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Design, Fabrication, and Programming of an Acoustic Property Measuring System for Ultrasound in the MHz Range

May 5th, 2016

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Abstract

Accurate measurement of acoustic material properties for various microfluidic devices is essential for optimal device design. In this project, a continuation of the design of a novel microfluidic channel for *in situ* removal of photocurable polydimethylsiloxane (PDMS-a silicone), an apparatus was designed to measure the speed of sound through various liquids and solids. The apparatus was designed to hold piezoelectric transducers (pulser/receiver) and sample using the program AutoCAD™, fabricated on a three axis CNC mill, and tested by coupling input and output signals to an oscilloscope whose data was analyzed through algorithms implemented in MatLab™. The setup was calibrated and tested using the NIST standard with various glycerol and water solutions, and had an error of within 2%. Additionally, further acoustic properties such as attenuation, acoustic impedance, and material properties such as the modulus of elasticity are calculable with the same device, as well as reflection and transmission coefficients with minor modification.

Introduction

Microfluidics refers to the study of the flow of fluids in the microscopic regime, typically in volumes on the micro or nanoliter scale[1]. Advantages of these micro-electromechanical systems (MEMS) are that they have potentially high throughput, can exploit microscale transport phenomena, and have an abundance of uses in both energy systems and biological applications[2]. The motivation behind studying the acoustic properties of materials in such devices is that many of them are coupled with piezoelectric crystals[3] for ultrasound generation in lab-on-a-chip (LOC) formats. Analogous to electrical impedance matching, certain performance characteristics of a device are vastly improved if the impedances of different material interfaces are matched[3]. In order to design for optimum performance, the speed of sound through the materials must be measured.

In fluids, sound waves travel only as longitudinal or compression waves, due to the lack of strong intermolecular bonds between orthogonal particles. The speed of sound through a fluid is a function of the compressibility and density of the fluid. This functional dependence is given by the Newton-Laplace equation

$$c = \sqrt{\frac{K_s}{\rho}}, \qquad (1)$$

where K_s represents the bulk modulus of elasticity of the material, and ρ represents the material density. In the scope of this project, the goal is to back calculate these parameters after measuring the speed of sound directly via the pulse and response of an acoustic wave.

In addition to fluid property measurements, the apparatus is designed to measure the speed of sound through polymers such as polydimethylsiloxane (PDMS). Sound propagation through polymers is significantly more complex than that through fluids, or even other solids. Since polymer chains are of various lengths in the bulk phase, there is a distribution of local densities within any given sample[5] leading to variations in the speed of sound for different regions of a single sample. Additionally, since PDMS is a crosslinked polymer, the degree of crosslinking that occurs during polymerization has a large effect on the speed of sound due to the change in local specific volume from this effect[6]. Most existing correlations are empirical because of the inability to fully characterize all aspects of a polymer without destruction of the sample.

Design and Fabrication
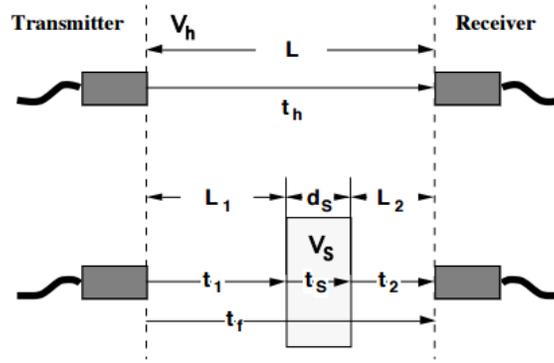
Figure 1: Schematic of Transducer Set Up

Figure 1 depicts a schematic of the setup of the apparatus, as viewed from the top down. Two ultrasonic transducers are connected to a pulser-receiver unit and oscilloscope. The sample is loaded between the two mounted transducers. A sine wave is sent through the transmitter and then received by the receiver. This is done in pure water, the host medium. Once the transducers are calibrated, the distance between the transducers measured, and the speed of sound in the host medium determined, only the thickness of the sample is needed to calculate the speed of sound in the sample material, as

$$Sample\ Velocity = V_s = \frac{V_h}{1 - \frac{V_h\left(t_h - t_f\right)}{d_s}},\qquad(2)$$

where t_h is the time for the wave to travel in the host medium without a sample, V_h is the sound velocity of the host medium, t_f is the time the wave takes to reach the receiver with the sample in place, and d_s is the thickness of the sample.

