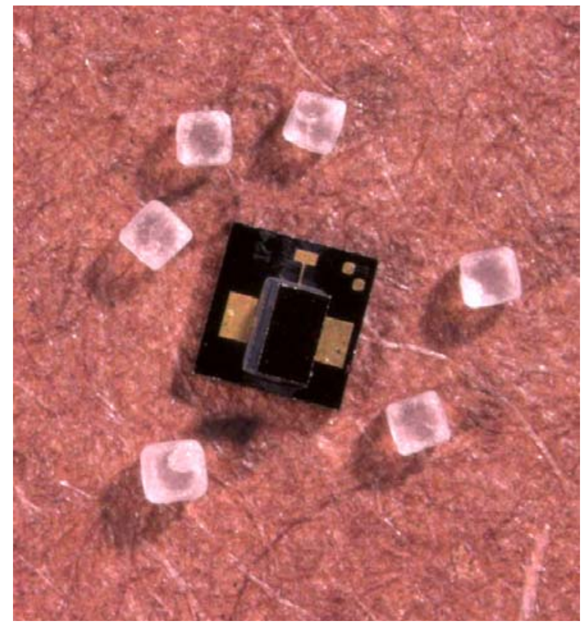
Accurate volume determination is extremely important in many disciplines

$$P\,V = n\,R\,T$$

The ideal gas law is used in many, many applications
- Semiconductor industry
- Aerospace
- Defense
- Automobile
- Energy

In this talk, we will focus on the measurement of VOLUME.



30 nanoliter MEMS device surrounded by grains of table salt.

Precision gas volume measurements

Selected previous methods

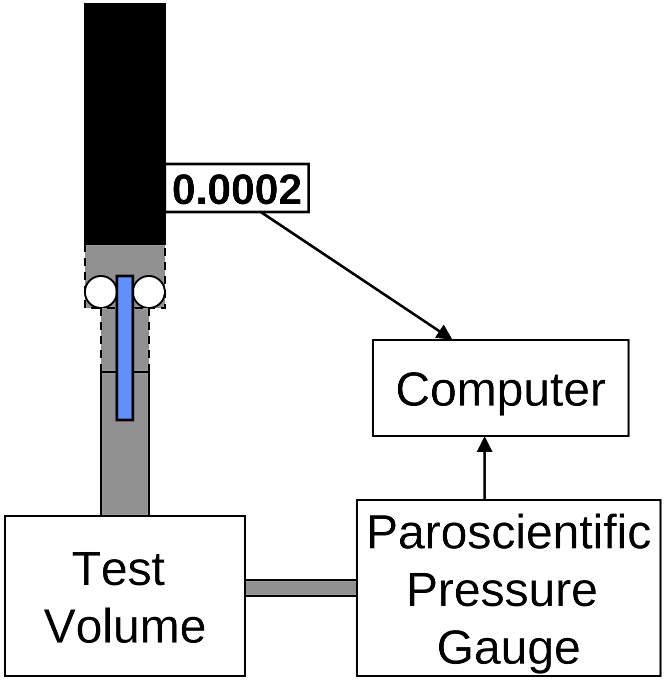
- Gravimetric: liquid filled (even mercury filled)
- Volume expansion – still need a standard
- Pressure rise with calibrated leaks

