

Design of Diffusiometer  
for Aerogel Monoliths

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## ABSTRACT

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Aerogels' have great potential to be used as catalytic substrates and filters. To use them as such, we must first understand how gas flows through them. Diffusivity is a property of aerogels that relates the gas flow through a sample with the pressure gradient driving the flow. This project sets out to design an experimental apparatus (diffusimeter) and methodology that can be used to obtain diffusivity values of various kinds of aerogels. Adapting the method outlined in Stumpf et. al 1992[1], a diffusimeter was designed and built for aerogels at sub-atmospheric pressures. This device utilizes a control mass system rather than the control volume system approach which can be affected by small flow rates and significant gas leaks. The diffusimeter utilizes two volumes, each monitored via one Baratron® Type 122A absolute pressure transducer. By allowing a known amount of gas to diffuse into an aerogel monolith, we can obtain a pressure curve which can then be used to solve for the diffusivity. System analysis includes the consistency of the vacuum pump and gas regulator as well as the response time of the pressure transducers. Over a one-minute interval, the vacuum pressure fluctuated over a range of 50 Pa while the gas regulator charging the system fluctuated over a range of 300 Pa. The pressure transducer's time response was found to be 0.857 second. One test was performed on a silica aerogel monolith, confirming the effectiveness of the gas baffle implemented to protect the fragile samples during testing.

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Table of Variables

Name	Symbol	Units	Fundamental Units	Notes
Knudsen Number	K		m/m	Dimensionless Number
Mean Free Path	$\lambda$	m	m	
Characteristic Dimension	d	m	m	Equal to pore diameter for porous media
Throughput	$\dot{e}$	W	$\frac{kg * m^2}{s^3}$	Energy Flow
Diffusion Flux	J	molecules/m <sup>2</sup> /s	$\frac{\# \text{ of molecules}}{m^2 * s}$	
Energy Flux	J <sub>e</sub>	J/m <sup>2</sup> /s	$\frac{kg}{s^3}$	
Diffusivity	D	m <sup>2</sup> /s	m <sup>2</sup> /s	
Concentration	$\phi$	molecules/m <sup>3</sup>	$\frac{\# \text{ of molecules}}{m^3}$	
Energy Concentration	$\phi_e$	J/m <sup>3</sup>	$\frac{kg}{m * s^2}$	Equal to pressure in Pascals
Path Length	x	m	m	
Pressure	P	Pa	$\frac{kg}{m * s^2}$	
Volume	V	m <sup>3</sup>	m <sup>3</sup>	
# of Moles	n			
Universal Gas Constant	R <sub>0</sub>	J/mol/K	$\frac{kg * m^2}{s^2 * mol * K}$	
Temperature	T	K	K	
Kinetic Energy	e	J	$\frac{kg * m^2}{s^2}$	
Cross-Sectional Area	A	m <sup>2</sup>	m <sup>2</sup>	Perpendicular to flow direction
Sample Length	l <sub>s</sub>	m	m	Parallel to flow direction
Capillary Radius	R	m	m	
Capillary Length	l <sub>t</sub>	m	m	Equal to l <sub>s</sub>
Viscosity	$\mu$	kg/m/s	$\frac{kg}{m * s}$	
Average Pressure	p <sub>ave</sub>	Pa	$\frac{kg}{m * s^2}$	Across length of sample in direction of flow
Pressure Difference	$\Delta p$	Pa	$\frac{kg}{m * s^2}$	Across length of sample in direction of flow
Pore Radius	R <sub>p</sub>	m	m	

Molar Mass	M	kg/mol	$\frac{kg}{mol}$	
Time	t	s	s	
Porosity	$\phi$		$m^3/m^3$	Ratio of empty space to total volume of aerogel
Volume Ratio	$R_v$		$m^3/m^3$	Ratio of aerogel volume to total system volume

## Background & Introduction

### Aerogels

Aerogels are ultralight nanoporous ceramic materials that are comprised of 90-99% empty space by volume (Figure 1). The process of making an aerogel consists of polymerization and solvent extraction. First the solid linked network structure is created through the combination of chemical solutions. After polymerization, the solvent used in the process persists throughout the pores of the nanostructure; this is called a sol-gel. Extracting the solvent from the pores without collapsing the solid structure is difficult and requires complex extraction methods. Union College uses a patented method for solvent extraction called the Rapid Super-Critical Extraction method which uses a hot press to bring the solution past its supercritical pressure and temperature, thereby releasing it from the porous structure [2]. Afterwards, all that remains is the solid porous structure, an aerogel. Aerogels can be made of a variety of materials including carbon, metals, and metal oxides but the most common are silica aerogels [3].



*Figure 1: A silica aerogel monolith [3]*

Aerogels exhibit a number of unique qualities which make them particularly interesting candidates for use in a variety of applications. They exhibit an extremely low thermal conductivity due to their nanoscale porous structure which inhibits bulk fluid flow and limits conduction. Depending on the composition, some aerogels can even inhibit radiation. Their structure also allows them a large surface area to volume ratio which is ideal for catalytic substrates which require relatively large surface areas for reactions to occur. Lastly, with such small pathways for gas to slip through, aerogels have great potential as gas filters. Improved knowledge of how fluid flows through an aerogel is required before implementation in such fields.

## Flow Regimes

When characterizing fluid flow through some space with physical boundaries the Knudsen number, defined in equation (1), is an important non-dimensional number [3].

$$K = \lambda/d \quad (1)$$

The Knudsen number is a ratio of the mean free path of fluid molecules to some characteristic dimension of the space the fluid is moving through. Based on the value of the Knudsen number the fluid flow can be broken up into two distinct flow regimes, viscous and molecular. For the sake of this discussion, the flow regimes will be described in the context of fluid flow through porous media. For this purpose, the characteristic dimension is the average pore diameter.

The flow regime we most often encounter in everyday life is the viscous flow regime. This occurs when  $K \ll 1$ , i.e. when the mean free path is much smaller than the

pore diameter. Essentially, as long as the fluid is not very low density or the pore size is not significantly small, then the flow through the porous medium is viscous. This regime is modeled by and studied using the Navier-Stokes Equations which employs the continuum assumption. The assumption ignores the discrete nature of fluid molecules; however, it is reasonable because of the extensive interactions between fluid molecules.

The continuum assumption starts to break down when the mean free path of the molecules is on the order of the pore diameter; i.e. when  $K \approx 1$ . This situation arises when the fluid density is very low or when the pore size is very small. Such a situation arises in gas flow through porous media with significantly small pores. In this regime, the fluid molecules interact with the pore walls just as much as they do with each other [4]. The regime is a transition regime, and it occurs when both viscous and molecular flow play a role.

The molecular flow regime arises when  $K \gg 1$ , i.e. when the mean free path of molecules is larger than the pore diameter. In this regime, molecules' interactions with the pore walls dominate the collisions. Molecular flow, also known as Knudsen flow, is modeled and studied using rarefied gas dynamics [5]. The terms molecular flow regime and Knudsen flow regime will be used interchangeably.

For the purpose of this paper, we characterize fluid flow as energy throughput,  $\dot{E}$ , having units of Watts. This can be thought of as the energy flow rate rather than the traditional volumetric or mass flow rates. It is unclear why this is the convention in



diffusion and vacuum studies. However, through its use, the units work out and, in the end, we obtain the sought-after relationship.

An important parameter when dealing with fluid flow through a porous medium is the diffusivity of that medium. Diffusivity can be thought of as a proportionality constant relating fluid flux to the gradient that drives the flow. The relation between diffusivity and flow is found in Fick's First Law of diffusion [6],

$$J = -D \frac{d\varphi}{dx} \quad (2)$$

where  $J$  is the diffusion flux with units of number of molecules per square meter per second,  $D$  is the diffusivity with units of square meters per second,  $\varphi$  is the concentration of the fluid with units of number of molecules per cubic meter, and  $x$ , with units of meters, is the length across which the changing concentration is being considered. In simplified terms, Fick's First Law relates the rate of flux of fluid molecules to the gradient driving the flow using diffusivity. It is a relatively easy jump to use mass or moles instead of number of molecules as these values are discrete and proportional to the number of molecules through Avogadro's number and the molar mass of the fluid. However, under certain conditions, kinetic energy is also linearly related to these quantities. The moles of a gas can be related to the kinetic energy of that gas using the ideal gas law,

$$PV = nR_0T \quad (3)$$

where  $P$  is the pressure of the gas in Pascals,  $V$  is the volume of the gas in cubic meters,  $n$  is the number of moles,  $R_0$  is the universal gas constant in joules per mole per kelvin,

and  $T$  is the temperature of the gas in Kelvin. Assuming constant temperature,  $C = R_0T$  is also a constant. The average kinetic energy,  $e$ , of an ideal gas is given by

$$e = PV \quad (4)$$

and therefore,

$$e = Cn \quad (5)$$

By assuming constant temperature, kinetic energy is proportional to number of moles, and we justify the use of energy rather than moles, mass, particles, or any other discretized property of gases as the diffusing substance.

We define our energy flux,  $J_e$ , through a sample of aerogel as

$$J_e = \dot{e}/A \quad (6)$$

where  $A$  is the cross-sectional area of the aerogel sample in square meters taken perpendicular to the flow direction. Using equation (4), the concentration of energy,  $\varphi_e$ , in a particular volume of space of an ideal gas held at constant temperature is the pressure of that gas; i.e.

$$\varphi_e = \frac{e}{V} = P \quad (7)$$

where  $\varphi$  is energy concentration in joules per cubic meter. Substituting equations (6) and (7) into equation (2) yields

$$\frac{\dot{e}}{A} = -D \frac{\Delta P}{l_s} \quad (8)$$

where  $l_s$  is the length of the aerogel sample parallel to the gas flow.