The design of the apparatus is meant to be modular. It consists of a Delrin® (acetal resin) block as a base board with various holes and grooves to lock in screws, which serve as a stand for the sample and two symmetric holders for the transducers (see Figures 2, 3 and 4). This arrangement allows the distance between the transducers to be varied as needed. Additionally, spacers are used to vary the height of the transducers. The entire set up is submerged in a tank of deionized (DI) water during testing, as the transducers are not meant for use in air. A center frequency of 3.5 MHz is used for testing, though other center frequencies can be used with the same Olympus 5077PR pulser receiver unit[7].
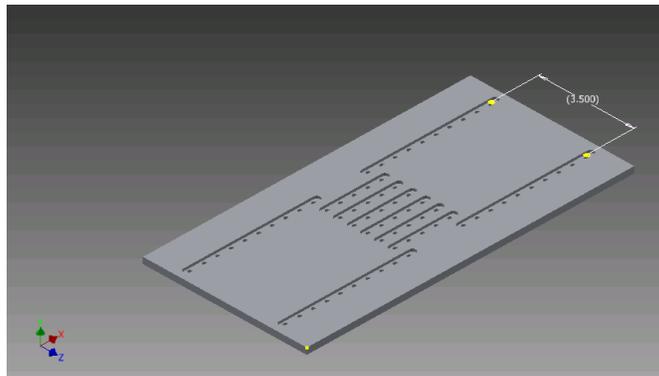
Figure 2: AutoCAD Image of Pegboard Design
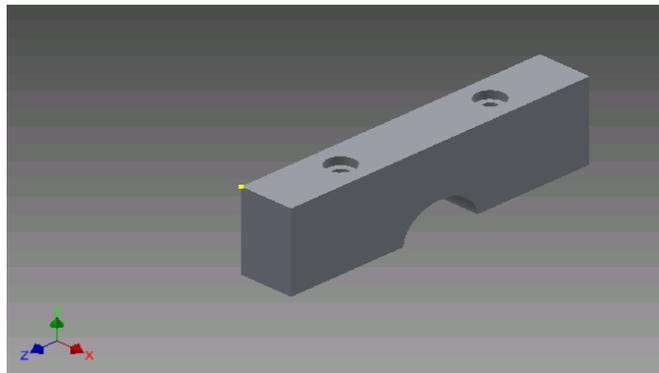
Figure 3: AutoCAD Design of Transducer Holder (top)

A photograph of the apparatus in use is shown in Figure 4. Delrin® parts were fabricated using a three axis mill and the CAM ribbon attachment in the program AutoDesk Inventor™. This allowed precise control of the position of various holes in the base board, which is essential for the alignment of the two transducers. If the transducers are not well aligned, the assumption that a plane longitudinal wave is being emitted/received by the transducers is not accurate, as a spherical wave is actually emitted due to the small aperture size. The plane wave assumption yields a 1% error for liquid solutions, but misalignment would reduce accuracy.
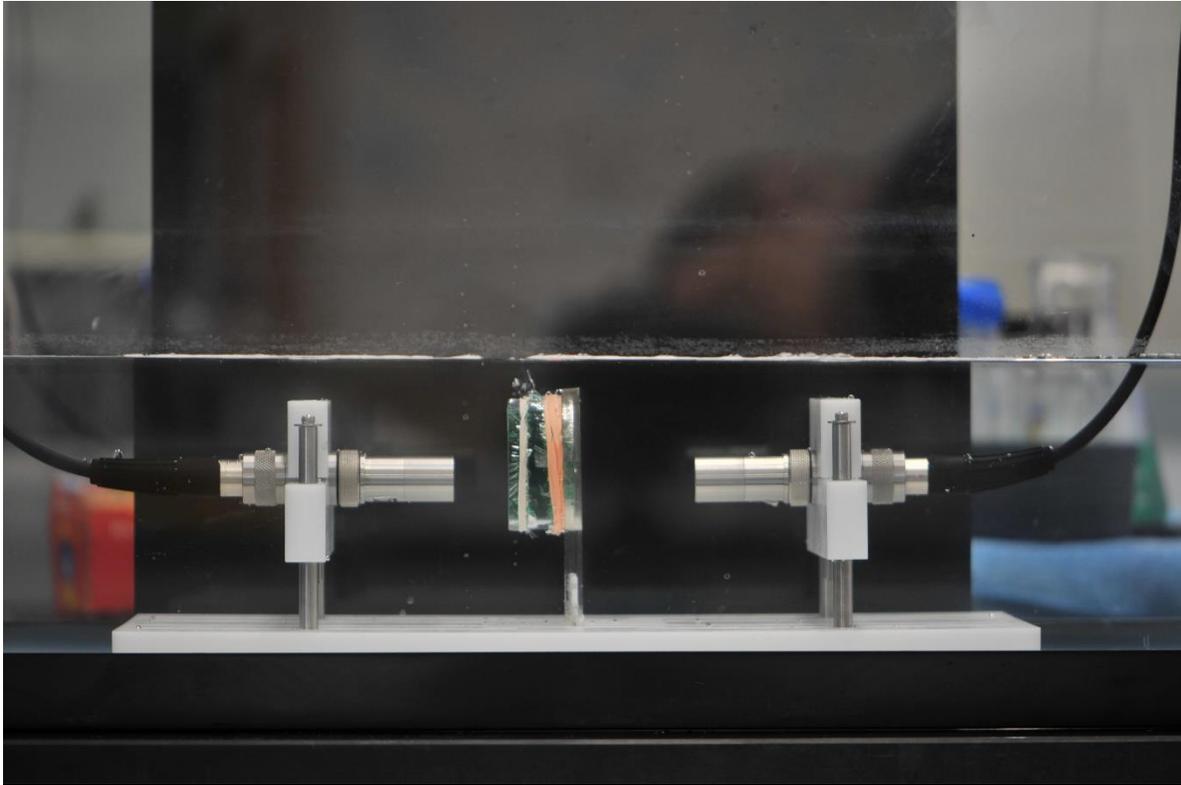
Figure 4: SOS Apparatus Measuring a Battery Precursor Solution

