Minute Volume Viscometer

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Abstract

In the field of pharmaceutical research, injectable treatment therapy is currently one of the most highly examined methods of drug delivery. The continuation of this research is highly dependent on knowledge of fluid properties, specifically loss and storage modulus. Most drugs in the developmental stages of this method are extremely expensive and limited in quantity. Currently, devices that measure fluid properties require samples sizes larger than 150 µL. A device that would minimize the sample volume would be quite valuable to pharmaceutical companies. We have designed a device that will be able to quickly and accurately measure the loss and storage modulus of sample volumes of less than 20 µL.

Three designs were considered throughout the planning of this project. One utilized polarized lenses with an alternating current to calculate the relative “brightness” of light through a sample liquid. A second method employed tuning forks vibrations as they propagate through a sample liquid to calculate waveform impedance. We have concluded that the optimal design for this project is one which calculates impedance of shear waves based on a loaded piezoelectric crystal.
Introduction

In the pharmaceutical industry, it is important to know the fluid properties of the drug being developed. Most up and coming drugs are extremely expensive and limited in quantity. A device that can measure fluid properties, such as loss and storage modulus, with minimal sample sizes would save the manufacturer money. There are many devices on the market that can be used to obtain rheometric measurements, but none of them operate on a volume of less than 150 µL. The small volume requirement is the greatest design obstacle that eliminates many of the methods that we considered. Successful rheometric measurement has been accomplished previously using piezoelectric crystals, but this design is limited to the frequency of the crystal\(^1\). Therefore, we considered alternatives to this method that applied shear stresses similar to that of piezoelectric crystals, such as tuning forks. We also considered making alterations to the current piezoelectric setup that would allow a frequency range. Finally, we explored a more novel approach that involves polarized light.

Background Information

To be able to expand in the fields of science and technology scientists need to conduct research to explore new ways of improving technology. This means that they need a better way to conduct their research, which implies more advanced equipment needed to satisfy this criterion. One of the many areas where accurate information is required is that of rheological properties of fluid. Many devices are currently on the market where these measurements can be conducted but they also come with many down sides. A scientist conducting research needs to be able to measure the physical

properties of a solution with accurate precision. Designing a device that would measure a fluid’s rheometric properties with accuracy and at minute amounts will open a new window for research.

The ability to accurately measure properties of small quantities of fluid is essential to many research scientists. This will not only give better reading measurements but will also cut cost on many expensive research materials. The viscometer device will allow many companies to increase their research capacity as well as extending into many other research fields. A well designed rheometer will contain the following properties: light weight; high durability; low cost; accurate measuring; small mobile size; and long life time. Many local and global industries require a device that would measure rheometric properties, indicating that this device has a global marketing potential. Not only can a rheometer be used in the medical field but also in many other industrial areas.

**Current Market for Small Volume Viscometers**

The majority of the market consists of two main types of viscometers, those that measure the rate of flow (tube) and those that apply a shear stress to the liquid (rotational). Tube viscometers measure the rate of flow of the liquid through a tube with a pressure difference. There are two types of rotational viscometers. The rotating cup-and-bob viscometer shears the liquid between two concentric cylinders rotating about the same axis. The cone-and-plate viscometer consists of a cone with a plate that runs through its apex.

Table 1 lists a few of the most popular models on the market. Those with small sample volumes were listed since that is one of the key specifications of the design project. The Automated Falling Sphere Microviscometer from Anton Paar (Ashland VA) can be used with sample volumes as small as 150 μL, however, it
comes at a high price of $12,600 not including necessary software and accessories. The other devices function with larger sample volumes with their minimum necessary volumes ranging from 0.5 mL to 2 mL. A viscometer that could use less than 20 µL would be alone in the field.

<table>
<thead>
<tr>
<th>Product Type*</th>
<th>Company*</th>
<th>Product Name*</th>
<th>Sample Size*</th>
<th>Price**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capillary flow viscometer</td>
<td>Cannon Instrument (State College, PA)</td>
<td>Cannon Manning Semi-Micro Viscometer (Extra-Low Charge)</td>
<td>0.5 mL</td>
<td>$105.00</td>
</tr>
<tr>
<td>Cone and Plate viscometer</td>
<td>Brookfield Engineering Laboratories (Middleboro, MA)</td>
<td>Wells-Brookfield Cone/Plate</td>
<td>0.5 mL</td>
<td>$1645-2895</td>
</tr>
<tr>
<td>Rotating Cup and Bob</td>
<td>Brookfield Engineering Laboratories (Middleboro, MA)</td>
<td>Brookfield Viscometer</td>
<td>2mL</td>
<td>$3295-3895</td>
</tr>
<tr>
<td>Rotating Cup and Bob</td>
<td>Anton Paar (Ashland, VA)</td>
<td>Automated Falling Sphere Microviscometer</td>
<td>150µL</td>
<td>$12,600.00</td>
</tr>
</tbody>
</table>