A common model for porous materials is the capillary tube model which treats the porous medium as a bundle of capillary tubes aligned in the direction of flow [7]. The radii of these tubes are commonly represented using a distribution of radii, or an effective radius,  $R_{eff}$ . When in the viscous flow regime, fluid flow through cylindrical tubes can be modeled using Hagen-Poiseuille flow. Assuming steady state, laminar flow, and incompressible gas, throughput of a single capillary tube is defined by

$$\dot{e} = \frac{\pi R^4}{8\mu l_t} p_{av} \Delta p \quad (9)$$

where  $R$  and  $l_t$  are the radius and length of the tube respectively,  $\mu$  is the viscosity of the fluid, and  $p_{av}$  is the average pressure across the entire length of the tube [8].

Substituting equations (8) into (9) we obtain an expression for the diffusivity of the aerogel in the viscous flow regime;

$$D = \frac{\pi R^4}{8\mu A} p_{av} \quad (10)$$

Note that the length of the capillary tube,  $l_t$ , is the same as the length of the sample,  $l_s$ , as sample length was taken in the flow direction.

As the Hagen-Poiseuille model operates under the continuum assumption, it is unfit to describe gas flow when in the molecular flow regime. The diffusivity of porous media when in this flow regime is defined with Knudsen diffusivity [7];

$$D = \frac{4}{3} R_p \sqrt{\frac{2R_0 T}{\pi M}} \quad (11)$$

where  $R_p$  is the pore radius in meters,  $R_0$  is the universal gas constant in Joules per mole per Kelvin,  $T$  is the temperature in Kelvin, and  $M$  is the molar mass of the gas in kilograms per mole.

Comparing equations (10) and (11) we see that the diffusivity of porous media is proportional to the average pressure across them when gas flow is in the viscous flow regime but has no dependence on pressure when in the molecular flow regime.

Assuming the transition region from molecular flow to viscous flow is small, then, according to the capillary model, the only parameters needed to define the linear relationship between diffusivity and average pressure are the effective pore radius, the viscosity of the gas, and the sample area through which the gas is flowing. By knowing this linear factor, and the average pressure at which diffusivity begins to scale with pressure for a particular gas, we should, theoretically, be able to predict the diffusivity of that gas through a particular kind of aerogel for any average pressure. Had we a collection of these diffusivity parameters for various kinds of gases and aerogels, we could predict and potentially model gas flows through aerogels.

## Literature Review

The most common method for measuring the diffusivity of aerogels has been a control volume approach. These methods involve inducing a flow through an aerogel and measuring the pressure on either side of the sample.

One of these such tests was run by Hosticka et al. 1998 [9]. Their method involved the incremental increase of upstream pressure while maintaining a constant

downstream pressure. The flow rate was measured via a flow meter while the differential pressure across the sample as well as the downstream pressure were monitored via electronic pressure transducers. The experimental set up is presented in Figure 2.

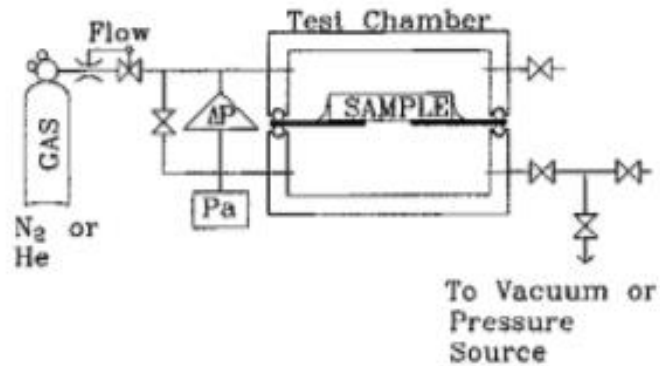


Figure 2: Experimental setup from Hosticka et al. 1998 [9]. Control volume approach.

The sample is sealed into the test section on a perforated partition using sealing wax. The quality of the seal was ensured through aerosol tests. The pressure difference across the sample varied from 1 to 4 kPa with the last step in pressure difference being used to compute the diffusivity. Tests were run for absolute pressures ranging from 6 to 200 kPa and across this range a transition from flow looking more molecular to a flow looking more viscous can be seen at very low pressure. These results are presented in Figure 3.

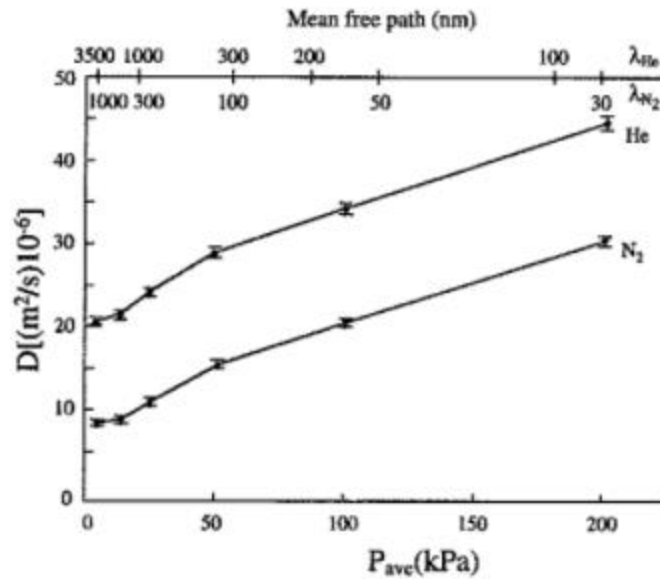


Figure 3: Results from Hosticka et al. 1998 [9]. Diffusivity of silica aerogels over a range of average pressures from 6 to 200 kPa for both nitrogen and helium gas.

Looking at Figure 3, diffusivity values tend to stay constant for very low pressures and then deviate into a more proportional relationship with average pressure across the sample. It is noted later in the paper that this non-characteristic relationship between diffusivity and average pressure could be due to a very large pore size distribution which inhibited pure viscous flow at high average pressures and pure diffusive flow at very low average pressures. Average pore size for the silica aerogel samples was found to be 24 nm however, there was significant pore sizes detected throughout the sample ranging from 2 to 200 nm. Leaks detected in the system were calculated to be as high as 0.5  $\mu$ l/s which makes a difference when measuring flowrates around 20  $\mu$ l/s.

A similar experiment was performed with partially densified silica aerogels in Beurroies et al. 1995 [10]. It should be noted that these partially densified aerogels have

average pore sizes around 15 to 25 nm with maximum pore size never exceeding 35 nm. Their experimental set up is presented in Figure 4.

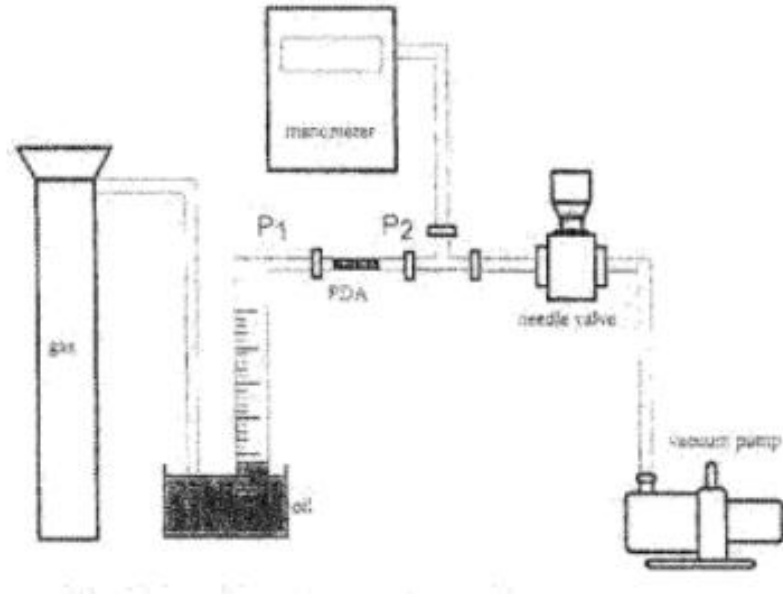


Figure 4: Experimental set up from Beurroies et al 1995 [10]. Control volume approach.

Looking at Figure 4, the tube to the left of the partially densified aerogel (PDA) sample is filled with gas. The downstream pressure is regulated by the needle valve. The gas flow is measured by the change in volume of graduated cylinder multiplied by the pressure P<sub>1</sub>. This experiment was conducted for average pressures ranging from 507 to 1013 mbar. The results of the experiment run for two different types of PDA is presented in Figure 5.

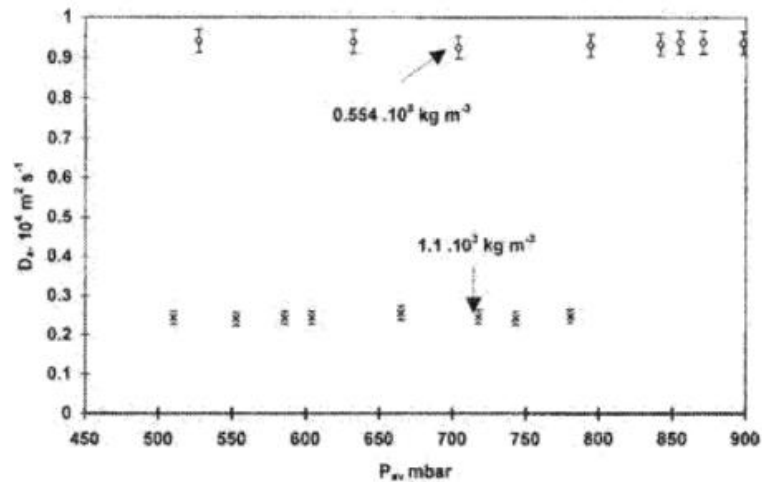


Figure 5: Results from Beurroies et al. 1995 [10]. The diffusivity values of two different types of densified aerogels stay constant over an average pressure range from 507 mbar to 1013 mbar. Flow in every test is in the molecular flow regime.