Programming

The waveforms are saved as voltage vs. time signals by a Tektronix TDS2001C oscilloscope. They are saved as comma separated value files, and a script in MatLab™ was written to convert and concatenate the data into an array with the first column being time, the second column being voltage of the square wave timing pulse output by the pulser-receiver (this has a 45 nanosecond lag time before the output of the actual pulse), and the third being the response data. The actual speed is measured from 45 nanoseconds after the spike in voltage is read from the synchronization pulse, and the initial sensing of a signal in the receiving transducer beyond the level of noise (~0.07mV). In order to calibrate the device, however, two other scripts are required. One inputs the temperature of the water and back calculates the distance of the two transducers to within the accuracy of National Institute of Standards and Technology speed of sound data for pure water[8] as a function of temperature (measured with a thermocouple). The second script uses this distance to find the speed of sound within the water as a reference, in order to get both the t_h and v_h values needed in Eqn. 2 and to serve as a check that the system is operating correctly, as this value should be the same as the true value of the speed of sound of water at the measured temperature. Appendix A contains copies of these scripts. The primary deficiency of the collection mechanism was the discrete sampling of a continuous response. The low coverage and limited values that can be calculated from the system resulted in artificially decreased

intrasample variability. While measured values may be close to actual values, exact precision cannot be calculated for a specific sample with a large amount of trials.

Results

Using control data from Slie et al.[8], speed of sound measurements for various water and glycerol mixtures were tested using the SOS apparatus as performance validation. Experimental samples were relatively thick (d_s=15.6 mm) to allow sufficient time within sample without attenuation of signal. The results shown in Figure 5 demonstrate the comparative accuracy to the prior experiment, concluding that the current SOS apparatus is sufficiently capable of replicating literature values.
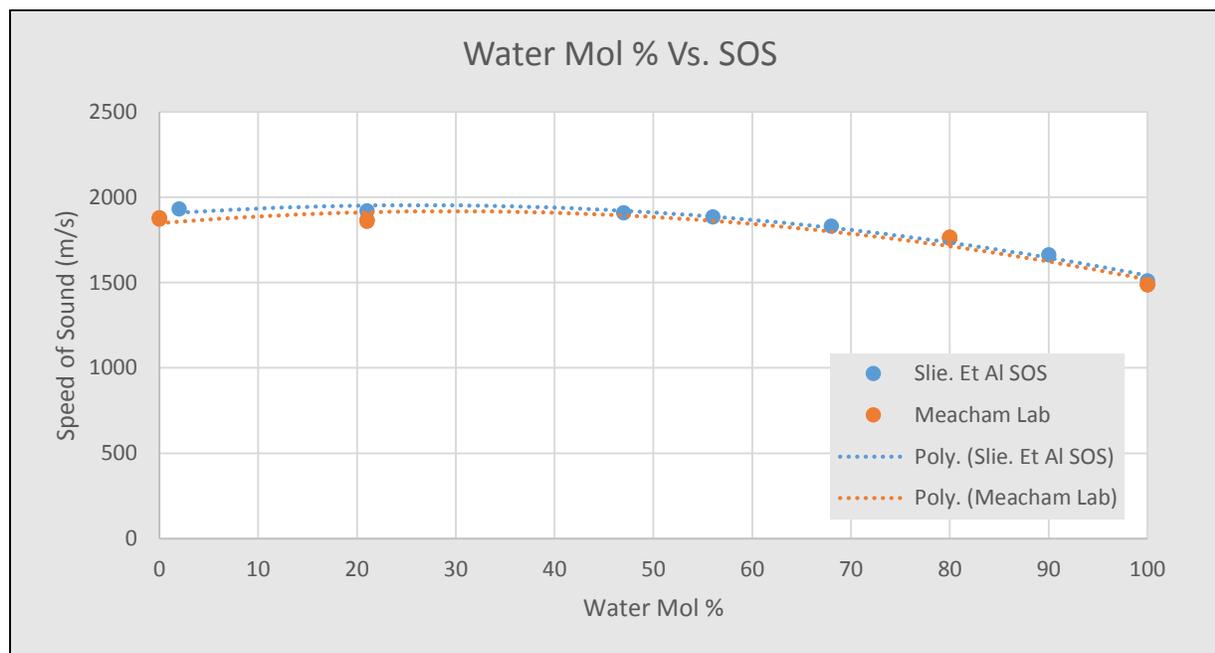
Figure 5: Glycerol-Water Solutions Speed of Sound Compared to Literature Data

