

MonoSorb Surface Area Analyzer: Repurposing an Instrument from 1981

Penny P. Osborne; Dr. Joseph DiVerdi • CHEM 532 • Instrument Project

Objective:

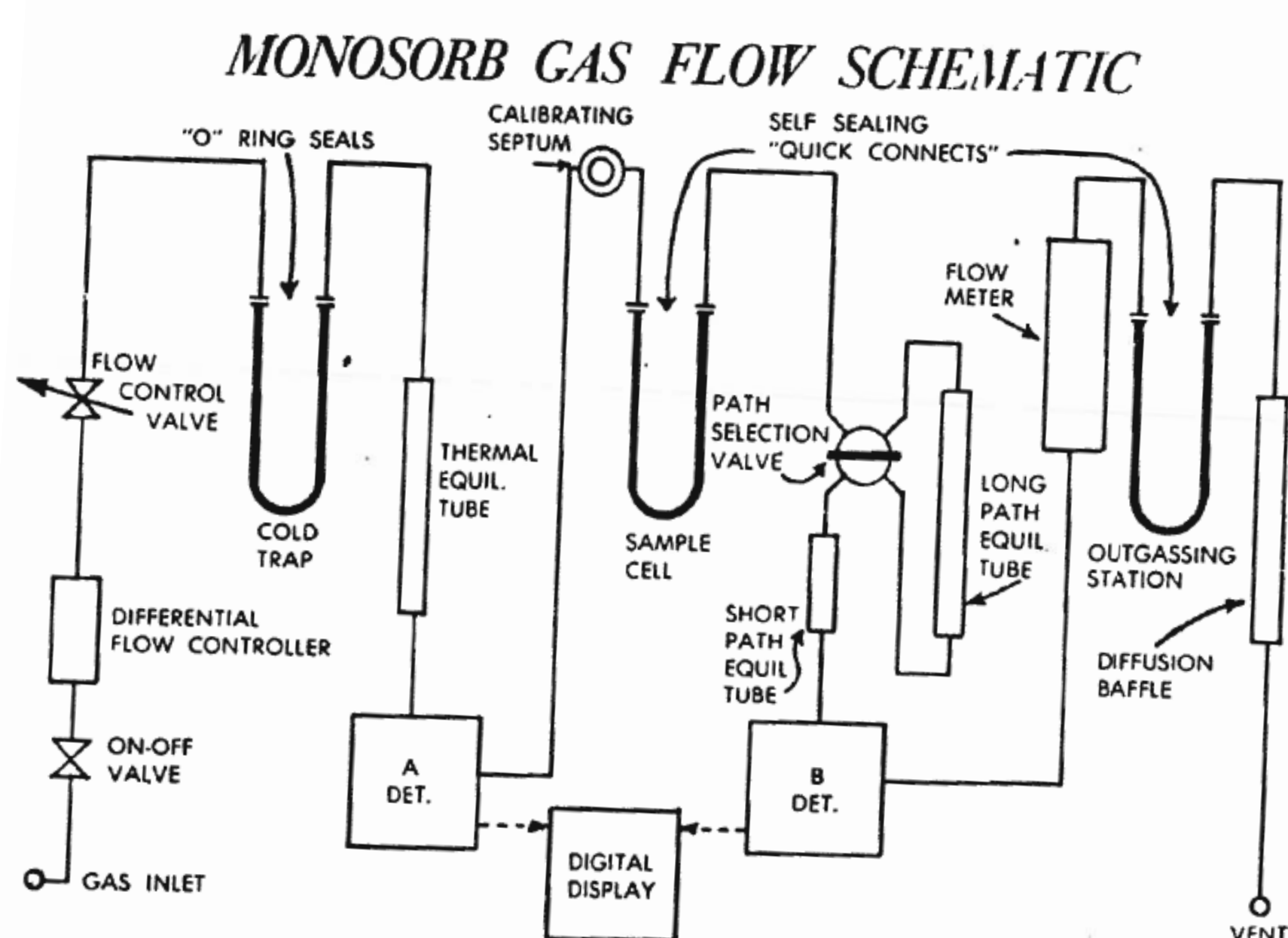