Looking at Figure 5, the diffusivity values remain relatively unchanged with increased average pressure. This means that these average pressures all produced molecular diffusive flow. This is due to the small pore sizes that were produced through the densification process.

Both of the methods presented require sealing the aerogel into a chamber in some way so that gas does not leak around the sample into the low-pressure side. In both cases, high precision instrumentation is required to obtain significant measurements.

## Previous Work

The last attempt by Union's Aerogel Lab to experimentally obtain diffusivities of aerogels utilized a common method for these types of measurements. Most recently worked on by Adam Ashcroft '18, the device seals an aerogel into a gas channel where a constant pressure of gas is introduced to one side while the other side outlets to



atmospheric pressures [11], [12]. Assuming the aerogel is fully sealed on the edges, the gas flows directly through the aerogel and out the other side. Using a flow meter located at the outlet of the device, the flow rate moving through the aerogel can be determined. By applying a range of pressures across a sample, a pressure-flow rate relationship can be obtained which can then be used to extract the relationship between diffusivity and average pressure. The main chamber where the aerogel would be located is presented in Figure 6.



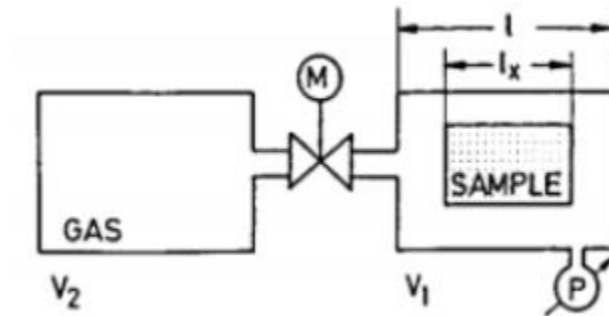
*Figure 6: Most recent attempt by Union's Aerogel lab to measure gas flow through aerogels. Gas enters through the tubing on the top right of the device and exits through the tube on the bottom left where it goes through a flow meter.*

Obtaining accurate data proved non-trivial. The acrylic chamber was not air tight due to the solvent sealer which was used to “glue” the acrylic casing together. When dealing with a restricted flow such as the steady state conditions experienced in this device, even the smallest gap or pathway through the chamber walls can cause problems. Gas

also tended to leak through the seal into the bottom chamber rather than go through the aerogel. Incomplete sealing caused many of the results to be flawed or inconclusive. The difficulty experienced while trying to seal the aerogel into place was one of the main motivations for finding a different method for obtaining the diffusivities of aerogels.

### “New” Method

Using the method outlined by Stumpf et al. in 1992 [1], the current work looks to employ the use of a closed system rather than an open one. Instead of a continuous flow of gas, a predetermined amount of gas is introduced to a single monolithic sample of aerogel. The setup is presented in Figure 7.



*Figure 7: The general setup of the device used by Stumpf et al. 1992 to obtain the diffusivities of silica aerogels at different pressures [1].*

The valve, denoted “M” in figure 3, connects a gas reservoir to a closed chamber containing a monolithic silica aerogel sample. The chamber containing the aerogel is brought to a pressure just below the average pressure of interest. The valve is opened for a short period of time allowing more gas to flow into the chamber. This causes the

pressure in the chamber to jump up a small amount, typically around 10 mbar, or 1 kPa. Initially, the gas fills only the empty space surrounding the aerogel. Over time, the gas diffuses into the aerogel itself. This continues until pressure equilibrium is achieved, and no further diffusion occurs. The pressure within the aerogel chamber is continuously recorded throughout the process. By solving the differential equation for diffusion in a porous medium, presented in equation (12), with the proper boundary and initial conditions, an ideal equation for the pressure-time relation can be found.

$$\frac{\partial p}{\partial t} = \frac{D}{\phi} \Delta p \quad (12)$$

This equation is used to model the gas diffusion into the aerogel sample over time. The  $\phi$  in equation (12) is the porosity of the aerogel. Porosity is defined as the ratio of empty space, or total pore volume, to the total sample volume and is used to describe how much air space is contained in a porous medium. Using the geometry of the device as well as the known properties of the aerogel to set boundary and initial conditions, an ideal pressure-time curve for the experimentally obtained data is found and subsequently fit to the data set. The resulting curve is then used to extract the diffusivity. An example of how the pressure in the chamber changes over the course of one run is presented in Figure 8



Figure 8: Example of how the pressure in the chamber changes over the course of a single run [1]

Looking at Figure 8, the pressure in the chamber peaks when the additional gas initially envelopes the aerogel, before any diffusion has occurred. It then decreases over time until stabilizing once the gas has completely diffused into the sample. Each run produced a single diffusivity value which was characteristic of the aerogel at the average pressure that the aerogel experienced. For example; the chamber containing the aerogel is filled with nitrogen until the chamber reaches a steady 992.5 mbar. After allowing some time for the gas to settle, the valve is opened and more nitrogen flows into the chamber until it reaches 1002.5 mbar. Over time the pressure drops as the gas diffuses into the sample and eventually reaches a steady state of 997.5 mbar. The ideal pressure curve is fit to the data and a diffusivity of  $11 \times 10^{-6} \text{ m}^2/\text{s}$  is extracted. Therefore, the diffusivity of this particular silica aerogel at 1000 mbar is  $11 \times 10^{-6} \text{ m}^2/\text{s}$ .

The difficulty in implementing this method comes from the instrumentation required to accurately execute and measure all the steps. In order to regulate the amount of gas that flows into the chamber very quickly, an accurate flow meter with automatic shutoff is required. Measuring small pressure changes in rapid succession

requires a highly accurate pressure transducer with a small response time. Both of these tools are very expensive and unavailable with our current resources. Therefore, the methodology has been modified to accommodate for the current project budget.

Opening and closing of the valve between gas reservoir and the aerogel chamber by hand in a very short time span would be neither accurate nor repeatable. Therefore, a middle step is introduced which involves charging a separate volume with a predetermined amount of gas. This volume is called the pressure tank. Once the pressure tank is charged, a valve connecting it to the aerogel chamber is opened and the additional gas is allowed to expand and envelope the sample. The pressure within the total volume, now consisting of the pressure tank and the aerogel chamber, is recorded as the gas diffuses into the sample.

It is assumed that the diffusivity stays constant over the course of a single test. However, using equation (10) it is known that the average pressure across the sample is directly proportional to diffusivity of the aerogel. By keeping the pressure in the chamber very close to the pressure of interest over the entire course of a single test, Stumpf et al. made the assumption that the diffusivity does not change significantly. As we increase the pressure drop to accommodate for instrumentation accuracy, this assumption becomes less and less reasonable. At this time, it is unclear the extent to which this will affect the data however, it must be kept in mind when analyzing the results of the tests. Increasing the pressure drop will also decrease the amount of time it takes for the gas to fully diffuse into the aerogel; the larger the pressure difference, the faster the gas will flow into the aerogel. Based on the results of Stumpf et al., it took

approximately 50 seconds for the pressure to stabilize at equilibrium conditions after introducing only 1 kPa of additional pressure. It is unclear the effect a larger pressure jump will have on stabilizing times. For this reason, the time response of the chosen pressure transducer must be rapid enough to obtain several data points for a relatively fast flow rate. Without a sufficient amount of data, the accuracy of the curve fit will be too low to draw any significant conclusions from.

A problem with this method arises when adsorption effects are taken into consideration. Adsorption is the accumulation of molecules on the surface of a solid caused by adhesive forces between atoms. The gas molecules do not penetrate the surface but rather create a film-like buildup on any surface. This effectively takes those molecules out of the gas phase thus changing the total amount of gas molecules that contribute to the pressure in the system. This is particularly important for aerogel testing because of its large inner surface area. Depending on the amount of adsorption that occurs over the course of a single run, the final pressure of the system could be significantly different than what was originally anticipated. The adsorption effects of the test gas on the aerogel surfaces were thought to have an effect on the diffusivity values obtained by Stumpf et al. The effects of adsorption have been studied before [7] and further investigation into the effects will be conducted once the device is operational.

## Problem Definition

We wish to build a device that allows us to execute the method of diffusivity coefficient measurement described above. The main parts include the pressure tank,

the aerogel chamber, and a valve between the two. Two pressure transducers are required; one in the aerogel chamber to record the data and one in the pressure tank to charge it with the correct amount of gas. The gas reservoir must also be attached to the pressure tank in order to charge it. Therefore, another valve to seal the pressure tank from the gas reservoir once it is charged is required. We want to see the difference in diffusivity behavior between molecular and viscous flow. Based on the findings of Stumpf et al, that transition in diffusivity behavior happens at average pressures below atmospheric. Therefore, a vacuum pump must be connected to the system. This connection also needs a valve to seal off the pump while running the tests.