The speeds of sound in a number of additional liquids of interest were also evaluated (d_s=15.6 mm). Figure 6 shows the variation in speed of sound for a battery precursor, solutions of $Li_{1.2}Mn_{.54}Ni_{.13}Co_{.13}O_2$ in DI water, tested under increasing solution concentrations. Similar to the relationship between speed of sound and concentration of NaCl in water, these solutions show a linear dependence of speed of sound on concentration. Measurement of the speed of sound in a cell culture medium (RPMI with 10 wt% Fetal Bovine Serum (FBS) and 1 wt% Penicillin) suggests that this culture medium does not behave much differently than pure water (see Table 1). Demonstration of the low variability within sample collection can be seen with five of the six values being equivalent.

After establishing the accuracy of our method by measuring the speed of sound in various liquids, the PDMS polymer, heat cured with various ratios of oligomer to curing agent octomethyltetrasiloxane, was tested. Results are reported in Figure 7.
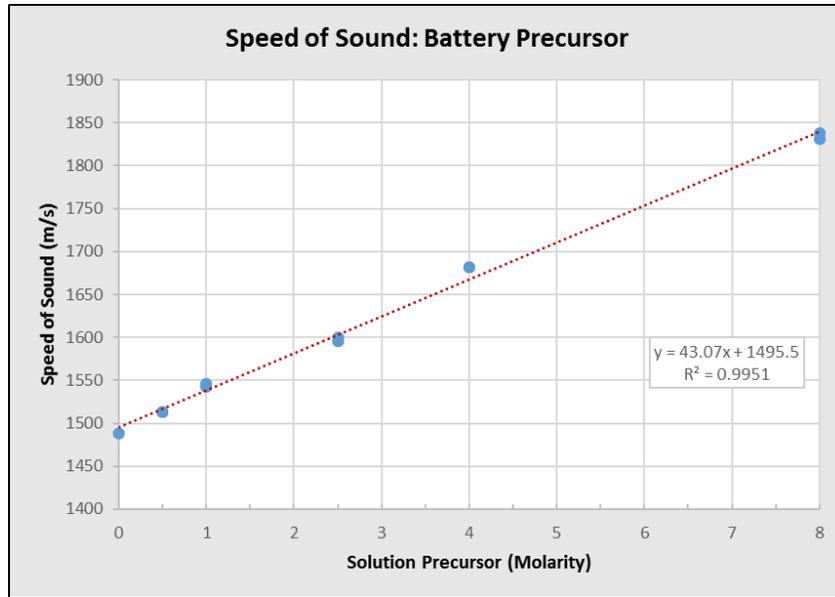
Figure 6: Battery Precursor SOS vs. Concentration