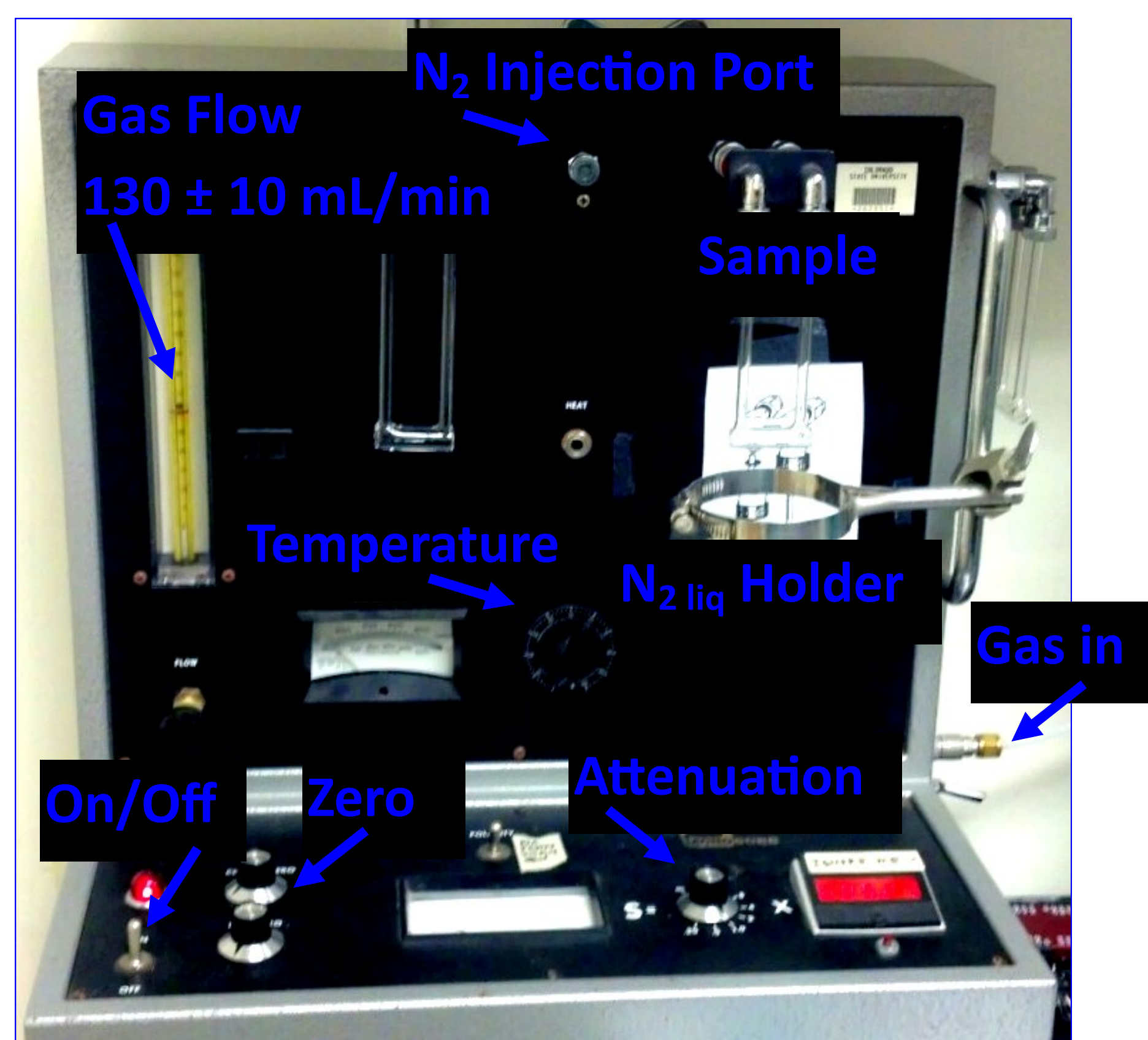
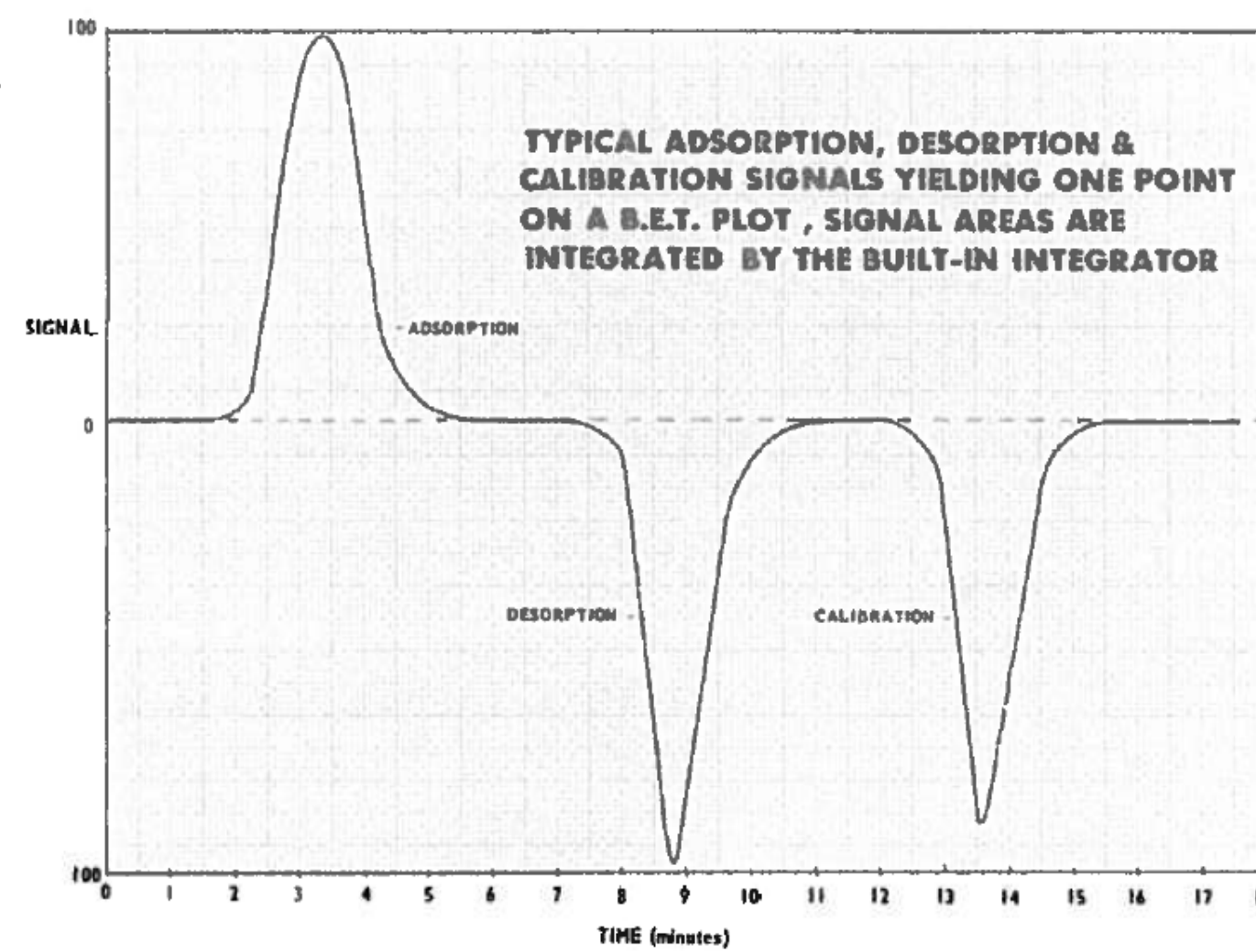
The surface area analyzer was recently updated with a LabJack interface to collect real-time data (1000 Hz).