Table 1: Current Rheometric devices on the market (* taken from \(^1\); ** taken from distributors’ websites and catalogs)

In preliminary research, the loss and storage modulus has been obtained with as little as 8 to 10 µL of sample \(^1\). By using the shear stresses produced by piezoelectric crystals, Saluja and Kalonia were able to accurately determine the loss and storage modulus of sample solutions of sucrose, urea, PEG-400, glucose, and ethylene glycol at 25 °C ± 0.5 °C. The measurement itself took only 2 to 3 minutes and the setup did not create any unwanted initial stress on the crystal. They used two crystals of different fundamental frequency (5- and 10-MHz) in the design. The main
drawback of this setup is that it is limited to the frequencies of the crystals; each crystal can only provide one frequency. It is desirable to be able to measure liquid response to a range of frequencies. Our design will be able to make measurements at varying frequencies.

**Patent opportunities**

As is the case with any original invention, certain opportunities exist for the copywriting of the project, namely, a patent. A United States patent is a grant of the property rights to one's original idea. The patent gives the “right to exclude others from making, using, offering for sale or selling”\(^2\) the invention in the United States and excludes others from importing the invention into US. There exist three types of patents; the utility patent, the design patent, and the plant patent. The utility patent would apply to this particular project, and may be granted to anyone who invents or discovers a new and useful process, machine, article of manufacture, composition or matters, or an added useful improvement to a pre-existing design.

Research of patent approved designs in a field similar to this project was conducted to estimate the patent opportunities for this viscometer. Several rheometer designs for a ranging volume of liquid have been approved for patent in recent past. One such patent in the field is for the method for determining a characteristic viscosity-shear rate relationship for a fluid, patent number 6,796,168, submitted September 28, 2004 by Larry Goldstein and William Hogenauer. This method finds “the viscosity of a fluid flowing through a system at any point in the system whereby the method involves determining a characteristic relationship for the fluid between viscosity and shear rate; obtaining a shear rate of the fluid as it moves through at least one position in the system; and determining the viscosity of fluid at the at least one

position by applying the shear rate to the characteristic relationship” (Goldstein, Hogenauer, 1\textsuperscript{2}). This particular patent is in fact following up to a previous patent entitled “Single riser/ single capillary viscometer using mass detection or column height detection”, which is itself a follow up to the patent “Mass detection capillary viscometer”\textsuperscript{2}. This illustrates the patent requirement that the design may be an improvement to a pre-existing device, which is likely what the viscometer in our project will be. While the viscometer may in fact be an improvement, or alteration, on a previously existing idea, patent searches proved that no patented machine exists which would function in the specific way, under the specific conditions (i.e. small volume, multiple frequencies) as our proposed design. Thus, this would be considered an original design and would be appropriate for patenting.
Discussion

Three different setups were considered for this design: Tuning Fork Method, Polarized Light Method, and Piezoelectric Crystal Method. All three will be discussed in detail in the following sections.

Tuning Fork Method

Tuning forks may be utilized to create longitudinal waveforms in a liquid medium. Tuning forks employ vibrations to produce sound waves. The fork itself is a relatively simple looking device, made up of a handle attached to two tines, or prongs. When the tines of the fork are hit with a hammer, or some automated device, they begin to vibrate, thus disturbing the surrounding air molecules. The tine vibrates outward from its original position, and compresses the air around it, creating a high pressure area known as a compression. The tine then vibrates inward from the extended position, allowing the high pressure area to expand, producing an area of low pressure, known as rarefaction. This association of the tines inward and outward generates an alternating pattern of high and low pressure regions, which transport the vibration through the surroundings.