## Design Description

A schematic of the apparatus is presented below in Figure 9;

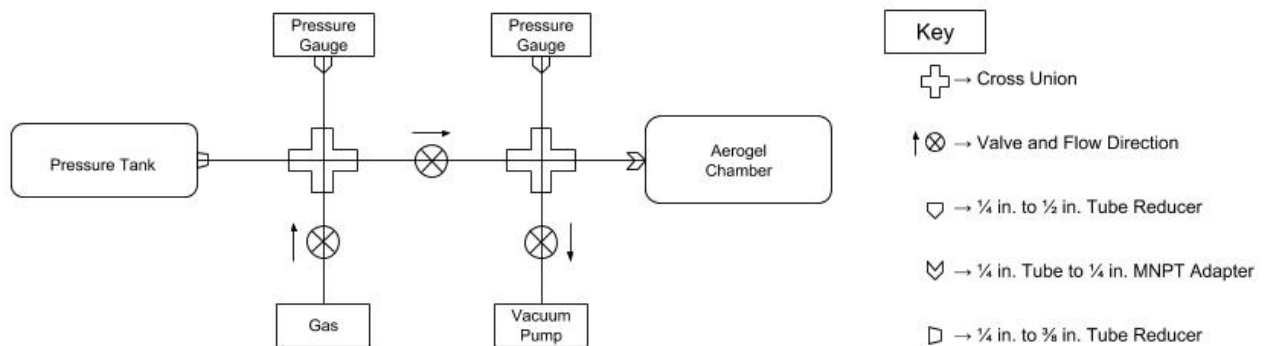


Figure 9: Schematic of the experimental apparatus. Lines connecting each part are 1/4" copper tube.

The completed design is presented in Figure 10;

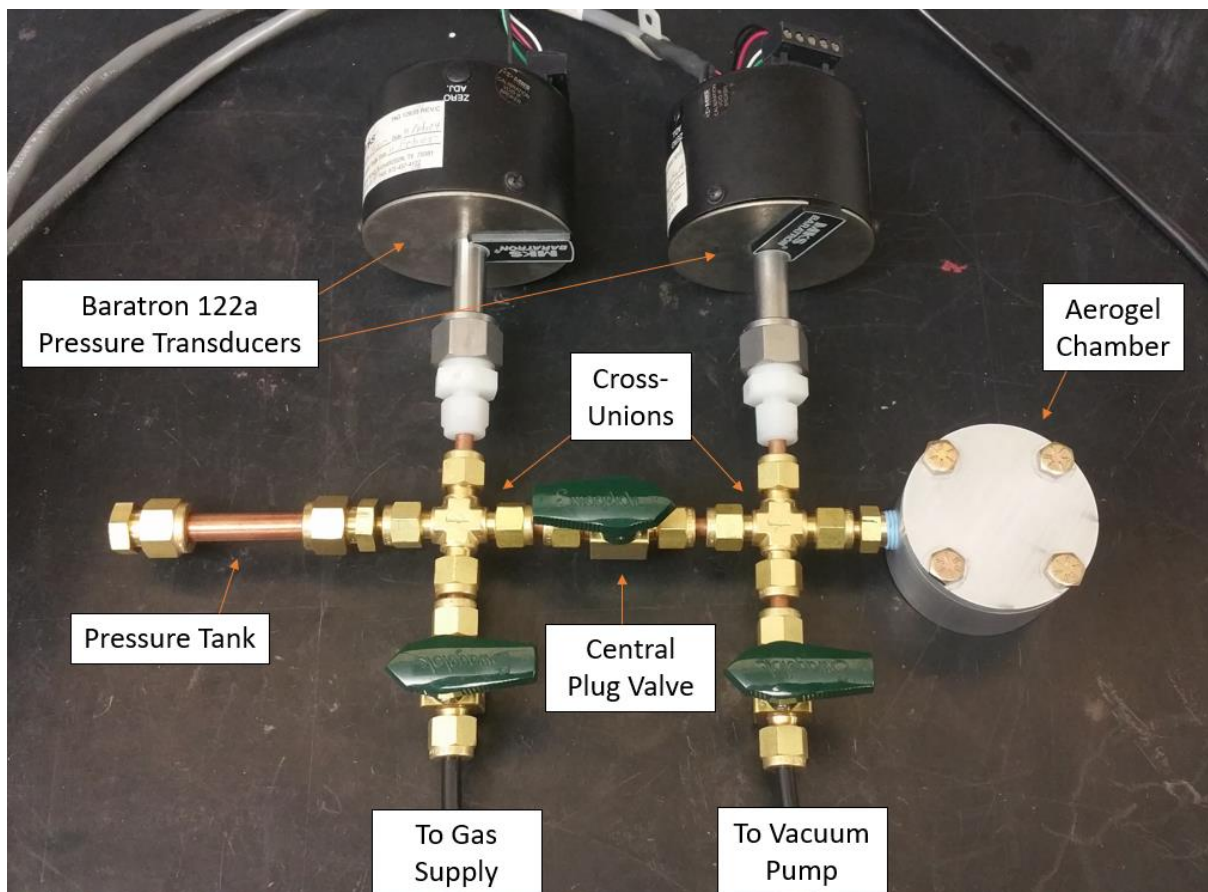
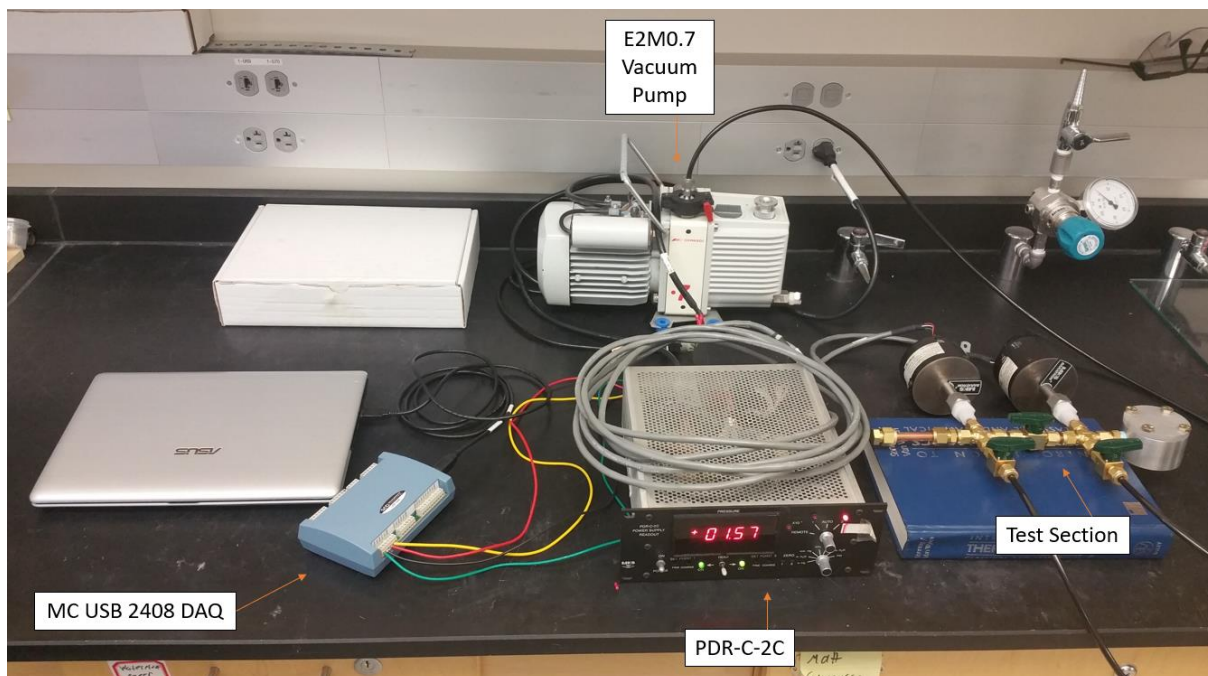


Figure 10: (Top) Test set up. (Bottom) Test Section



All the connections in the design are made with  $\frac{1}{4}$ " outer diameter (OD) brass tube fittings. The two 4-way cross connections are  $\frac{1}{4}$ " Swagelok tube fittings. As throttled flow was unneeded, all three valves are  $\frac{1}{4}$ " Swagelok plug valves. The valves have built in O-rings that ensure a proper seal when closed. After preliminary calculations using the pressure transducers available to us at the time, it was found that the volume referred to as the "pressure tank" did not have to be relatively large ( $\sim 3.24$  mL). This allowed us to create the tank out of  $\frac{3}{8}$ " OD brass tube and an end-cap. The pressure tank connects to the main body of the device using a  $\frac{3}{8}$ " to  $\frac{1}{4}$ " Swagelok reducer. The pressure transducers that were used in the final design are connected to the main body of the device using Teflon  $\frac{1}{2}$ " to  $\frac{1}{4}$ " Swagelok reducers. The stainless-steel nut used to connect the pressure transducer to the Teflon reducer was used due to time constraints. The vacuum chamber was machined out of 6061 cold-rolled aluminum. The chamber is sealed using a Series 2-221 O-ring that is compressed by the lid which is secured using four  $\frac{1}{4}$ "-20 bolts.  $\frac{1}{4}$ " female NPT threads which were machined into the side of the chamber so that a  $\frac{1}{4}$ " tube to  $\frac{1}{4}$ " male NPT adapter could be used to connect the chamber to the main body of the device. The vacuum pump that is currently being used is an Edwards Model E2M0.7 Rotary vacuum pump. It was taken from a broken laboratory instrument that was sitting in the Aerogel Lab. The regulator being used (connected to the gas supply, not shown in Figure 10) is a Matheson model 1L-580.