Table 1: Complete Cell Culture Growth Medium SOS Measurements

Trial	Solution	Speed [m/s]	Average [m/s]	STD [sqrt(m/s)]
1	RPMI	1499.068257	**1500.225117**	**2.586817254**
2	RPMI	1504.852556		
3	RPMI	1499.068257		
4	RPMI	1499.068257		
5	RPMI	1499.068257		
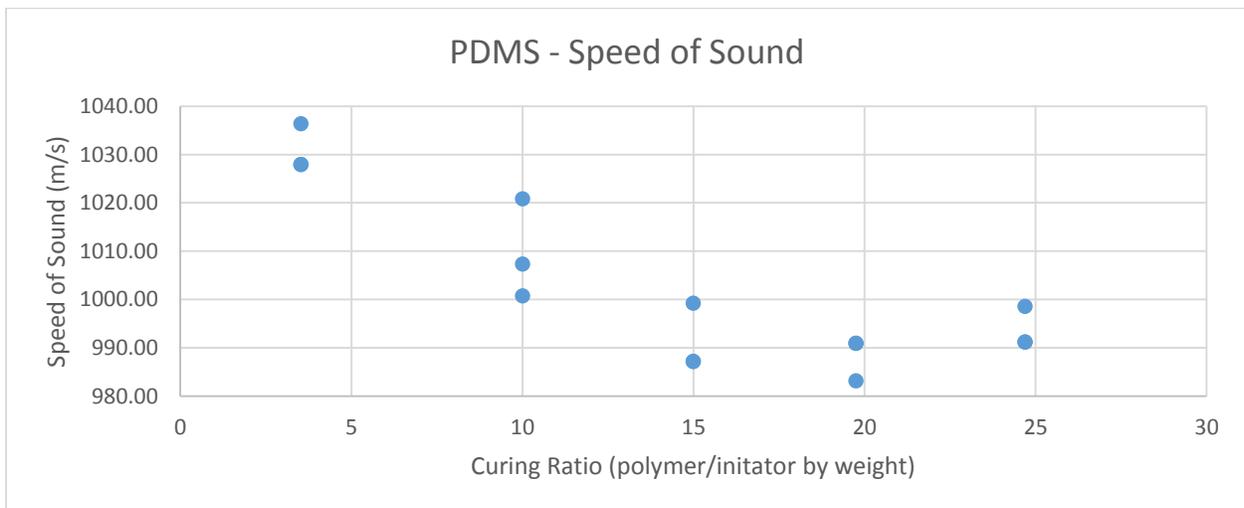
Figure 7: Speed of Sound in PDMS as a function of Weight Ratios

Discussion

As seen in Figure 5, the maximum error for high concentrations of glycerol was 1.5% from the literature value. As previously stated, this is roughly the accuracy that a plane wave analysis is expected to give. Thus, we can verify the accuracy of results of both the apparatus and the method of signal processing of the data.

In Figure 6, the data are highly consistent with what was expected. In the case of liquids, increasing solute concentration has a direct effect on fluid density, and in a suspension of a soluble liquid, this increase should be uniform in the fluid. Thus we should expect a linear increase in the speed of sound proportional to $(\rho)^{-1/2}$, as predicted by the Newton Laplace equation. The results of the cell culture medium, while consistent with literature, demonstrates the inability of the system to properly sample a continuous function. Because of this, only one value deviates from 1499 m/s. To properly determine the exact value for the speed of sound for any of these samples, a large sample size of tests is required.

Unfortunately, the data for the PDMS was extremely erratic, and no clear pattern could be observed. This could be attributed to the thin sample used (<5 mm) or other unexpected variables. In the audible range, PDMS is often used in cochlear implants[9] and other devices due to its similar acoustic impedance to water, one of the main motivations behind our interest in its use and the development of the apparatus. The PDMS in the experiments reported above was heat cured, using the standard procedure described by the manufacturer, Dow Corning[8]. The same samples were used for multiple trials and gave differing values of the speed of sound. This may be due to the settling of the polymer after days have elapsed from initial polymerization. Smaller polymer chains are slowly pushed away from the center of the material as the material settles to an equilibrium state of Van der Waals attraction between chains. The results differed for measurements taken after some elapsed time between trials, but the difference was fairly consistent. The larger issue is that the trend between different curing ratios was not as expected, which is likely due to the thickness of the cured samples. Solid samples must be thick enough that multiple wavelengths of the acoustic wave can traverse the thickness of the sample. Since the samples were under a centime thick, it is entirely possible that at the center frequency of the transducers, not enough wavelengths of the signal were within the loaded sample, introducing measurement error.