In order to measure the rheometric properties of a liquid, longitudinal waves must be allowed to flow through the liquid so impedance shifts can be calculated. While sound waves can exist as transverse or longitudinal waves in solids, they are manifested only as longitudinal waves in fluids. These waves travel outward in a parallel/anti-parallel plane to the direction of the energy transport, vibrating the air molecules in this plane. The molecules move away from their original position and then vibrate back into that original position, creating no true net displacement of the air. In this way, sound waves are able to propel energy from one location to another without transporting any matter.
This design also offers a thermometer reading along with the rheometric measurements, such that a correlation between temperature and rheometric properties of that particular fluid can be analyzed. This allows the user to estimate, based on this temperature versus viscosity curve, the properties of the sample *in vivo*, in storage, in transport and in manufacture.

The setup of this design will be executed such that the vibrations of the tuning fork are initially measured in air as a constant frequency. Because tuning forks are only capable of resonating at a set frequency, the amplitude of the wave is measured here. This functions as a control of the device. One of the tines would then be inserted, or “dipped”, into the sample container, and the other tine of the tuning fork would be placed within the loaded container. The mechanical hammer would then hit the tuning fork, initiating oscillations. A shift in the impedance will inevitably occur, due to the propagation shift of the longitudinal waves through the viscous sample. An impedance analyzer will measure this shift, and then the properties of the fluid, specifically loss and storage modulus, may be computed through correlated calculations.

The set up will also account for a variety of test frequencies. In order to generate this range of frequencies, many tuning forks must be used. To conserve money and resources, the same sample will be “dipped” by several different forks. The volume of the sample is not a variable of the correlated equations, thus small differences in the sample size as a result of several dippings will not affect the results.
Calculations

Our client wants the final fluid properties as well as the raw data to be displayed on a personal computer. This will allow our client the ability to have more thorough documentation of the measurements and more freedom in evaluating the sample’s properties. An AD5933 Analog Device impedance analyzer will initially analyze the signal and convert it from analog to digital. The signal is generated using a sensor that will measure the longitudinal wave’s amplitude. This signal will then be compared with that from the tuning fork and analyzed using the AD5933 chip. The signal will carry the impedance shift information which will be used to calculate the rheometric properties. The data will be sent through the USB communicator to the computer using a MAX3346E (MAXIM Dallas Semiconductor) chip. The device will be connected to the personal computer through USB connections. Overall circuit performance will be tested by selecting test points with predictable outputs throughout the circuit that can be measured with an oscilloscope. The test points will be determined while the device is being built but most likely will be at the points where measurements need to be defined for device accuracy.
The client also prefers a wall outlet power source. A commercially available power supply will be used to power the device. Functionality of the power supply will be tested using a digital multi-meter.

After the impedance values are sent to the computer, a program such as one created with Labview will be used to create the user interface and display the raw and analyzed data. This program not yet designed will calculate the loss and storage modulus from the signal received through the USB cable. This will include the equations used to finding loss and storage modulus from the impedance shift generated. Another Labview program will be made to stimulate the data received which will be used to test the overall performance of the user interface.

Our client is interested in measuring two fluid properties; loss modulus $G''$ and storage modulus $G'$. $R_2$ is the change of the real part (resistance) of the impedance when the tuning fork is loaded against the sample. $X_2$ is the change in the imaginary part of the impedance for the loaded fork ($X_2=\omega L_2$). $A$ is a known constant for each fork based on resonance frequency. $\rho_{\text{Liq}}$ is the density of the sample. Using tuning forks will allow measurement of both $G'$ and $G''$.

\begin{align}
G' &= \frac{R_2^2 - X_2^2}{A^2 \rho_{\text{Liq}}} \quad (\text{Eq.} \, 1') \\
G'' &= \frac{2R_2X_2}{A^2 \rho_{\text{Liq}}} \quad (\text{Eq.} \, 2')
\end{align}

Determining both $G''$ and $G'$ is important when studying Non Newtonian fluids, fluids that store some of the energy that is applied to them. Energy storage occurs in fluids with large molecules at high concentrations where molecular movement is inhibited by neighboring molecules. The intermolecular interaction that causes energy storage is an important fluid property that must not be confused with viscosity. However, in many viscometers, the storage modulus is ignored and causes the measured viscosity
to deviate from actual rheometric properties. This is not a problem with Newtonian fluids because with Newtonian fluids \(X_2 = R_2\) which causes \(G'\) to go to zero. The following equation is normally used when calculating Newtonian viscosity.

\[
\eta_{Liq} = \frac{2R_2X_2}{A^2\rho_{Liq}} \quad (Eq. \, 3^1)
\]