The silica aerogels used in Stumpf et al. 1992 experienced a transition from molecular to viscous flow at an average pressure of approximately 15 kPa absolute [1]. Based on these results, it was decided that we would test at average pressures ranging

from <10 to 100 kPa absolute. With this in mind, multiple absolute pressure transducers were considered but the PX409-030AUSBH Omega pressure transducer (dubbed the Very Low Pressure Transducer, or VLPT) which was already owned by Professor Bruno's lab was chosen. This transducer was to be connected to the vacuum chamber side of the device (the total volume to the right of the central plug valve in Figure 10) and was to be the main transducer used to obtain data for the idealized curve fit. The pressure transducer on the pressure tank side was only to be used to charge the pressure tank to the correct pressure. Therefore, we decided to use a different PX409 series pressure transducer that was available in Professor Bruno's lab. Its range was 0-150 psi absolute and it was dubbed the Low Pressure Transducer, or LPT.

After designing the device with this pressure transducer in mind, two Baratron 122a pressure transducers, Teflon adapters, and an MKS Type PDR-C-2C power supply and digital readout were donated to the project by former Professor Huisman of the Union College Chemistry Department before he left at the end of the Fall 2017 term. One transducer has a range of 0-10 torr (0-1.33 kPa) while the other has a range of 0-1000 torr (0-133.3 kPa). Both pressure transducers have an accuracy of  $\pm 0.5\%$  (of reading) meaning we can measure near vacuum condition pressures with more accuracy than the VLPT which had an accuracy of  $\pm 0.08\%$  of a 0-207 kPa range. With this large increase in accuracy at low pressures, the Baratron 122a pressure transducers were chosen over the VLPT as the primary means of data collection.

The device was designed, however, with the VLPT and LPT as the intended pressure transducers. As the uncertainty of the VLPT ( $\pm 16.56$  kPa) is large compared to

the low average pressures we plan to test for, we wanted to increase the pressure drop we observed thus making the uncertainty less significant over the course of a single run. Assuming ideal gas, we determined the driving factor that effects the pressure drop to be a ratio of volumes between the closed system volume and the volume of the aerogel. The ratio is defined by

$$R_V = V_A / V_{Total} \quad (13)$$

where  $R_V$  is the volume ratio,  $V_A$  is the total volume of the aerogel, and  $V_{Total}$  is the total volume contained in the device consisting of the pressure tank and aerogel chamber. Using Boyle's law, the pressure drop was found to respond to the volume ratio as follows;

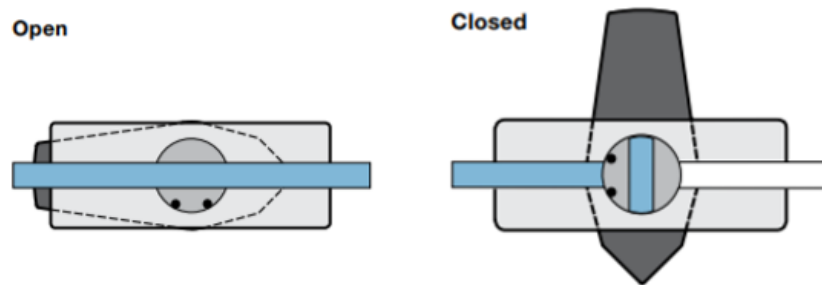
$$P_i - P_e = P_i R_V \quad (14)$$

where  $P_i$  is the initial pressure in the aerogel chamber before diffusion occurs, and  $P_e$  is the equilibrium pressure the chamber will reach once diffusion is complete. Using equation (14), it was found that, as  $R_V$  increases for a specified initial pressure, the pressure drop also increases. As the pressure drop is dependent on the initial pressure, testing very low pressures, such as those needed to induce molecular flow (<15 kPa), will produce small pressure drops. Therefore, we want to increase  $R_V$  as much as we can so that the transducers accuracy does not become a limiting factor at these low-pressure tests. As the effects of increasing the pressure drop on stabilization time and diffusivity accuracy are unknown, the only value that limits the pressure drop, and therefore drives design parameters, is the maximum size of an aerogel monolith. After

talking to Professor Ann Anderson of the Mechanical Engineering Department, it was estimated that the largest silica aerogel monolith that could be created using Union's hot press is a 1-inch diameter by 1-inch tall cylinder. This brings the total aerogel volume to approximately 13 mL. A  $R_v$  value of 0.5 was chosen as a middle ground between a small total system volume and a small pressure drop. This allowed for a total system volume of 26 mL, of which, 20 mL would be used for the aerogel chamber side and 6 mL would be used for the pressure tank side. Note that 26 mL is not the actual system volume as the Baratron 122A pressure transducers added unaccounted for volume after the device had been designed.

As the volume of the system was critical to the experiment, the inner volumes of all the individual parts need to be known. Swagelok's sales drawings are publicly available and show certain nominal dimensions for all parts. The drawings can be found in the "Downloads" section on each part's individual webpage. The gas pathways through every part are cylindrical. Therefore, in order to calculate the total volume contribution from each part, the inner diameter and total length of the pathway within every part is required. When determining the volume contribution of every part, the inner volumes of the female connecting ports, both NPT and Swagelok Tube Fittings, were ignored. This is because, when fully connected, these ports will be filled with tubing or male NPT connections. The diameter and total path length were obtained for the cross-union connectors using the sales drawing [13]. The nominal total volume contribution was calculated to be 0.76 mL. The sales drawing of the male NPT adapter was also used to calculate a nominal inner volume of 0.583 mL [14]. The drawing for

the female NPT adapters does not include the nominal length of the female NPT port. Therefore, the port length had to be measured from a sample part [15]. The port length was found to be 20.63 mm. Using this measurement, the nominal inner volume was found to be 0.276 mL [16]. The volume contribution of the quarter turn plug-valves depended on what side the O-ring was on. A layout of the internal mechanism is presented in Figure 11.



*Figure 11: Bottom up view of the internal plugging mechanism of a quarter turn plug valve. The black dots are the cross section of the O-ring [17].*

The side of the valve that has the higher pressure is the side with the O-ring when closed, this is to ensure no flow occurs. As this valve will be located between the pressure tank and the aerogel chamber, the O-ring will be on the side of the pressure tank. A sample quarter turn plug valve was taken apart and measured with calipers to obtain the inner diameter (4.8mm) and the length (12.5mm) of the trapped section [15]. Using these measurements along with the sales drawing [18], the volume contribution from the side with the O-ring was calculated to be 0.110 mL. For the purpose of this work, the volume trapped in the valve when closed is added to the volume of the side without the O-ring. This is because, once the valve is opened, that trapped volume will

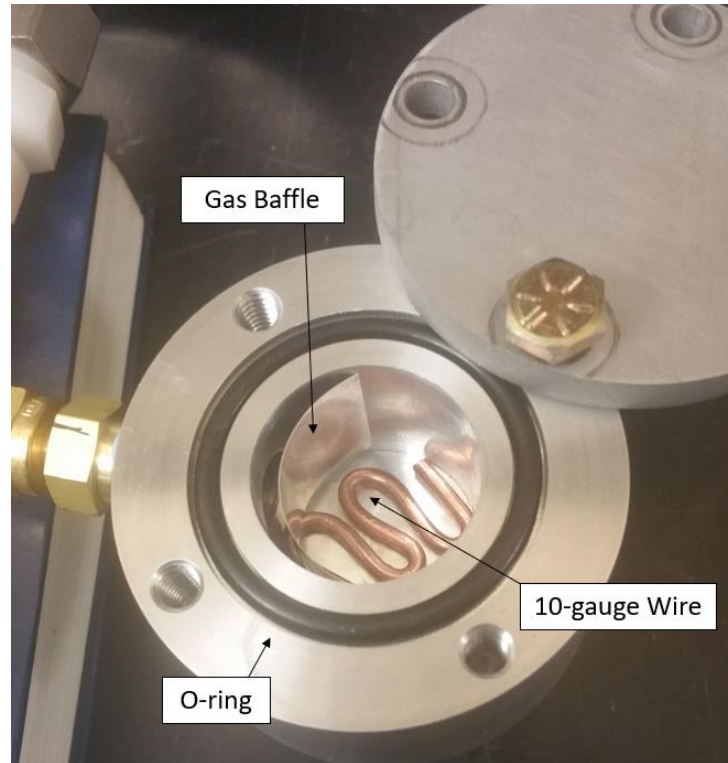
be added to the total volume along with the aerogel chamber volume. Therefore, the trapped volume is part of the aerogel chamber side. The total volume contribution from the non-O-ring side was calculated to be 0.337 mL. For design purposes, the lengths of the ¼" OD copper tubes that connected the valves to the cross-unions were chosen to be 1.375" which gave each tube a total volume of 0.573 mL. After the device was built, the actual lengths varied and each tube was evaluated individually for the calculation of the actual system volume. The total volume contribution from the connectors, valves, and tubing on the pressure tank side of the device was calculated to be 2.76 mL. As the total pressure tank volume was initially designed to be 6 mL, a pressure tank with 3.24 mL of inner volume was needed. Rather than manufacture such a small pressure tank out of raw material, it was decided to use a 2.7-inch length of 3/8" OD tube instead. This brought the total pressure tank side volume to approximately 6 mL. The total volume contribution on the aerogel chamber side was calculated to be 2.63 mL, which left 17.37 mL for the aerogel chamber itself. A comprehensive table containing the volumes calculated for each component is available in Appendix A.

After building the device and implementing the use of the Baratron 122a pressure transducers, the volume of the system was evaluated. The pressure transducers along with their required adaptive ports added a large amount of volume to the system. Using the sales drawings as well as measurements taken from the device, the total volume contained in the pressure tank was found to be 11.77 mL and the aerogel chamber side was found to be 25.64 mL. This does, however, change the volume ratio we initially intended for to 0.66. As stated earlier, the accuracy of the

Baratron transducers is much greater than the VLPT. Therefore, the necessity for large pressure drops has become less of a concern.