The ultimate goal of this project was to create a UV curable PDMS, and find the correct concentration of photoinitiator to add in order to have the most water-like acoustic properties in the ultrasonic range. Without observing consistent and predictable (and somewhat expected) trends in the behavior of heat-cured PDMS, the current testing method is unlikely to produce good results. For UV curable PDMS, the presence of unreacted curing agents as crystals in the polymer would disperse or refract sound waves in the material. Also, degree of crosslinking in heat-cured samples was not controlled; this process would be even more difficult to control without extreme precision in a UV light source. These issues can yield local variation of density across samples,

even while only changing cure time, or curing agent concentration. Finally, because UV curable PDMS involves a photoreaction, only 200-300 micron thick samples can be cured in a feasible amount of time. In order to reach a sufficient thickness for measuring the samples, nearly 50 hours of cure time are needed, with additional layers being added after each hour. Possible methods to address this are discussed below.

Conclusions and Further Work

Overall, the speed of sound measuring apparatus proved capable of measuring the acoustic properties of various materials. The device and method successfully measure the speed of sound to a high degree of accuracy in liquids, and to a lesser degree in solids. The speed of sound measurements against a control of glycerol and water solutions were very similar to other published results, thus ensuring that the device makes accurate measurements. Trends in the variation of properties for salt solutions were as expected, exhibiting replicable measurements that fit the expected linear trend as a function of solute concentration.

Unfortunately, the apparatus and method require modifications to accurately predict acoustic properties of polymers. Further work in signal processing includes increasing the resolution of the device in order to gain more data on samples with very similar speeds of sound. Additional algorithms for further signal processing are easily added to the current operating procedure, in order to take measurements of attenuation in samples, as well as reflection and transmission coefficients. Finally, in the scope of a larger project, the speed of sound of UV curable PDMS will be measured as a function of photoinitator concentration and time after curing in order to calculate the 'relaxation' time and its effect on the speed of sound.

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Appendix A – SOP for Speed of Sound Measurements

1. **Materials**
   a. **Equipment**
      i. Transducer holder w/ Fish Tank
      ii. Immersion Transducers
      iii. Ultrasonic Pulser/Reciever (PR)
      iv. Thermocouple
      v. BNC Cords (3)
      vi. Oscilloscope
      vii. Thermocouple
      viii. Computer Excel & Matlab
      ix. Liquid Sample Holder
      x. Cellophane (Saran™) Wrap
      xi. Rubber Bands
      xii. Sample Holder (Adjustable Height)
      xiii. Screw Driver
   b. **Chemicals**
      i. Deionized water (DI water)
      ii. Sample To be Measured
      iii. Glycerol (Optional)
   c. **Hazards**
      i. Skin
         1. Glycerol: May cause skin irritation. May be absorbed through skin
      ii. Eyes
         1. Glycerol: May cause eye irritation with stinging, redness, burning sensation, and tearing, but no eye injury.
      iii. Ingestion
         1. Glycerol: Low hazard. Low toxicity except with very large doses. When large doses are ingested, it can cause gastrointestinal tract irritation with thirst (dehydration), nausea or vomiting diarrhea. It may also affect behavior/central nervous system/nervous system (central nervous system depression, general anesthetic, headache, dizziness, confusion, insomnia, toxic psychosis, muscle weakness, paralysisconvulsions), urinary system/kidneys (renal failure, p. 5 hemoglobinuria), cardiovascular system (cardiac arrhythmias), liver. It may also cause elevated blood sugar.
      iv. Inhalation
         1. Glycerol: Due to low vapor pressure, inhalation of the vapors at room temperature is unlikely. Inhalation of mist may cause respiratory tract irritation.
      v. Hazard Classification (MSDS)
         1. Fire
            a. Glycerol: 1
         2. Health
            a. Glycerol: 1
         3. Reactivity
            a. Glycerol: 0
         4. Specific Hazards
            a. Glycerol: NA