Need a method for determining volumes…

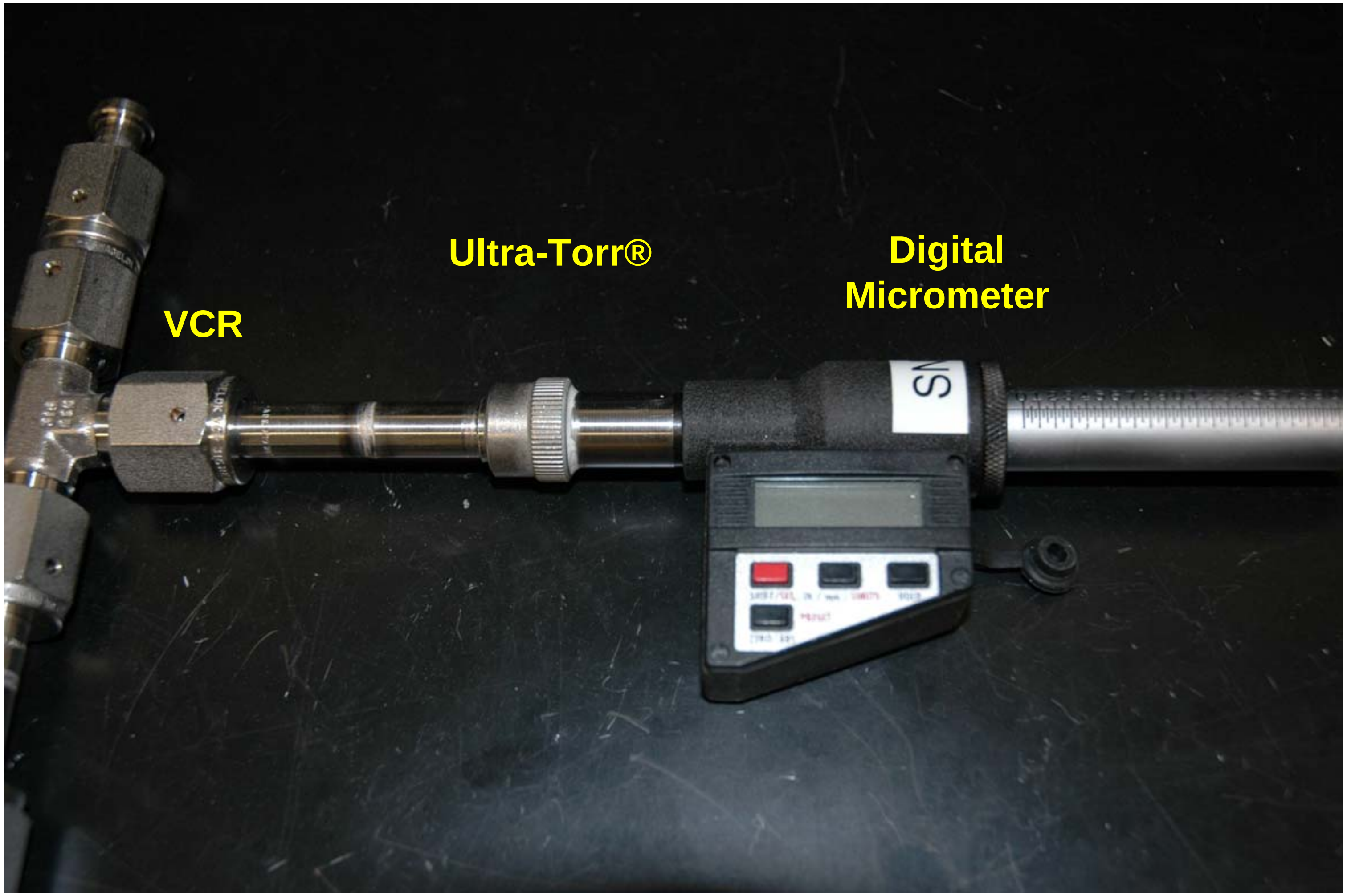
- Of complex manifolds
- Without exposing the manifold or part to a liquid
- That minimizes impact of outgassing and virtual leaks

Block diagram of the experiment

The experiment involves moving a piston (shown in blue) in or out of the test volume a known distance and recording the induced pressure change.