Each tuning fork must be measured in order to determine the constant \(A\) before any fluid properties can be obtained. A fluid with known density and loss and storage modulus will be used to determine the constant \(A\), which is equal to:

\[
A = \frac{N\pi}{4K^2\omega_s C_0 Z_q} \quad (Eq.\, 4^1)
\]

Where \(N\) is the overtone number (primary overtone will be utilized), \(K\) is the coupling constant, \(\omega_s\) is the series resonant frequency, \(C_0\) is the static plus stray constant, and \(Z_q\) is the mechanical impedance of the fork. \(A\) is necessary because it allows us to calculate the mechanical impedance from the measured electrical impedance (where \(Z_{Elec} = A*Z_{Mech}\)). Therefore, the electrical values (\(X_2\) and \(R_2\)) can be used in determining \(G'\) and \(G''\) when \(A\) is included in the equations.

Once the device is assembled and all components are tested, fluids with known \(G'\) and \(G''\) values will be used to calibrate the device (determine the constant \(A\)) and test for overall accuracy and functionality.

**Tuning Forks**

Fisher Scientific sells an adjustable tuning fork (frequency range: 128-240 Hz) that will be perfect for our design. Because the fork is adjustable, it will eliminate the
need to have multiple forks which would have required the user to move the sample from one fork to the next. Having one fork that can vary over a range of frequencies simplifies the setup. The fork is adjusted by simply turning two dials on each side of the fork. This moves weights that are attached to the fork which in turn changes the length of the fork that can vibrate. Since the length of the fork determines the frequency, the frequency can be easily adjusted.

**Container**

The container that holds the sample must be an appropriate size and have no chemical interactions with the sample. Fisher Scientific sells dishes that are a smaller version of Petri dishes commonly found in laboratories. They measure 50 X 11 mm (diameter X height), which is large enough for easy handling and small enough for the sample size we are designing for. They come with a cover for easy storage and are inexpensive. They are also disposable, eliminating many contamination possibilities. These dishes are used for many different applications and, since they are made of polystyrene, are known to be un-reactive with the types of samples our device will be measuring.

**Mechanical Hammer (Solenoid)**

The mechanism used here to strike the fork is similar to that of a door bell. A simple circuit is made that contains an electromagnet, spring, and piston. The piston consists of an iron core with allows it to be attracted and expelled by the magnetic field. A push button is used to close the circuit; this allows the magnet to produce a magnetic field which then draws the piston into it core. As the piston is drawn into the core, its metal head will hit the tuning fork with will cause it to vibrate. After pushing the button the circuit will open and the magnetic field will be removed from the coil, and the spring will pull the piston back into position. The cycle can be repeated as
many times as the fork needs to be vibrated. Figure 2 shows the setup using the piston technique to mechanically hit the tuning fork.

![Diagram of mechanical hammer setup](image)

Figure 2

The voltage source of the mechanical hammer must not exceed 6 volts since high voltage sources are not be used for device with such a small scale. The power supply must be a DC supply to prevent the polarity of the magnet from changing, instead remain constant and attract the piston in one direction. The circuit is very easily made; it can be implemented on a printed circuit board which will be used in the design. The circuit also includes a regular push button that closes the circuit which then activates the electromagnet. The idea of the electromagnet can be implemented by coiling a copper wire; this permits a magnetic field which draws the piston into the coiled area thus hitting the tuning fork. After the user has pushed the button the circuit is then open which terminates the density flux due to the magnetic field. A spring is used to pull the piston from the coiled region back into its original position; most commonly used are brass springs.
**Polarized Light Method**

**The Basics of the Rotation of Polarized Light**

The molecules that will be studied with this device will have an overall charge of zero but will have localized polarities that can be used to orient the molecule. Suppose a charge was applied to the solution, the molecules would rotate and align with the voltage difference according to their poles. This movement would not be instantaneous, however, because of the intermolecular interactions between molecules. From this time delay it is possible to determine to what degree the molecules are interacting with each other. To measure the movement of the molecules we are expecting to use the optical properties of the molecules.