2. **Precautions and Hazard Mitigations**

a. **Equipment**
  i. Read all SOP for equipment of use prior to procedure listed in Section 3
b. **Chemicals**
  i. Review all MSDS for new chemicals listed in procedure (Section 3) prior to beginning experimentation.
  ii. Review Chemical Hygiene Plan prior to operation for adequate preparation
3. **Procedure**
  a. **Verify that all equipment is present and cleaned**
  b. **Apparatus Set Up**
    i. Connect 1 Transducer to 'T/R' port on the PR using a BNC cable.
    ii. Switch the Transmission mode to T/R.
    iii. Connect the Other Transducer to the 'R' port of the PR using a BNC Cable.
    iv. Connect the RF SIGNAL OUT port on the back of the PR to the CH1 port of the Oscilloscope
    v. Connect the +SYNC OUT port on the back of the PR to the CH2 port of the Oscilloscope
    vi. Place the Transducers into the Transducer Holder. Make sure the Red Caps of the transducers are removed.
    vii. Tighten the Top Screws of the Transducer Holder so the transducers are secure
    viii. Plug in All devices, but keep the PR in the off position
    ix. Turn on the Oscilloscope, Set the Horizontal Sensitivity of the scope to .5 uS/div and the Vertical Sensitivity to 0.2 V/div.
    x. Put in a Flash drive of total memory under 2GB into the USB port on the front of the Oscilloscope. The machine will configure after a few minutes
    xi. Fill the Tank with DI Water ≈ ¾ Full.
    xii. Measure the Distance between the Transducers with the micrometer, or calipers. Record this value.
    xiii. Place the Transducer Holder into the Water.
  c. **Acoustic Calibration**
    i. Use the Thermocouple to measure the temperature of the water and record this value.
    ii. Look up the speed of sound of water at this temperature on the NIST Chemistry Web Book.
    iii. Turn on the PR.
    iv. Center the Waveforms on the Oscilloscope. You should be able to see the entire square wave as well as the response (the response is on the yellow CH1, the square wave is on the blue CH2.)
    v. On the oscilloscope, navigate to 'Save Waveform.' Save both the CH1 Waveform and CH2 Waveform.
    vi. Record the names of these files, exactly.
  d. **Sample Measurement**
    i. Turn of the PR.
    ii. Remove the Transducer Holder from the DI Water.
    iii. Load the Sample to be measured in between the path of the transducers.
    iv. Return the holder to the DI Water.
    v. Turn on the PR.
    vi. On the oscilloscope, navigate to 'Save Waveform.' Save both the CH1 Waveform and CH2 Waveform.
    vii. Record the names of these files, exactly. The Oscilloscope should naturally increase the file number by 1 on each save. Remember to record which channel was associated with the response and the square timing pulse.

     viii.   Remove the Flash Drive from the oscilloscope, and put it into a computer with Excel and Matlab.

     ix.   Turn off the PR and remove the transducer holder from the tank.

     x.   Wipe the Transducer holder down, and place the Red Caps back onto the transducers.

  e.   **Data Analysis**

     i.   Rename the saved files as .xls files (they are saved as .csv files by the oscilloscope).

     ii.   In the data_grabber.m script, rename the files as they appear. Make sure to follow the comments for where to put the response waveform as opposed to the square pulse.

     iii.   The script will automatically save relevant parameters. In the speed_calib_trans file, input the distance between the transducers, and run the script. This will calculate the speed of sound in pure water. Check to make sure it is close to the literature value.

     iv.   Input the speed of sound to the speed_calib_inverse.m function. This shold give a value very close to the distance between the trasnducers.

     v.   Now rerun the data_grabber.m script eith the sample waveform and data.

     vi.   Input the sample thickness into speed_sample_trans.m. Matlab will have the parameters from calibration already saved, so the output of this function is the speed of sound. Record the calculated value in the testing matrix.

     vii.   Return the Flash Drive to the oscilloscope when analysis is complete.

4. Storage

a. Apparatus

     i.   Turn off Pulser/Reciever Unit

     ii.   Remove Transducer Holder From Water

     iii.   Dry With Paper Towels

     iv.   Remove Sample and Store with Label

5. Waste

  a. Pour excess solution into the correctly labelled chemical waste storage container

  b. Make sure label has correct chemicals listed

     i.   Non-compatible chemicals are strong oxidizing agents and strong acids

  c. All chemically contained gloves from procedure should be disposed of in chemical waste container

6. Accident Procedure

a. Skin

     i.   Flush skin with water for at least 15 minutes

     ii.   Clean with soap

     iii.   Contact supervisor

b. Eyes

     i.   Flush eyes with copious amounts of water from the eye wash station for at least 15 minutes

     ii.   Contact supervisor

c. Inhalation

     i.   If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen.

     ii.   Contact supervisor

d. Ingestion

     i.   If swallowed, wash out mouth with water provided person is conscious. Call a physician.

     ii.   Contact supervisor

7. **PPE**
   a. **Body Protection**
      i. Proper clothing listed in the Lab Blue Book should be worn when dealing with all chemicals in this procedure
      ii. No additional clothing requirements are needed
   b. **Gloves**
      i. Chemical resistant gloves should be worn at all times during the process
   c. **Eye Protection**
      i. Safety goggles are required when dealing possible eye irritants