Micrometer assembly



Theory - derivation of equation

- **Assume isothermal**
- **Simple equation**
- **We bypass the direct solution for V_1 in favor of the least-squares compatible form.**

$$P_1V_1 = P_2V_2$$

$$P_1V_1 = P_2\left(V_1 + dV\right)$$

$$V_1 = \left(\frac{P_2}{P_1 - P_2}\right)dV$$

$$dV = V_1\frac{P_1}{P_2} - V_1$$

Slope

Intercept

What are the contributors to the measured volume uncertainty?

$$dV = V_1 \frac{P_1}{P_2} - V_1$$

- ***dV***
  - Linear linear movement (largest contributor to uncertainty)
  - Piston diameter as a function of displacement
- **P_1 – measured using a Paroscientific pressure gauge**
- **P_2 – measured using the same Paroscientific pressure gauge as P_1**
- **Temperature – measures taken to create an isothermal environment (i.e., $T_2/T_1 = 1$)**

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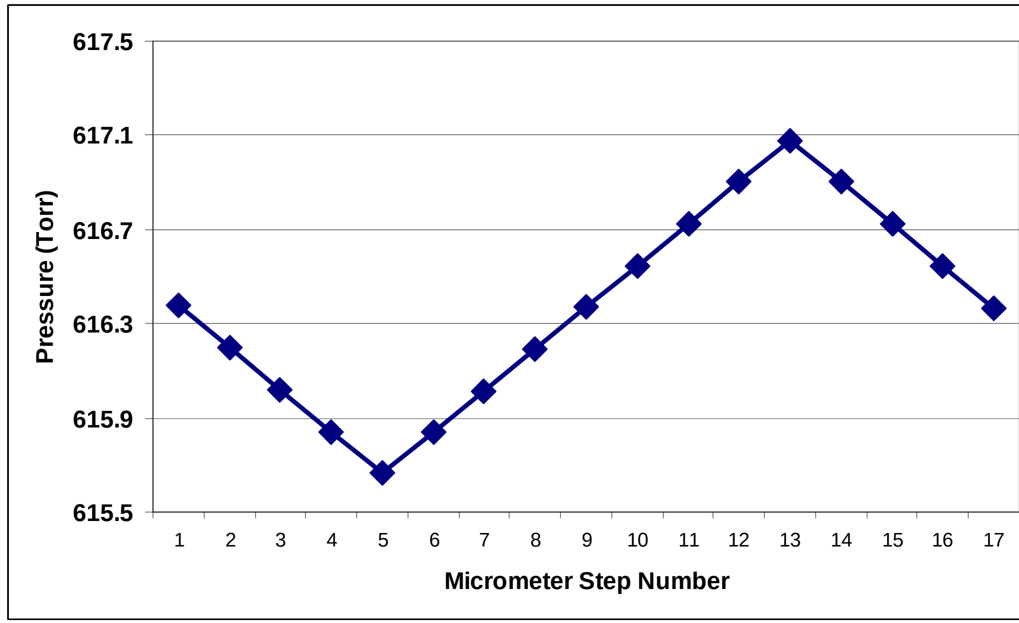
Typical pressure step cycle for a single volume measurement

- **The delta volume is induced by moving the micrometer and the pressures at each step are recorded.**
- **These data are amenable to least squares analysis.**