The molecules that will be studied with this device will be chiral (“of or relating to the structural characteristic of a molecule that makes it impossible to superimpose it on its mirror image”\(^3\)). Chiral molecules have the ability to rotate plane polarized light. A solution of chiral molecules (excluding a 50/50 solution of enantiomers) will rotate polarized light. This occurs because only the opposite enantiomer (“either one of a pair of compounds (crystals or molecules) that are mirror images of each other but are not identical”\(^3\)) can fully negate its mirror image. Each molecule has an optimal orientation to intersecting light, which results in the maximum rotation of light. In a single enantiomer solution, the molecules are randomly oriented to the intersecting light. Therefore, the light will not always hit the molecules at the optimal angle, instead it may rotate light at a fraction of the maximum or in the opposite direction of the optimal rotation. However, only the mirror image (enantiomer) can completely negate the optimal rotation. Therefore, in a single enantiomer solution, the net rotation of light will not be zero. Likewise, in a

\(^3\) www.dictionary.com
50/50 mix of enantiomers the net rotation will be zero. It is expected the solutions this device will be measuring will not have a 50/50 mix of enantiomers.

In our setup the molecules will have instances when they will not be randomly oriented. Each individual molecule will rotate the light at the same magnitude and direction. This cumulative rotation from the oriented solution will be measurably different from the net rotation of the randomly oriented solution. It is possible to measure the time it takes a randomly oriented solution to be become aligned and vice versa.

**Design Setup**

![Figure 3 – Polarized Light Design Setup](image)

Figure 3 illustrates the general setup for this design approach. Light is emitted by the light source and then is polarized into a known plane by the first polarizing device. The light then enters the sample and is rotated. The second polarizing device is oriented so that it maximizes light passage when the sample is oriented (while the maximum charge is applied). If the sample is not at maximum orientation (i.e. randomly oriented), the light passing through the second lens will be a fraction of the light that would pass through at maximum orientation. The brightness of the light that passes through the second polarizing device will be measured. The maximum and minimum brightness for the sample will be recorded in the analysis software for
calibration. Both the applied voltage and the change in brightness will be stored and plotted for analysis.

The design can be broken up into these major components: 1) Light source, 2) Polarizing devices, 3) Sample container 4) Photodiode and accompanying circuit 5) Calibration setup 6) Computational analysis.

**Light Source**

In most cases where polarized light is used to measure molecular properties, a monochromatic light source ranging between 600-700 nm in wavelength is used. In theory, any light source within in the visible light spectrum would be adequate for these kinds of measurements. In order to accommodate samples with different properties (i.e. some samples may absorb a specific light wave), multiple monochromatic light sources will be available in this design. Power Technology, Inc. (Alexander, AR) offers laser diodes that meet our requirements for our design (high power: 10mW, low dispersal: 8X8, and desired wavelengths: 635, 650, 670, 690). The light emitted by these diodes is polarized, eliminating the need for a pre-sample polarizing device. This will be beneficial to our setup because for each polarizer that the light passes through, over 50% of the light is lost. This will allow for a more accurate final measurement.

**Polarizing Devices**

Since the light source emits polarized light, the design will need only one polarizer (located before the brightness sensor). Plastic dichroic polarizing sheets (often found in science classrooms or sunglasses) will be the best type of polarizer for this design. Melles Griot (Carlsbad, CA) sells sheets that are fixated in-between two glass pieces, which is then mounted on an aluminum ring. The polarizing device fits
within our wavelength range (600-700 nm) and has desirable physical dimensions for our design (12.5 mm). Using a mounted polarizer will be more durable and easier to implement than the polarizing sheets that are typically available.

Sample Container

The design setup requires a container for the sample liquid that is optically transparent, holds less than 20 μL, and has electrodes. Hellma (Mullheim, Germany) sells ultra-micro cuvettes that are optically transparent and hold small volumes (10 or 20 μL). To be able to apply a voltage to the liquid, gold tipped electrodes can be placed inside the chamber (care must be taken in this extremely small volume so that the electrodes do not come into contact with one another). The cuvette has a light path of 10mm, which is standard in most light/liquid experiments. Light path length is an important property that cannot be sacrificed in these types of measurements because, in order to obtain an accurate and significant reading from the device, the light must be able to travel through an adequate length of sample. Hellma’s cuvettes are designed for small volumes, without compromising light path distance.

Photodiode

To analyze the difference in the brightness, as voltage, between the initial LED light and the impeded light due to alternating molecules in the sample fluid, a photodiode will be utilized. Photodiode devices are capable of converting light energy into electrical energy as electric charge or a voltage. The effectiveness of the photodiode to translate light energy into electrical is given by the quantum efficiency equation:

\[
Q.E. \ [% \ based \ on \ 100] = 1.24 \times 10^5 \frac{R \ [A/W]}{\lambda \ [nm]} \quad (Eq.5) \tag{4}
\]

\[\text{www.centrovision.com} \]

\[\text{19}\]
Where $\lambda$ is the wavelength of the light and $R$ is the responsivity of the diode. If the photodiode were to be connected to a resistor, in this case, the sample fluid, it would function as a current source; thus, the change in voltage due to the sample would be proportional to the power of the radiated light.