The aerogel chamber [Appendix B] was designed to have two added features; the first being a way to raise the aerogel so that it does not sit directly on the bottom of the chamber. If the aerogel does rest flat on the bottom of the chamber, there is a chance that the bottom face of the aerogel will not experience the same diffusion rates at the top. As the ideal pressure curve assumes equal gas access to all faces, this could potentially compromise the results. As a solution, a small piece of 14-gauge wire is press-fit to the bottom of the chamber with all edges touching the inside wall of the chamber. Looking at Appendix B, the height of the inside of the vacuum chamber is  $1/8''$  taller than the proposed aerogel height. 14-gauge wire is approximately  $1/16''$  in diameter, thus causing the aerogel to sit approximately in the middle of the chamber. For the preliminary testing, 10-gauge wire was used at the bottom of the aerogel chamber as that was easily available at the time. The other feature is a gas baffle. Depending on the pressure difference between the charged pressure tank and the aerogel chamber, the gas could flow into the aerogel chamber at relatively high flow rates which, if allowed to make direct contact with the aerogel monolith, could push the aerogel into the wall of the chamber. As the aerogels are fragile they could potentially crack, if not break, when pushed into the wall, effectively compromising the test. For this reason, we employ a thin sheet of aluminum bent around the aerogel facing the entrance of the chamber to redirect the gas around the sides of the cylinder. This thin sheet is taken from any aluminum soda can and cut to be  $1.125''$  tall and approximately

1.5" wide, effectively allowing it to disperse the initial influx of gas. The inside of the aerogel chamber is presented in Figure 12.



*Figure 12: Contents of the aerogel chamber for preliminary test.*

A breakdown of the parts bought from Swagelok throughout this project, including some that were not used in the final design, is presented below.



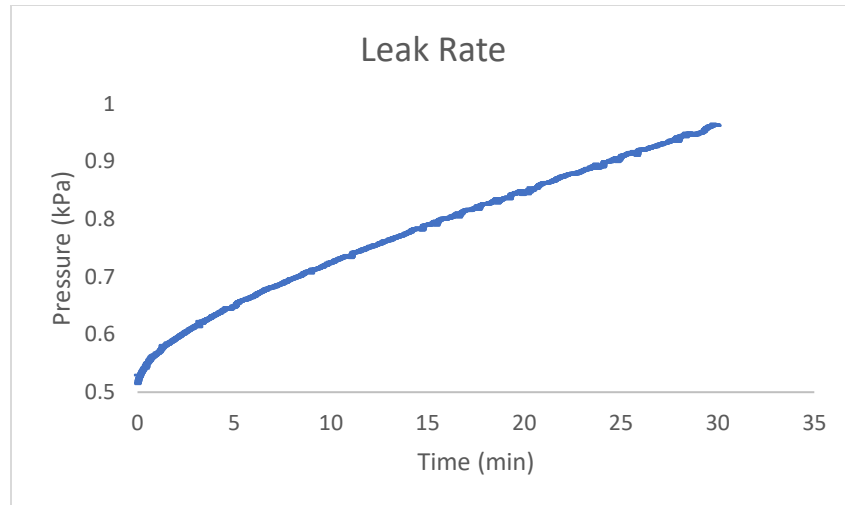
Description	Part Number	Quantity	Total
Cross Union (1/4" Swagelok Tube Fitting)	B-400-4	2	\$ 30.00
Quarter Turn Plug Valve (1/4" Swagelok Tube Fitting)	B-4P4T	3	\$ 123.90
Female Tube Adapter (1/4" OD Tube --> 1/4" FNPT)	B-4-TA-7-4	2	\$ 6.20
Male Tube Adapter (1/4" OD Tube --> 1/4" MNPT)	B-4-TA-1-4	1	\$ 2.60
Swagelok Tube Fitting Reducer (1/4" OD Tube --> 3/8" Swagelok Tube Fitting)	B-600-R-4	1	\$ 5.50
Swagelok Tube Cap (3/8" Swagelok Tube Fitting)	B-600-C	1	\$ 2.90
Brass Ferrule Set (3/8" Swagelok Tube Fitting)	B-600-SET	10	\$ 9.30
Stainless Steel Nut (1/2")	SS-812-1	2	\$ 9.06
		Total Price	\$ 189.46

## Analysis

Note that the pressure transducers and the PDR have not been calibrated therefore any pressure reported from tests may not be completely accurate. Specifically, the two 122a transducers do not read the same pressure when connected to the same closed system; there seems to be a 0.425 kPa offset between the two. Also, the analog voltage coming from the PDR does not scale correctly with what is being read by the pressure transducers. However, both transducers read close to 0 kPa when a vacuum is pulled from the system and a reasonable atmospheric pressure is read from the 1000 Torr transducer when open to atmospheric conditions. Therefore, for the purposes of the preliminary test, the pressures are accurate enough.

After completing the assembly of the device, we tried to obtain a more accurate volume measurement. The goal was to fill the system with water and then obtain the mass of the water by using a digital scale. This proved difficult as the passageways within the device are small and therefore the surface tension of the water was enough to prevent the liquid from flowing into the device. As a compromise for the time being, a simple pressure test was conducted to see how accurate the initial volume calculations were. First, the system was evacuated, then the pressure tank was filled with nitrogen gas until it reached a pressure of 140.37 kPa. The middle valve connecting the pressure tank to the aerogel chamber was opened and gas expanded into the chamber until an equilibrium pressure of 43.59 kPa was reached. Assuming ideal gas and no temperature change, the volume ratio of the total system volume to the pressure tank volume was calculated to be 3.220. Based on the volume calculations performed after the device was assembled, that same volume ratio is calculated to be 3.178. Although the ratios are close in value, the exact measurements of each volume (pressure tank and aerogel chamber) are required for the ideal curve fit. A more accurate calculation or experiment will need to be conducted before diffusion results can be obtained.

Ideally the system, when closed, would be completely leak proof. However, the real world is never ideal and this system has leaks. In order to quantify how much the system leaked, the system was evacuated and the pressure inside the system was monitored for 30 minutes. The results of that test are presented in Figure 13.

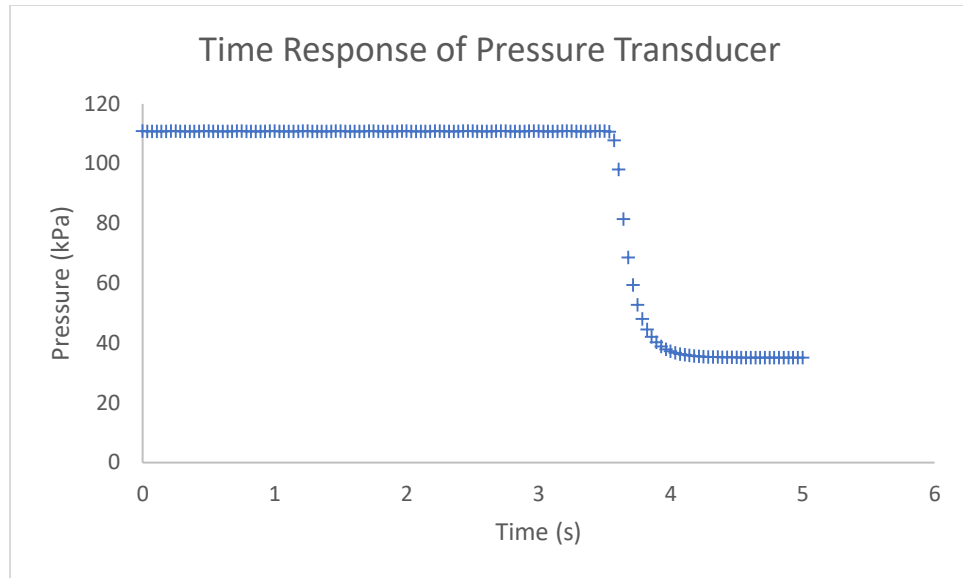


*Figure 13: Leak Rate Test on the evacuated system volume. Test time; 30 min. Sampling Rate; 3 Hz*

As you can see from Figure 13, the highest leak rate was found to be at the lowest pressure. This leak rate is approximately 0.12 kPa/min. Assuming each test, starting from pulling a vacuum to settling at equilibrium pressure, will only take approximately 5 minutes, the pressure drop can be tailored to make this leak rate insignificant. Still, in order to obtain the most accurate results, the leak should be sealed. The most likely location for the leak is in the connection between the pressure transducers and the ¼" OD copper tube. Swagelok tube fittings are meant to be used by compressing a ferule on to a material softer than what the tube fitting is made out of. In other words, a Teflon tube adapter is not meant to connect to a stainless steel or copper tube. That being the case, it is likely that the connection between the Teflon adapter and the metal tubing is not sealed properly. As the stainless-steel tube is part of the transducer, it is recommended that, instead of buying a new metal adapter that will permanently deform the tube, the connection point between the tube and Swagelok

fitting should be sanded and cleaned as best as possible. This will, hopefully, create a more leak proof seal between the two materials.

In order to try and determine a response rate of the Baratron pressure transducers, a simple pressure test, like the one conducted for the volume calculation, was preformed while the PDR was connected to an MC USB-2408 series data acquisition system. A sampling rate of 28 Hz was used to monitor the pressure inside the system as the central valve was opened allowing the gas from inside the pressure tank to expand into the previously evacuated aerogel chamber. There was no aerogel in the chamber during the test so the time elapsed from the valve opening to the system reaching equilibrium is only due to system fluctuations (pressure waves), and the delayed response time of the pressure transducer. The results of this test are presented in Figure 14.