Appendix B – MatLab code for Signal Processing

Data_grabber.m – a script reads data from the oscilloscope and converts it into arrays for processing.

```matlab
%Data-grabber creates arrays of data from the oscilloscope
%note that the data must be moved into excel and made into a .xls format
%file

tic
%~~~~~Data for Calibration Run (grabs all relevant data)~~~~~~~~~~~~
global data1 time_length

data_ch1=xlsread ('5_1_PDMS_Resp_1.xls', '','','basic');  %ch1 response data
data_ch2=xlsread ('5_1_PDMS_Pul_1.xls', '','','basic'); %sync out data
time_length=size(data_ch1,1);
data1=zeros(time_length,3);

%extracts data from xls/cvs for ch1 (actual response)
for i=1:time_length
data1(i,1)=data_ch1(i,3);  %time    data
data1(i,2)=data_ch1(i,4);  %voltage data
end

%adds in sync data (1st sync pulse leads main bang by ~45 nanoseconds
for i=1:time_length
data1(i,3)=data_ch2(i,4);
end

clear i %since this is a script we don't want to save out counter
%all data is now stored in data1

%hold on
%plot(data1(:,1),data1(:,2))  %,data1(:,1),data1(:,3)) %recreates wave form
%toc %time to run
%hold off
```

Speed_calib_trans.m - a function that calculates the speed of sound in the host medium used in the apparatus, typically deionized water

```matlab
function [th vh]= speed_calib_trans(Dt)
%Speed_calib_trans calibrates the acoustic measurement system
%    the function requires an input of the distance between the two
%    transducers and calculates the speed, after the data_grabber script is
%    run

global data1 time_length data_ch1 data_ch2 th vh

%finding index of end of pulse from sync (i.e. t_start)

pulse_raw=data1(:,3);
pulse=(find(pulse_raw>7))
t_start=pulse(1);
time_init=data1(t_start,1); %end of pulse (~45 nSec before end of main bang)

%finding index of wavefront return

resp_raw=data1(:,2);    %response of second transducer
resp=find(abs(resp_raw)>0.9)   %noise killer
t_end=data1(resp(1),1) %transmitted signal reaches transducer

th=t_end-(time_init+45*10^-9) %wave travel time in seconds
vh=Dt/th   %speed of sound in host medium


assignin('base', 'th', th)
assignin('base', 'vh', vh)

end
```

Speed_calib_inverse.m - a function that back calculates the distance the transducers are spaced based off the NIST reference value for the speed of sound of water as a function of temperature.

```matlab
function [trans_distance ]=speed_calib_inverse(sos)
%Speed_sample_trans measures the speed of sound of the sample loaded
    %input needed is the sample thickness, and the calib function needs to
    %be ran first
%sos needs to be looked up
global data1 time_length data_ch1 data_ch2 th vh trans_distance

%finding index of end of pulse from sync (i.e. t_start)

pulse_raw_sample=data1(:,3);
pulse=find(pulse_raw_sample>0.5);
t_start=pulse(1);
time_init=data1(t_start,1);%end of pulse (~45 nSec before end of main bang)

%finding index of wavefront return to sensor

resp_raw=data1(:,2);
resp=find(abs(resp_raw)>0.07);
t_end=data1(resp(1),1); %transmitted signal reaches transducer

t_trans=t_end-(time_init+45*10^-9); %
trans_distance=sos*t_trans;

end
```

speed_sample_trans.m – a function that processes the sample data and calculates the speed of sound in the material being tested

```matlab
function [v_sample , t_sample, resp]= speed_sample_trans(Ds)
%Speed_sample_trans measures the speed of sound of the sample loaded
    %input needed is the sample thickness, and the calib function needs to
    %be ran first

global data1 time_length data_ch1 data_ch2 th vh

%finding index of end of pulse from sync (i.e. t_start)
kk=Ds;
pulse_raw_sample=data1(:,3);
pulse=find(pulse_raw_sample>2)
t_start=pulse(1);
time_init=data1(t_start,1);%end of pulse (~45 nSec before end of main bang)

%finding index of wavefront return

resp_raw=data1(:,2);
resp=find(abs(resp_raw)>.8)
t_end=data1(resp(1),1); %transmitted signal reaches transducer

t_sample=t_end+30*10^-9-(time_init); %wave travel time
%t_sample=t_end-(time_init+45*10^-9); %wave travel time
v_sample= vh./(1-vh*(th-t_sample)./kk) %speed of sound through sample

assignin('base', 't_sample', t_sample)
assignin('base', 'v_sample', v_sample)

end
```