Photodiodes are usually assembled from highly purified silicon wafers. There is a direct relationship between the silicon and the electrical resistivity, that is, the greater the purity of the silicon, the greater the resistivity of the device. A typical resistivity range for a photodiode is between 10 Ohm-cm and 10,000 Ohm-cm, indicating a wide range of current opposition. The basic construction of a photodiode is indicated in the following sketch:

![Figure 4 – Basic Construction of Silicon Photodiode](image)

Whereby the N-type silicon is the material used to construct the photodiode, and forms a junction with the “P” material, usually made of boron. The back contact
represents the device cathode, and the front contact represents the anode. The silicon nitride used for passivation coating functions as an anti-reflection coating and protects the active area of the diode. This coating regulates which wavelengths can be detected and thus is varied to accommodate various irradiation waves.

The depletion region represents an area where the silicon is exhausted of electrical charge. This region is the most sensitive to radiation, hence it is an integral component of the device, and can be directly regulated by increasing and decreasing the bias voltage across the region, thus increasing and decreasing the depth, respectively. The stored energy of the device, the capacitance, is also dependent on the thickness of the depletion region, as demonstrated in a graph of capacitance versus voltage and area:

![Figure 5 – Junction Capacitance, Voltage, Area Graph](image)

When the photodiode is used in practice, light is absorbed in the active area, protected by the silicon nitride, and an “electron – hole” pair forms. These electrons and holes are separated in the PN junction as the electrons move into the N region and the ‘holes’ move into the P region. The result is a phenomenon known as the
“photovoltaic effect”, whereby a current is generated which is facilitated by the light absorbed.

For the device in question, a concentrated light source will be utilized which will be subsequently reduced in intensity by polarizing discs and the actual sample fluid. Thus, a low level light sensor, such as the OSD35-LR presented by Centrovision, will be the optimal device to use. The low level sensor is equipped with a large area silicon diode, which is designed for setups that require a current extender, as with the device in question. The sensitivity of the sensor is measured by the current of the diode, given as amps/cm$^2$, over the radiant energy absorbed, given in watts/cm$^2$, which equates to the responsivity of the device given in amps/watts. The responsivity of the OSD35-LR is presented below:

![OSD35-LR Responsivity Graph](image)

Figure 6 – Responsivity Curve of Low Level Light Sensor

The LED that will be utilized for this device functions at a wavelength of 635nm. This wavelength is appropriate for absorption by this sensor, as indicated by the arrow on the graph, and thus will provide the user with suitable results.
Sending the Signal to the Computer

Our client wants the final fluid properties as well as the raw data to be displayed on a personal computer. This will allow our client the ability to have more thorough documentation of the measurements and more freedom in evaluating the sample’s properties. An impedance analyzer chip (AD5933; Analog Devices) will measure the impedance and convert the signal to digital. The data will then be prepared for USB communication with the computer with a MAX3346E (MAXIM Dallas Semiconductor) microprocessor. The device can then be connected to the personal computer through an USB cable. Overall circuit performance will be tested by selecting test points with predictable outputs throughout the circuit that can be measured with an oscilloscope.

The client also prefers a wall outlet power source. A commercially available power supply will be used to power the device. Functionality of the power supply will be tested using a digital multi-meter.

Calibration setup

Initial calibration is essential in proper results for this device. Before any sample is measured, the device must be calibrated for that specific sample. The second polarizer will be aligned so that when the molecules are fully aligned in solution, the maximum amount of light is received at the sensor. Therefore, when the alternating current is turned on, the graph of brightness vs. time will correspond to the alternating current being applied. This will make the calculations simpler.

Constants for the equations for finding loss and storage modulus will be found through recording the net rotation of light when there are no solute molecules in the
sample.