Previously it had a digital readout with no time component.

Now bugs need to be worked out regarding the data acquisition, analysis, and processing.

How The MonoSorb Works:

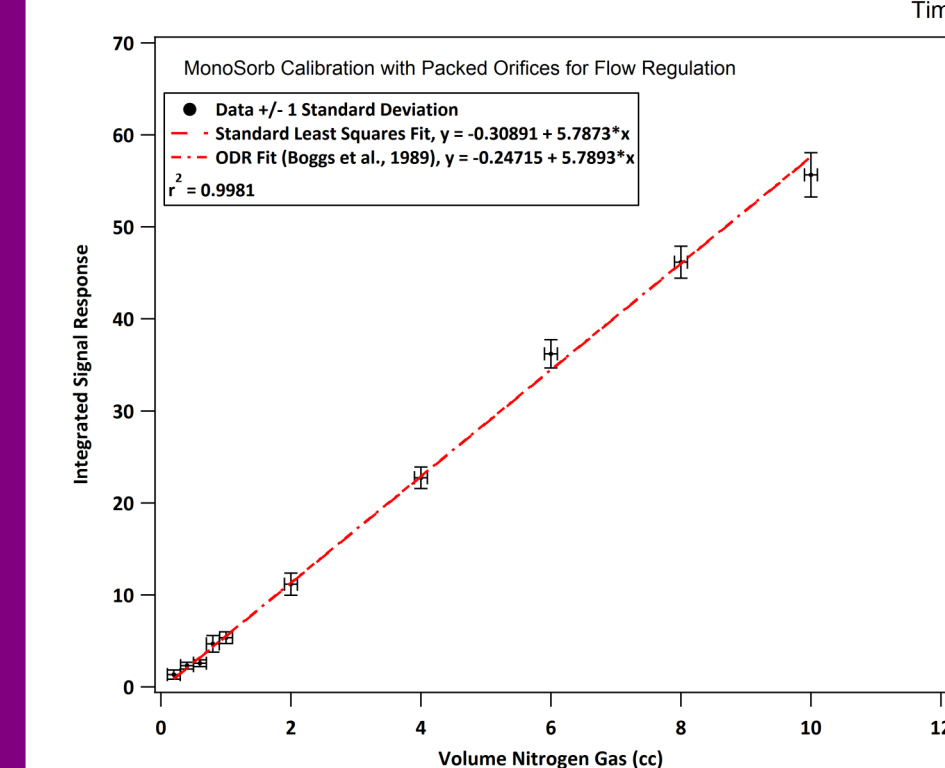
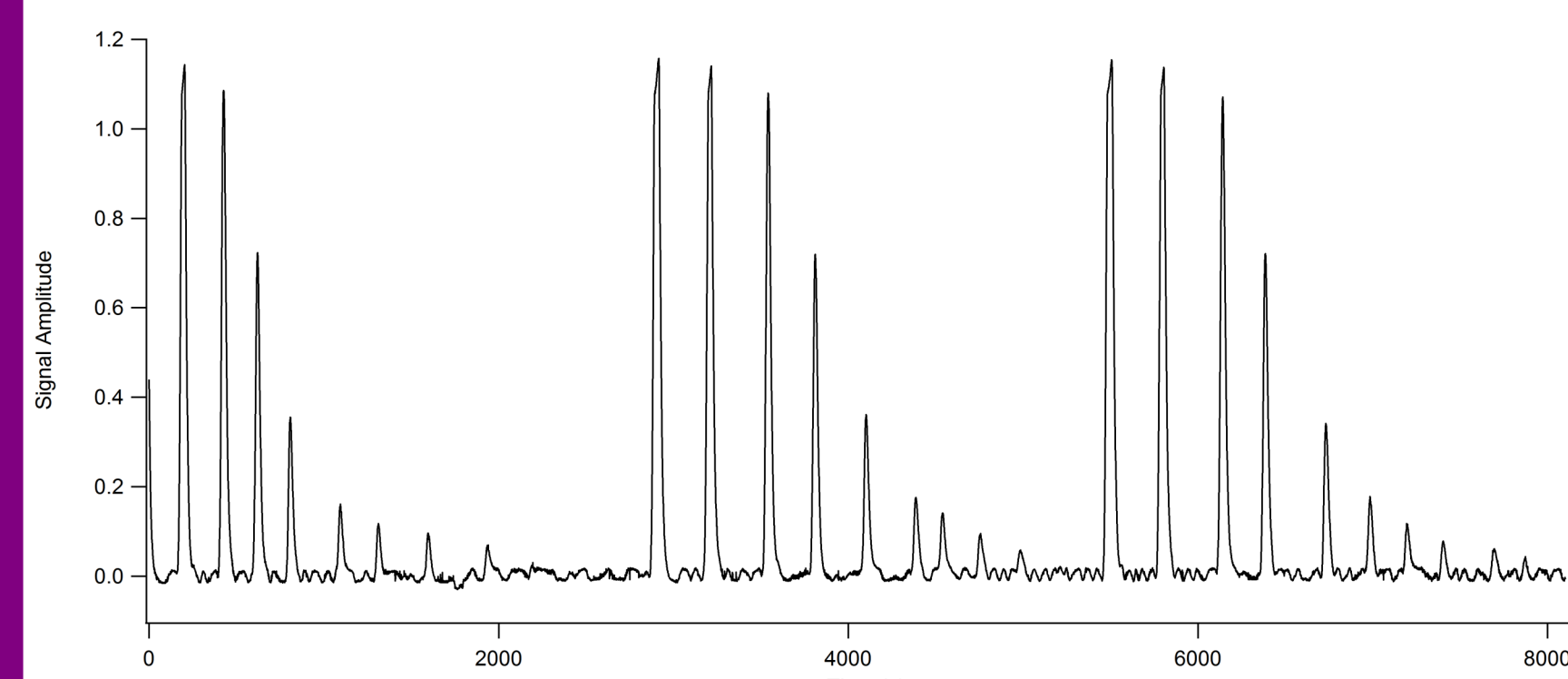
Sorbed nitrogen gas is proportional to sample surface area.