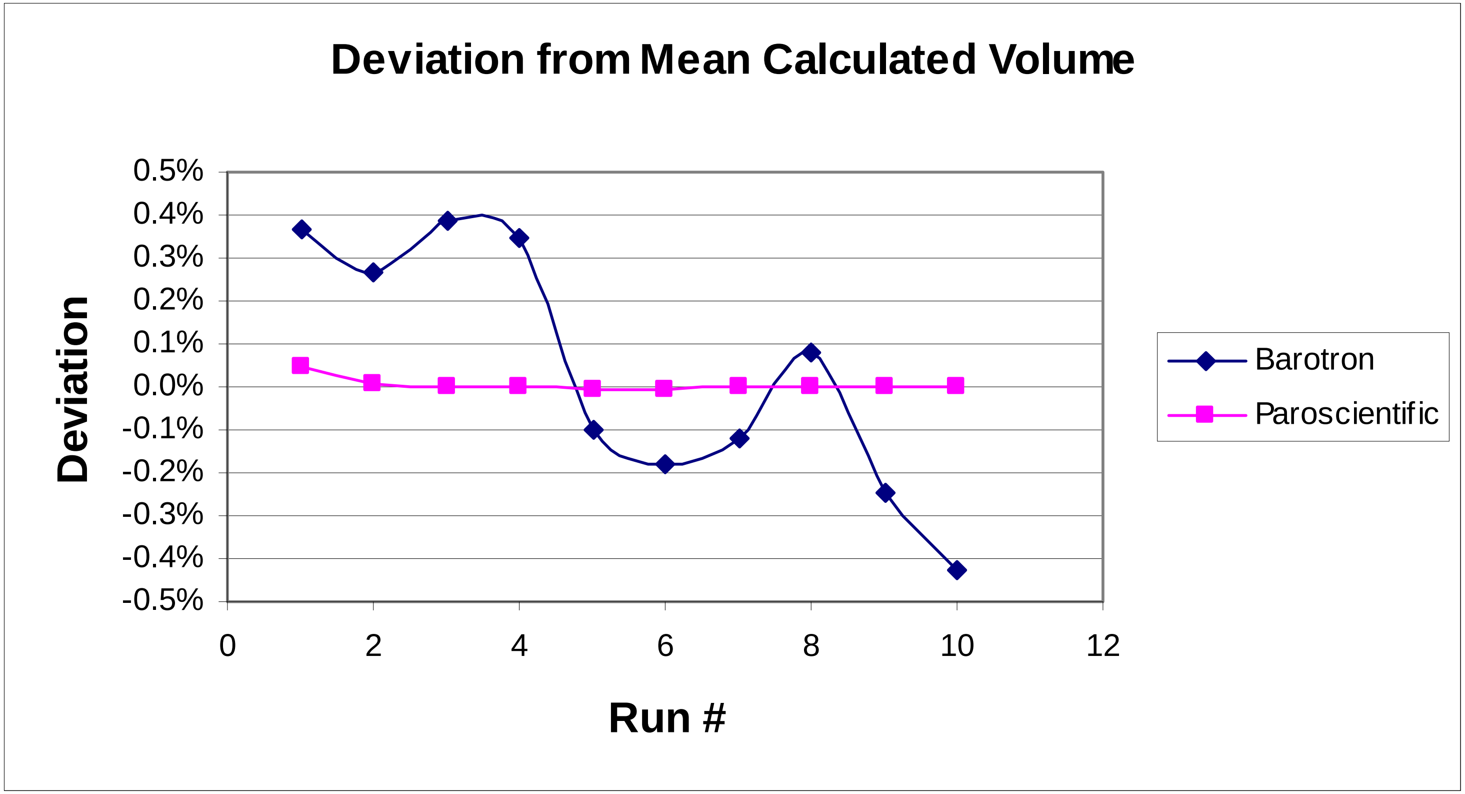


$$dV = V_1 \frac{P_1}{P_2} - V_1$$

$$\downarrow \qquad\qquad \downarrow$$

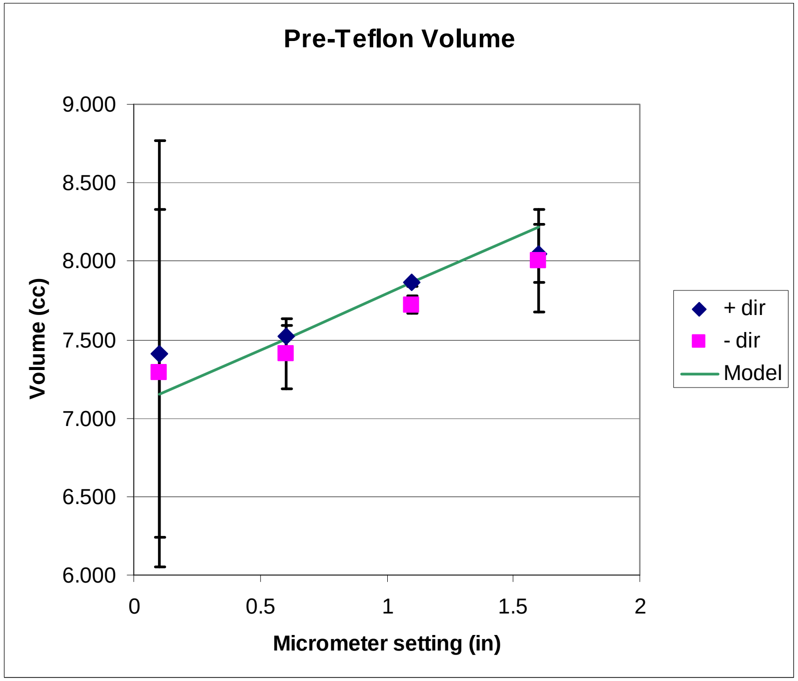
$$y = m\,x + b$$

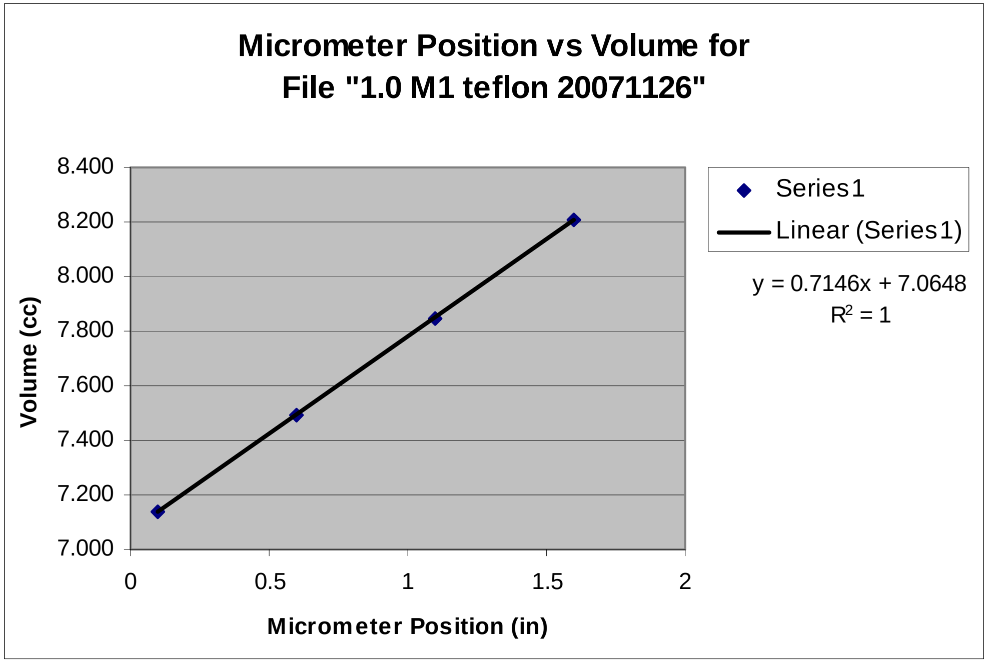
Choosing the correct pressure gauge for the application



These data are for a nominal 50 cc volume. The superior performance of the Paroscientific pressure gauge for this application is clearly seen.