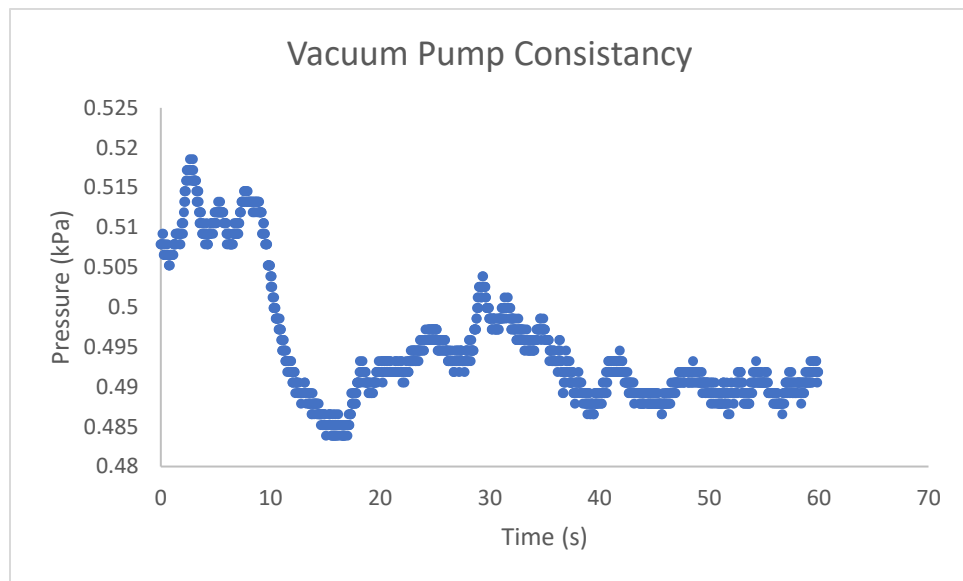


*Figure 14: Time Response of pressure transducer test. The pressure tank was filled with nitrogen gas to a pressure of 110.7 kPa. The central valve was then opened and the gas expanded to an equilibrium pressure of 35.0 kPa. Sampling Rate; 28 Hz*

Based on results from Stumpf et. al. 1992 [1], it took approximately 60 seconds for the gas to diffuse into the aerogel using a pressure drop of approximately 10 mbar (1 kPa). Looking at Figure 14, it takes approximately 0.857 seconds for the transducer to read equilibrium pressure. This is very long considering the fact the we intended to increase the pressure drop and, as a result, decrease the time it will take for gas to diffuse into the aerogel. Further testing is recommended to confirm the response time, however, if this is any indication of the response time of the Baratron transducers, then alternative pressure transducers should be found and used for testing.

In order to get a more complete diagnostic of how the system is running, two additional tests were run using the same MC USB-2408 DAQ system. The first test was to see the consistency of the vacuum pump. As the E2M0.7 is a rotary vacuum pump, it

is expected to see some small variation while the pump is running. It should, again, be noted that the pressure transducers have not been calibrated and, therefore, may not be displaying the correct pressure. The purpose of this test was to see how the pressure changed while the pump was running, not to see how good of a vacuum the pump can provide. The results of this test are presented in Figure 15.

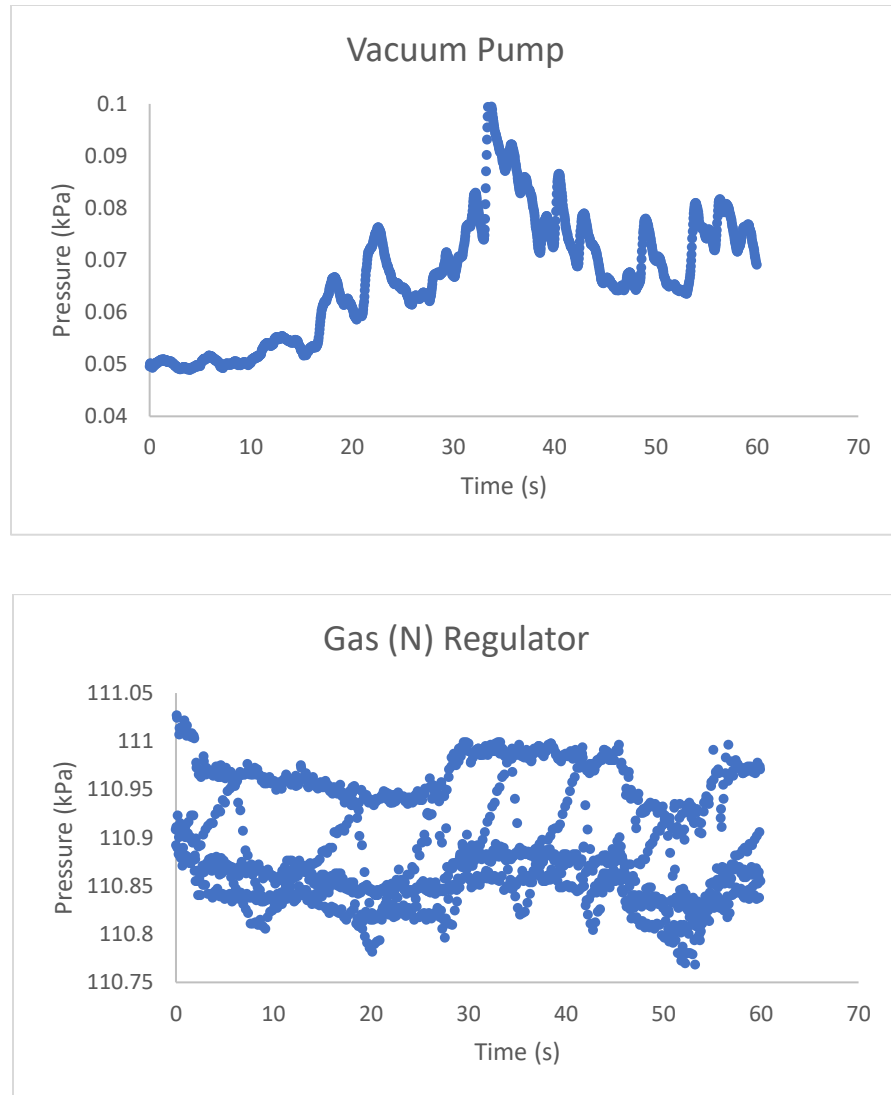


*Figure 15: Pump Consistency Test. The system was monitored via the 1000 Torr pressure transducer as the pump was actively pulling a vacuum. Test Time; 1 min. Sampling Rate; 28 Hz*

The test was only run for a minute at 28 Hz and the pressure fluctuated over a range of 0.04 kPa. This may not be too concerning depending on how good of a vacuum the pump actually makes. Both the 10 Torr and the 1000 Torr transducers read the same pressure fluctuations however the actual pressure readings were offset by approximately 0.425 kPa. The 10 Torr transducer gave pressure readings around 0.075 kPa. Once the pressure transducers are calibrated, this test should be redone for a

longer time and higher response rate in order to determine how critical the fluctuations actually are.

The second test was conducted while the vacuum pump was pulling a vacuum from the aerogel chamber side and the nitrogen gas tank was open to the pressure tank side via the gas regulator. Theoretically the aerogel chamber should be at or close to 0 kPa consistently and the pressure tank should be at the regulated pressure consistently. The purpose of this test was to see how precisely we can control the pressurized pressure tank and the evacuated aerogel chamber before closing them off and running the diffusivity test. Again, the actual pressure values are not necessarily correct, nor are they important for the test. The results of this test are presented in Figure 16.



*Figure 16: Charging and Evacuation Control Test. The aerogel chamber was actively being evacuated via the vacuum pump and the pressure tank was charged to a "steady" pressure via the gas regulator. Test Time; 1 min. Sampling rate; 28 Hz*

Looking at Figure 16, over the course of 1 minute, the vacuum pump fluctuated approximately 0.05 kPa while the gas regulator fluctuated approximately 0.3 kPa. The vacuum pump experienced relatively large drops in performance (spikes in pressure) at the 34, 40, 49, and 55 second marks, which could be due to the nature of the rotary pump. These spikes would not be as concerning if the average vacuum stayed constant

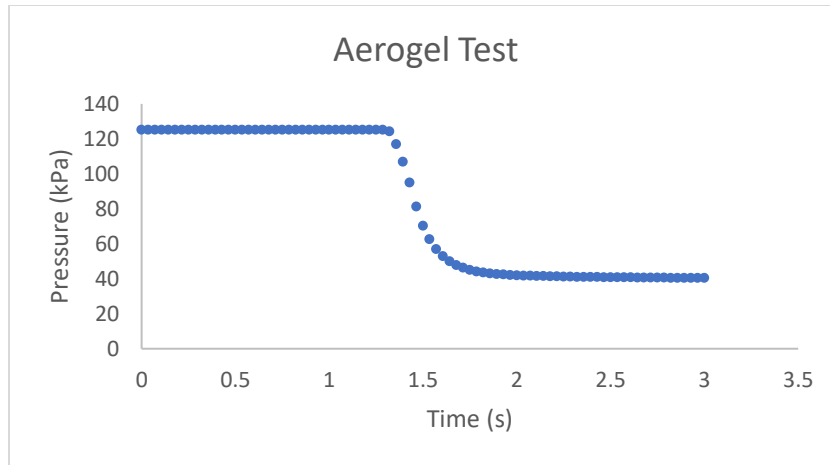


over the course of test however, it only held its best and most consistent vacuum for approximately 10 seconds and then declined from then on. The gas regulator, on the other hand fluctuated more sporadically about a relatively consistent average the whole time. Depending on how precisely the pressure in the pressure tank needs to be set for a single test, this fluctuation may be an issue. This test should be kept in mind when deciding on experimental parameters as they may greatly affect the accuracy a single experimental run can have on initial conditions.