Instrument Characterization:

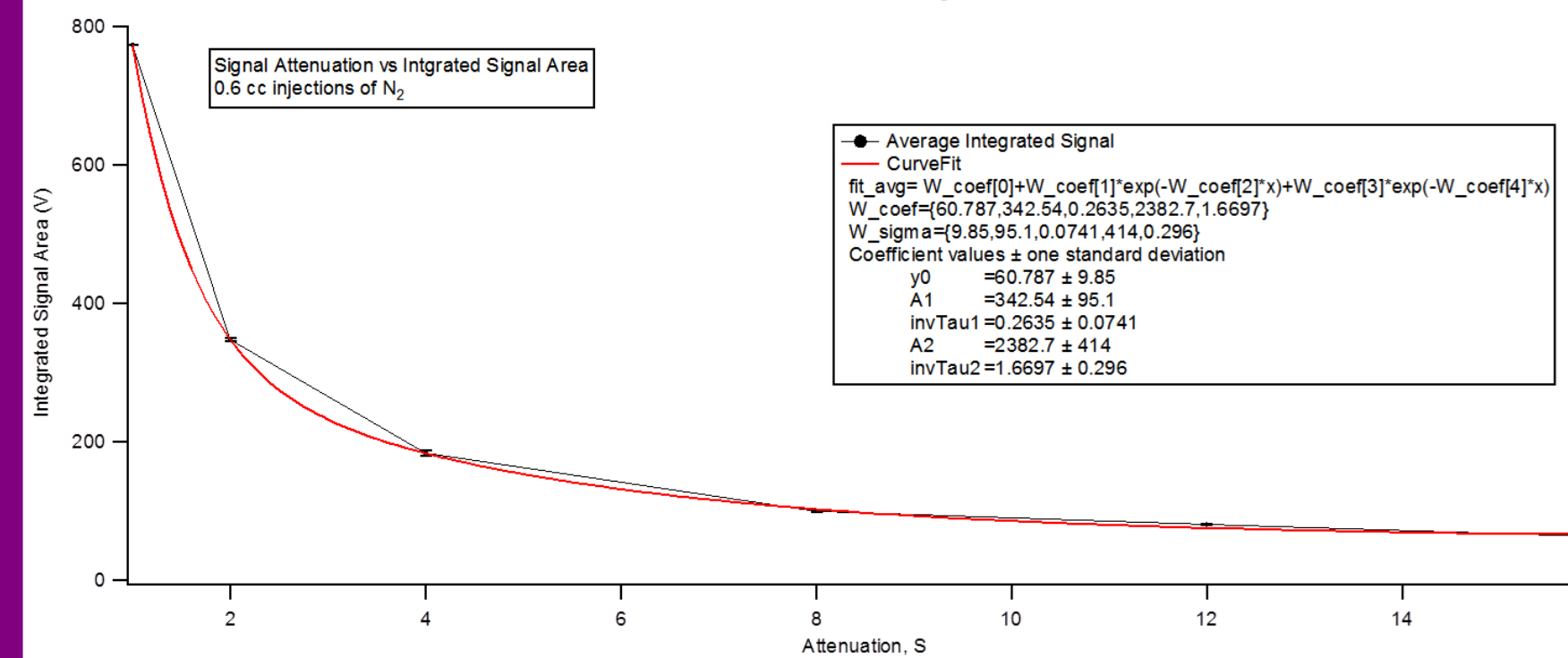
Calibration

Nitrogen gas injections, 10.0 to 0.2 cc.