Sealing to micrometer shaft is challenging



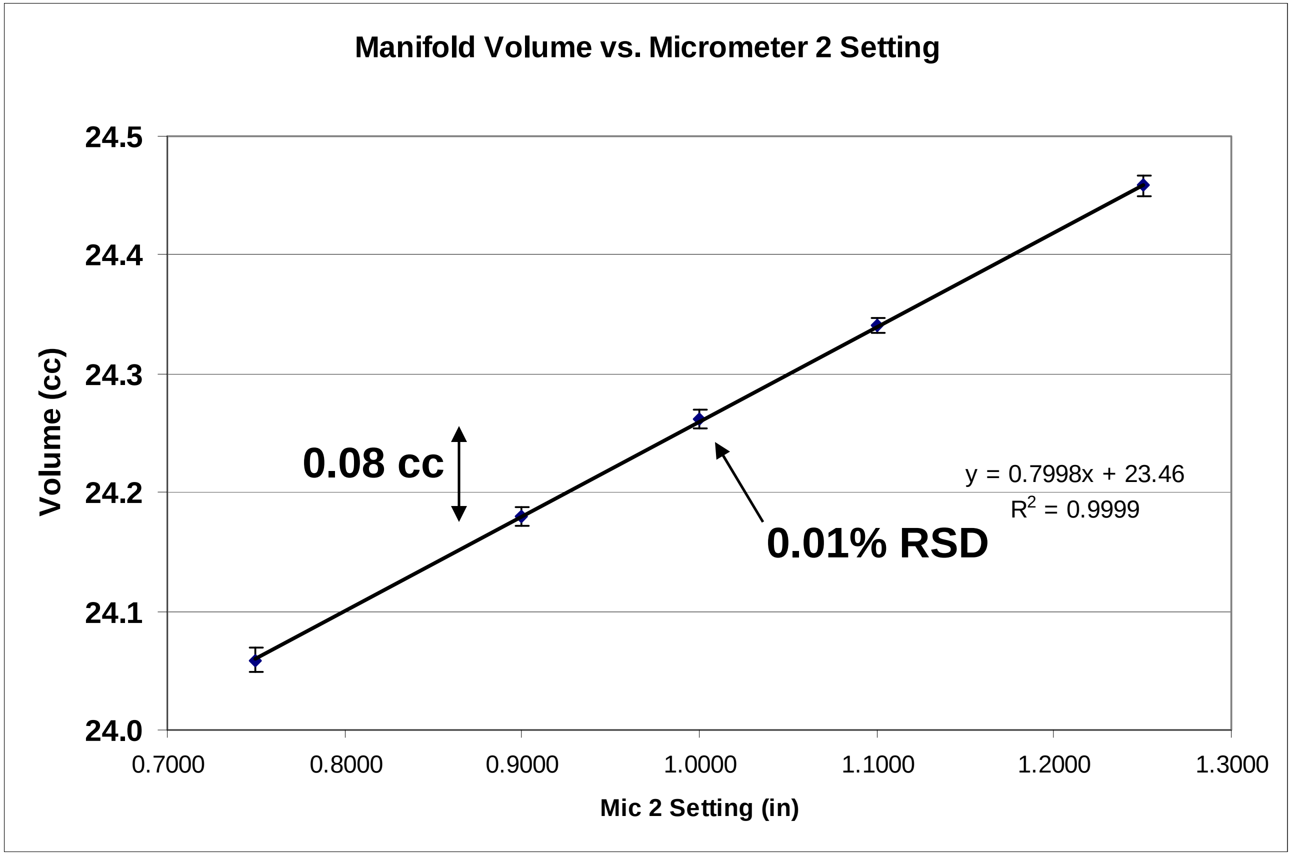


- **Implementation of a Teflon o-ring seal instead of the standard butyl o-ring in the Ultra-Torr® fitting improved repeatability tremendously and eliminated hysteresis.**

Improving Temperature Stability: "The Marble Sarcophagus"