It should also be noted that the gas regulator on the nitrogen gas tank that is currently being used only allows for pressure regulation above atmospheric conditions. In order to charge the pressure tank to a pressure less than atmospheric, an alternative pressure regulation system is needed.

## Testing

A single aerogel test was conducted. The test was run the same way the time response test was. First the system was evacuated, then the pressure tank was charged with nitrogen gas to a pressure of approximately 125 kPa. The central valve was then opened and the gas expanded into the aerogel chamber where it diffused into the aerogel. The aerogel that was used was a cylindrical monolith 1.8 cm tall with a 1.8 cm diameter. The results of that test are presented in Figure 17.



*Figure 17: First Aerogel Diffusion Test. Nitrogen gas from the pressure tank expanded into the previously evacuated aerogel chamber where it surrounded the aerogel monolith and then diffused in. Sampling Rate; 28 Hz*

Comparing Figure 17 to Figure 14, there was very little observable change in system behavior. The pressure drop was very large and therefore we expected a fast diffusion. This is most likely why there is no observable change between the gas surrounding the aerogel and the diffusion into the aerogel. As the aerogel that we tested was much smaller than the aerogel monoliths we will be using with this device, there was a lot of room for the monolith to move around inside of the chamber. As there was a 125 kPa pressure difference between the pressure tank and the aerogel chamber we know the initial flow rate of gas going into the aerogel chamber was relatively very fast. However, after the completion of the test, the aerogel was still completely intact. This is taken as proof that the gas baffle works as intended.

## Conclusions

### Next Steps

The system is not ready to start being used to measure diffusivity values. It is not far off, however, from being able to obtain a proof of concept measurement. One of the first things that needs to happen to continue working towards the goal of obtain diffusion constants is acquiring a new regulator for the nitrogen gas. The current one is too inaccurate for the purposes of this project and it cannot provide pressures below atmospheric which is absolutely necessary as the transition from molecular to viscous flow occurs at average pressures well below 100 kPa. Another solution is to modify the current regulator in some way, however, it may prove easier to acquire a new one.

It is also very important to make sure the Baratron 122a pressure transducers are fast enough to measure the quick changes in pressure the system will experience during testing. First, the time response test discussed earlier in the report [Figure 14] should be redone with a higher sampling rate; maybe 100 Hz. This should give a good idea of the actual time response of the pressure transducers. If it turns out that the response time for pressure changes is larger than 20 msec, then it may be advantageous to try and acquire different, faster pressure transducers. The VLPT and the LPT are fast enough and operate in the appropriate pressure range. The higher uncertainty should not prevent the experimenter from getting decent proof-of-concept diffusivity measurements. However, if it turns out that the Baratron transducers are fast enough, the next step would be to calibrate the pressure transducers as well as the PDR.

If the leak rate turns out to be critically affecting the data then it is recommended that the Teflon adapters be taken off, and the connection point sanded and cleaned. After talking with a representative from Swagelok [15], the best way to make this type of mis-matched material connection is to make the point of connection between the ferule set and the metal tubes as clean and smooth as possible. This may include sanding the pressure transducers' stainless-steel tube port with higher and higher grit sand paper until the surface is clear of all scratches. This should also be done on the copper  $\frac{1}{4}$ " pipe.

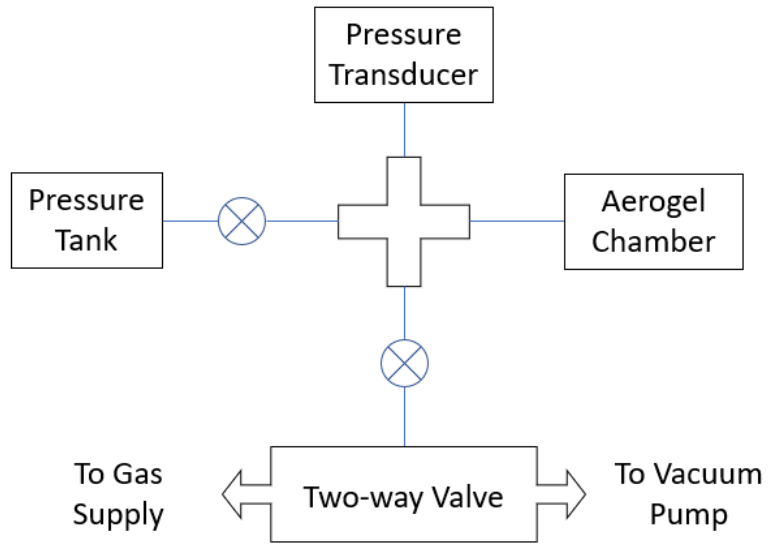
In regards to the actual tests that will be run with the purpose of extracting diffusion constants, it may be advantageous to try and get the pressure drops that the aerogel will experience to be as small as possible. Assuming the time response of the pressure transducers is fast enough, the Baratron transducers will be able to detect, with a relatively high amount of precision, pressure drops on the scale of 1 kPa. Therefore, in the interest of assuming the diffusivity of the aerogel stays constant over the course of a single run, the tests should be conducted more similar to that of Stumpf et al. Rather than evacuate the aerogel chamber every test, it should be charged with gas to a pressure just below the pressure of interest and the pressure tank should be charged with gas to a pressure large enough that, after the central valve is opened but before the gas has begun to diffuse into the aerogel, the system pressure is just above the pressure of interest. For example, if we want to obtain the diffusivity for a certain aerogel with regards to nitrogen gas at a pressure of 50 kPa, we charge the aerogel chamber with nitrogen gas to a pressure of 48 kPa. The pressure tank is then charged

with enough gas so that, immediately after the valve is opened but before diffusion occurs, the pressure in the system is 52 kPa. The system will come to equilibrium somewhere around 50 kPa. This causes the aerogel to be under approximately 50 kPa of pressure over the course of the entire test and therefore allows the initial assumption that the diffusivity stays constant to be more reasonable. At this time, it is unclear what pressure the pressure tank would need to be charged to in order to actually run a test at those conditions but, using ideal gas laws and isentropic expansion, it should not be overly complicated to solve for an equation that outputs the required charging pressure with only known parameters as inputs.

Another thing that must be evaluated before any diffusion constants can be obtained is the ideal pressure-time curve that will be fit to the experimentally obtained data. Currently the equation is unknown however it should not be impossible to find and/or solve for.

### Future Work

One of the biggest advantages of this design is that the aerogel chamber and the pressure tank can both be easily modified without disrupting the entire system. Once the system is proven to work, it may be worth it to refine the system such that it only requires one pressure transducer. This may allow the device to be more compact and free up space in the lab for other equipment. One possible modification is presented in Figure 18.



*Figure 18: Possible new device set up. More compact and only uses one pressure transducer*

This set-up only requires one pressure transducer and allows the pressure tank volume to be easily defined. This is desirable as it is currently non-trivial to measure the complete volume inside of the device.

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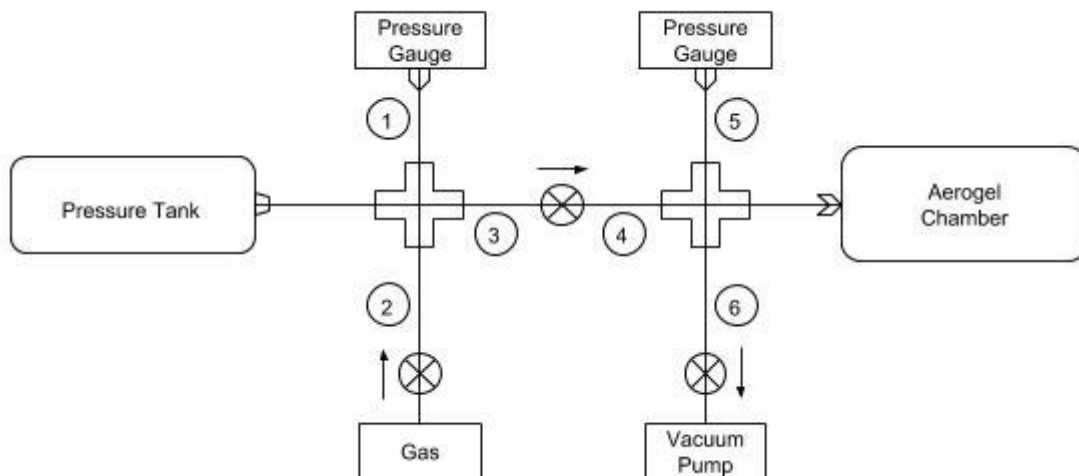
## Appendix A

Component	Volume Contribution (mL)
Pressure Tank (3/8 in. tube)	4.895
3/8" to 1/4" tube reducer	0.357
Cross-Union Connector	0.818
1/2" to 1/4" tube reducer	0.161
Baratron® 122a pressure transducer	3.845
Quarter Turn Plug Valve (O-ring side)	0.1104
Quarter Turn Plug Valve (Non-O-ring side) *	0.3366
1/4" tube to 1/4" MNPT Adapter	0.5388
Aerogel Chamber **	18.325
1/4" Copper Tube (1)	0.525
1/4" Copper Tube (2)	0.460
1/4" Copper Tube (3)	0.492
1/4" Copper Tube (4)	0.492
1/4" Copper Tube (5)	0.525
1/4" Copper Tube (6)	0.492

\*Includes the volume trapped inside the valve when closed

\*\*Includes the volume of the FNPT hole not filled by the MNPT adapter

NOTE: Refer to schematic below for specific assignment of 1/4" copper tubing





Appendix B