Signal Attenuation Effects

0.6 cc N₂ injections at varying attenuations

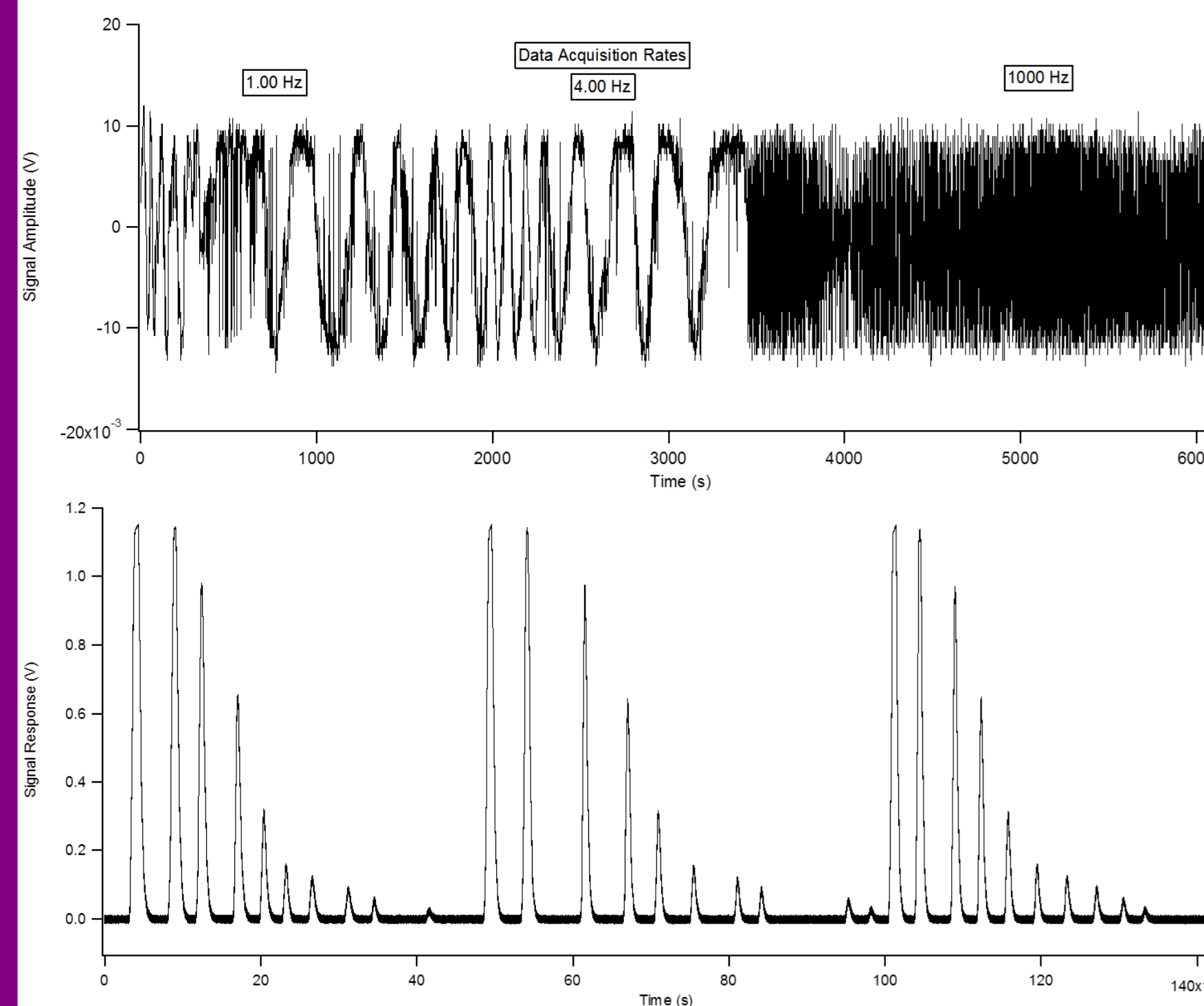


Temperature Settings

Percent of Maximum Voltage (%)	Temperature (°C)
10	30 ± 5
20	40 ± 5
30	70 ± 5
40	130 ± 10
50	170 ± 15
60	300 ± 20