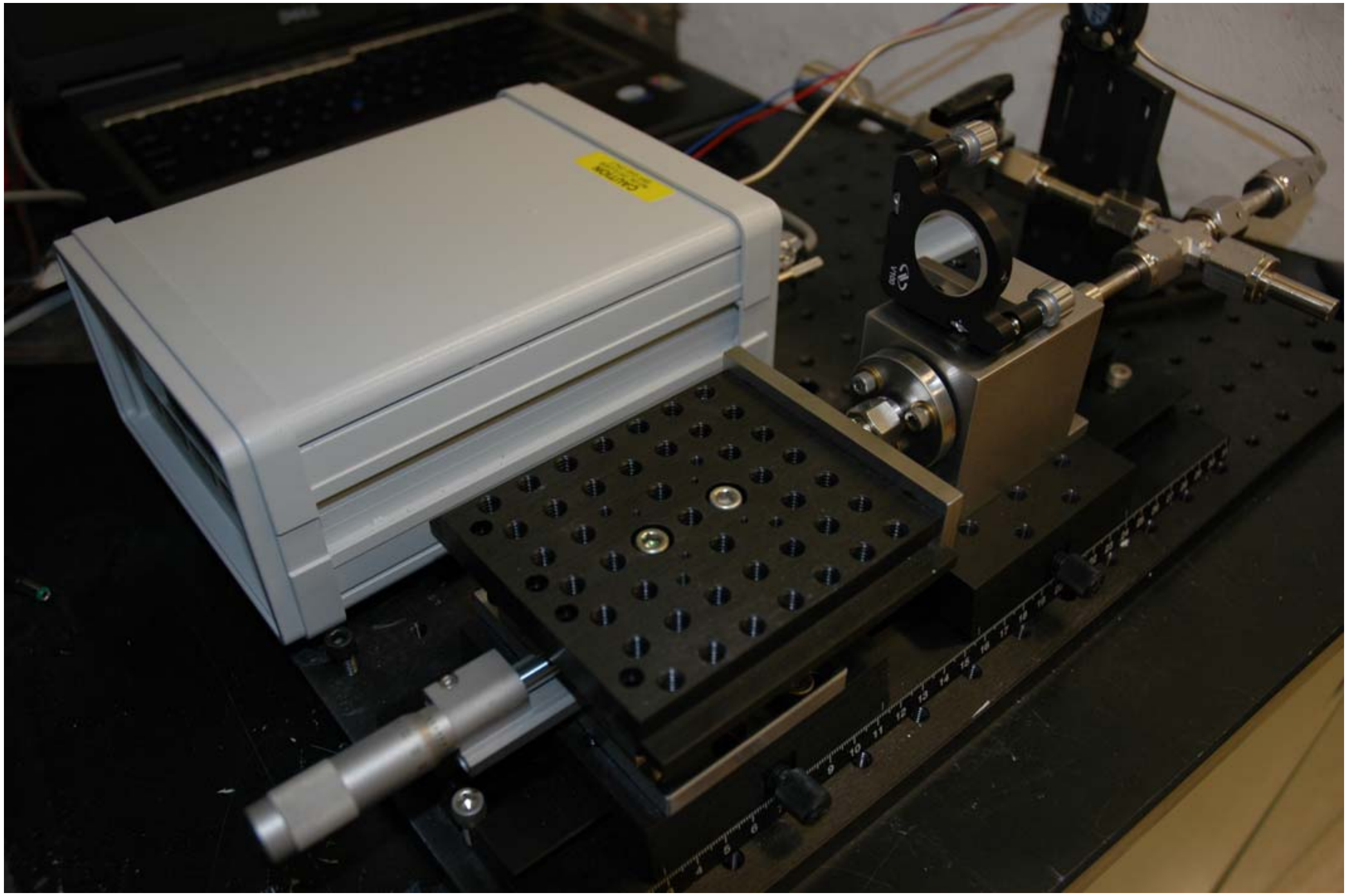
- **A marble "sarcophagus" (4 inch thick walls) was constructed as a thermal ballast.**
- **Temperature drifts are now less than 0.01°C/hour with drifts during typical 1-2 minute measurement cycles much less**
- **Provides stability without the mess of water baths**