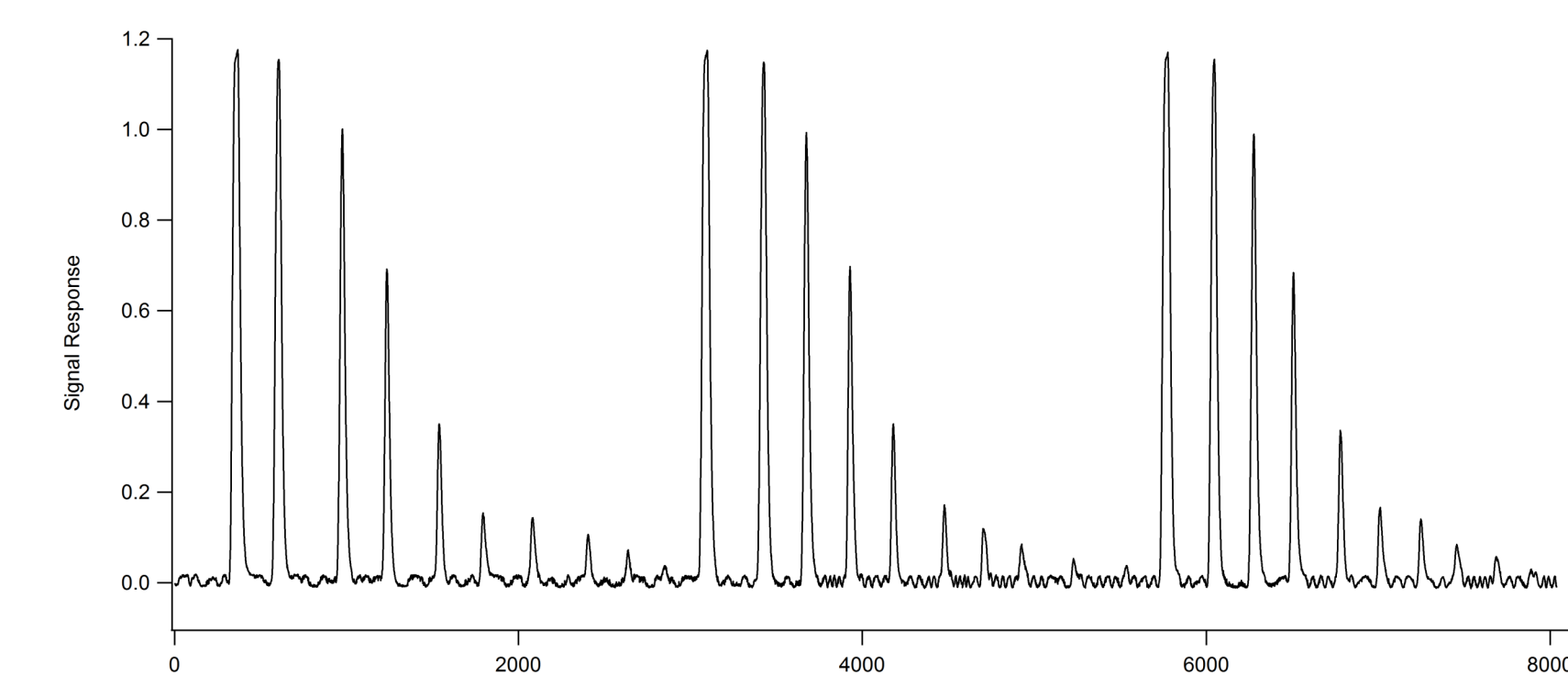
Data Acquisition Rates

Avoid aliasing by collecting at 1000 Hz.



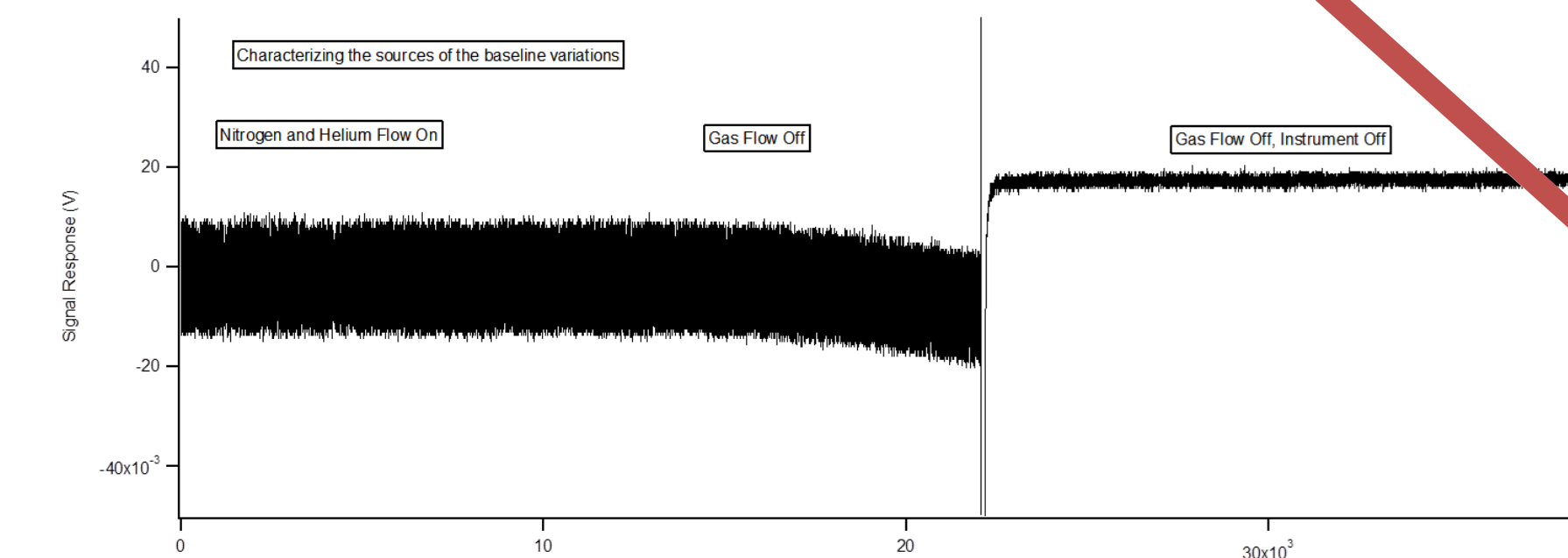
Addressing Baseline Variation:

Replace orifices with mass flow controllers



Baseline Variation STILL Present

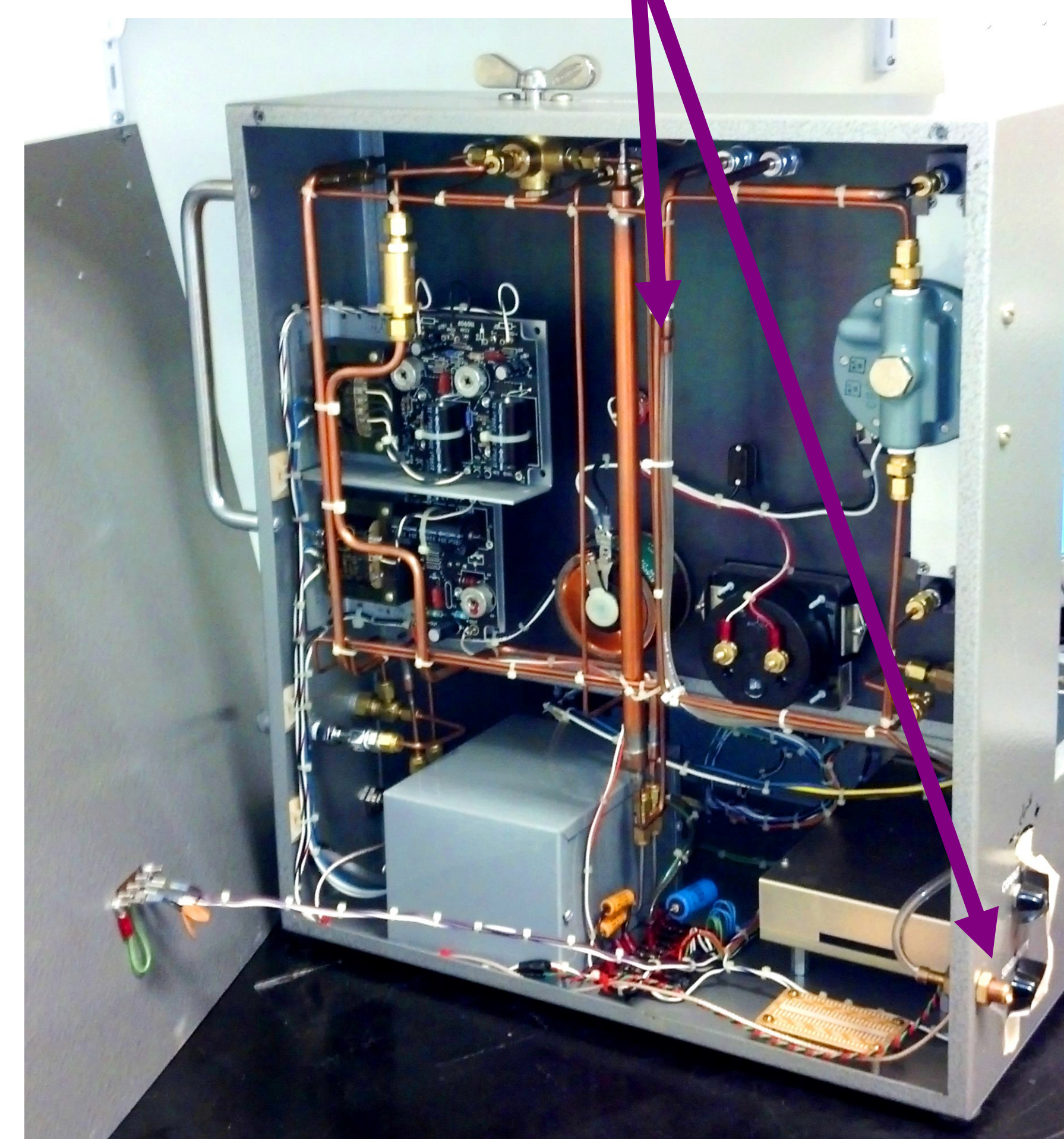
Where is this noise coming from?



Radio Frequency Interferences (RFI)

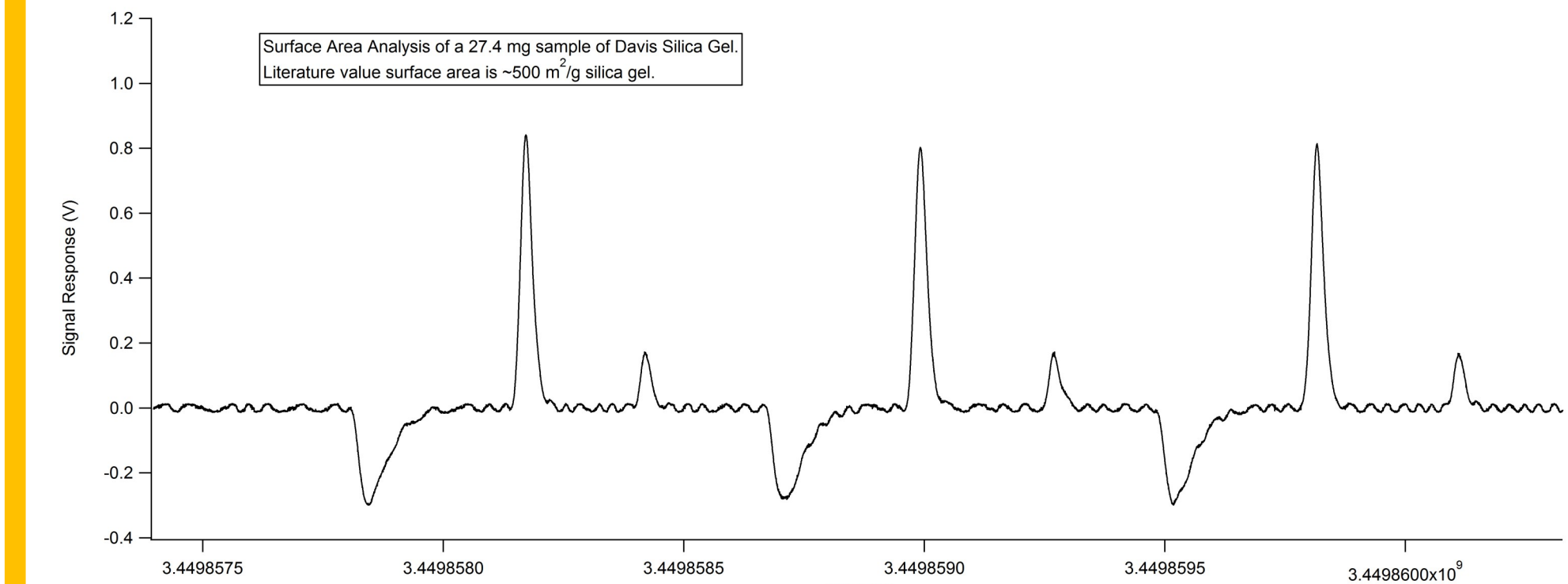
Venting

Make a proper gas vent



Experiment:

Determine the surface area of silica gel



$$St. = \left(\frac{P_{bar} \cdot N \cdot Ac.s.}{R \cdot T} \right) \left(1 - \left(\frac{P}{P_0} \right) \right) \left(\frac{I_s}{I_L} \right) Att.$$

$$Surface Area = \frac{St.}{m_{sample}}$$

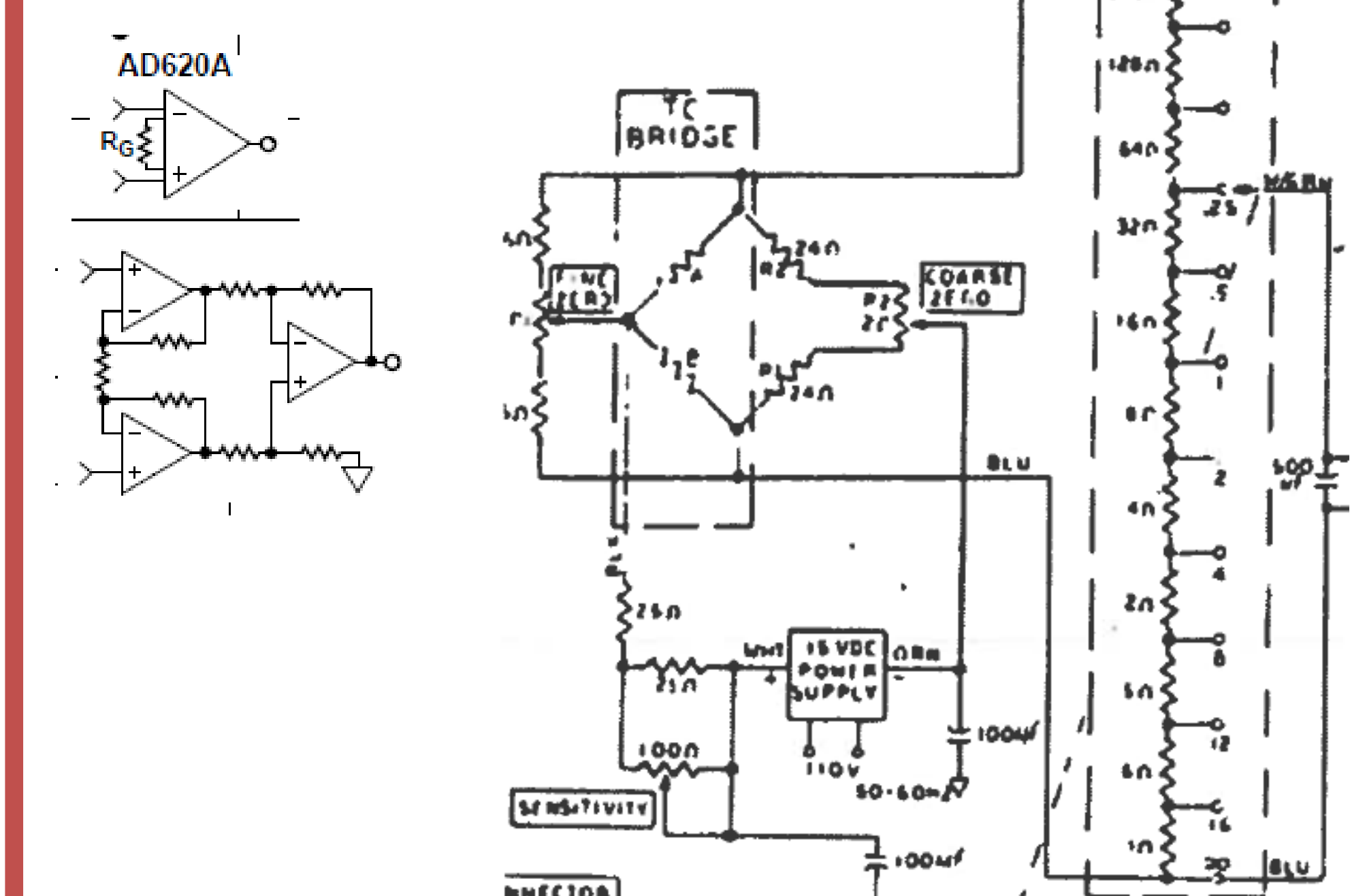
Where: St. (m²) is the total area of all the adsorbate molecules in one adsorbed monolayer or the total sample surface, P_{bar} (mm Hg) is the ambient atmospheric pressure, N (6.022 × 10²³ molecules/mole) is Avogadro's Number, Ac.s. (1.62 × 10⁻¹⁹ m²) is the adsorbate cross sectional area, R (82.396 cm³ mm Hg/(K mol)) is the ideal gas constant, T (K) is the temperature, P (mm Hg) is the mole ratio of N₂ to He gas ((N₂ vol/He vol) × (760 mm Hg)), P₀ (mm Hg) is the current ambient atmospheric pressure, I_s (V) is the integrated sample signal, I_L (V) is the integrated signal for a 1 cc nitrogen injection, Att. is the signal attenuator setting, and m (g) is the mass of the sample.

	Nitrogen from Silica	Nitrogen Injection (1cc)	St (m ²)	Surface Area (m ² /g)
Run 1	100.23	18.08	12.27	447.91
Run 2	99.83	17.59	12.56	458.50
Run 3	99.42	17.29	12.74	464.79
Average	99.83	18.00	12.52	457.07
Standard Deviation	0.40	0.40	0.23	8.53
Relative Uncertainty	0.40%	2.23%	1.87%	1.87%

St = total area of all the adsorbate molecules in one adsorbed monolayer or the total sample surface

The surface area of the 60-100 mesh size Davis silica was calculated to be **457.07 ± 8.53 m²/g**, compared to the literature value of **500 m²/g**.

Future Work:



Acknowledgements:

Thank you Dr. Joseph DiVerdi for providing the project instrument and for providing many hours of advice and assistance.

References:

- [1] J. Li, J. A. DiVerdi and G. E. Maciel, *Journal of the American Chemical Society* **2006**, *128*, 17093-17101.
- [2] MonoSorb Instrument Manual.