Volume measurements using "marble sarcophagus" have high precision



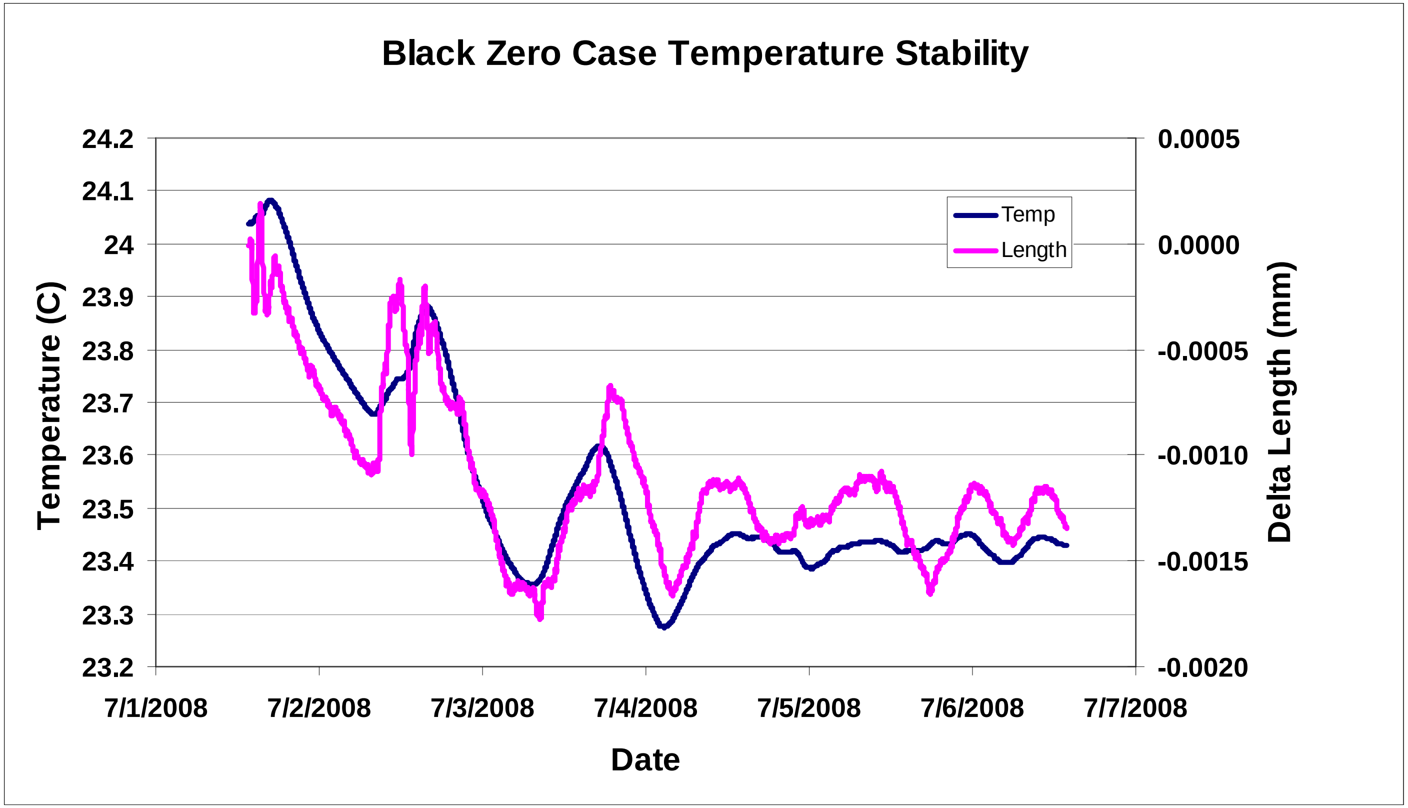
- Error bars are 3-sigma values for five replicate measurements at each point. Micrometer 2 was used to provide a know change in volume in the manifold.

Future Enhancement - Interferometer



The piston linear movement will be measured with a laser interferometer to reduce that uncertainty orders of magnitude.

Impact of temperature drift on interferometer length measurement



- **Maximum change is 18 ppm/°C so for a stable environment (+/- 0.002°C) the impact of temperature will be minimal on the length measurement.**

Better tools will enable reduced uncertainties for piston position

	Resolution (inches)	% of 0.025 inch movement
Vernier micrometer	0.001	4.0%
Digital micrometer	0.00005	0.2%
Laser interferometer	> 0.000001	< 0.04%

Conclusion

- The measurement repeatability has been greatly improved by:
  - Moving to a least-squares compatible form of the volume equation
  - Choosing a precise, stable pressure gauge
  - Using a Teflon o-ring to seal to the piston
  - Increasing the thermal mass around the calibration system to dampen temperature fluctuations
- Repeatability of volume measurements to better than 0.01% RSD for 5 measurements is readily attainable.
- The future enhancement of adding the laser interferometer will greatly reduce the linear movement error.