**Computational Analysis**

Since this approach has not been experimentally tried before, equations that calculate the loss and storage modulus are not readily available. Preliminary equations\(^5\) have been derived for a very similar polarized setup envisioned by our client. From those equations the following formula for viscosity \(\eta\) was derived (Eq. 6):

\[
\eta = A t \frac{-18 \ln(2 \rho) - 14.4}{L^3} \quad (\text{Eq. 6})^5
\]

Where \(\rho\) and \(L\) are the density and length of the molecules and \(t\) is the time the light takes to decay exponentially after a voltage pulse is applied to the sample.

\[
A = \frac{kT}{\ln\left(\frac{\Delta n}{\Delta n_o}\right) \pi t} \quad (\text{Eq. 7})^5
\]

The constant \(A\) will be determined through calibration. Where \(k\) is Boltzmann’s constant, \(T\) is absolute temperature, and \(\Delta n\) is the following (Eq. 8):

\[
\Delta n = \frac{\lambda \delta}{2 \pi l} \quad (\text{Eq. 8})^5
\]

Where \(\lambda\) is the wavelength, \(\delta\) is the phase difference and \(l\) is the length of the light path.

The previous equations are not specific to our design and would need to be changed. Those equations were calculated for a single pulse of current after which the

\(^5\) from Kalonia; personal communication
time of decay of the brightness of the light was observed. Our design involves using an alternating current. This means that we would have an alternating current as input and an output of an alternating current with a frequency shift and change in magnitude. This is the same output that is observed with the piezoelectric device (see optimal design for details). The same equations that are used to calculate storage and loss modulus for the piezoelectric setup could be used for this design (Eq. 1\(^1\) and 2\(^1\)).

\[
G'' = \frac{2R_1X_2}{A^2 \rho_{liq}} \quad \text{(Eq. 1)}
\]

\[
G' = \frac{R_2^2 - X_2^2}{A^2 \rho_{liq}} \quad \text{(Eq. 2)}
\]

An impedance analyzer chip (AD5933; Analog Devices) will be used to calculate R and X, the real and imaginary change in impedance. A is a constant that will be determined during calibration. \(\rho_{liq}\) will be dependant on the length of the molecule and the density of the sample. How \(G'\) and \(G''\) depend on the length of the molecule and its density for the polarized light setup will not be known until preliminary testing can be done on multiple known samples.

**Displaying the Results**

The raw and analyzed data will be displayed on a personal computer, as requested by the client. The program will accept the digital impedance sent from the device and display the input in a time vs. impedance graph in real time. The impedance will be compared to the calibrated impedance in order to determine the \(R\) and \(X\) values used in Eq. 1 and 2\(^1\) (the electrical real and imaginary impedance of the sample). The values for \(G'\) and \(G''\) (Eq 1 and 2)\(^1\) will then be calculated and displayed. Figure 7 is a flow chart of the proposed program.
Figure 7: Flow chart of Computer Program
Piezoelectric Crystal Method-Optimal Design

Design

Our design consists of four main parts: 1) a waveform generator with the capacity to create a frequency sweep, which stimulates a piezoelectric crystal. 2) A phase detector and two tRMS chips that can compute the phase and admittance of the signals from the crystal. 3) A data acquisition device that sends voltage from the computer to the circuit board to initiate a frequency sweep and sends data from the circuit to the computer to be analyzed by the 4) LabVIEW ® computer program, which, through an easy to use user interface, allows the operator to not only select a frequency range to excite the crystal at, but also has a graphical indicator of the raw impedance versus frequency and phase versus frequency data and calculates the $G'$ and $G''$ of the sample analyzed.

Crystal Setup

The focal measurement of this design is taken by a piezoelectric ceramic device, in this case, a quartz crystal. The crystal consists of a quartz plate, natural or cultured, with two gold plated electrodes, which provide contact points for electrical signals. The mechanics of the piezoelectric crystals can be equated to a relatively simple electrical circuit, as follows:

![Crystal Circuit](image)

Figure 8: Crystal Circuit

Where $C_o$ is the capacitance due to the electrodes on the crystal plate plus the stray capacitances due to the crystal enclosure, and is applicable regardless of the crystal oscillations. $R$, $C_1$ and $L_1$ are the equivalent motional arm resistance, the

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motional capacitance of the quartz, and the motional inductance, which is a function of mass, respectively. The ratio of $C_0$ to $C_1$ is equal to the constant $k$, the piezoelectric coupling factor. Although the crystal equivalent electrical circuit appears simplistic, the method of calculating frequencies is somewhat complicated, and can be found using the following equations:

- $F_s$ (Series resonance frequency) = $1 / [2\pi(L_1C_1)^{1/2}]$ (*Eq. 8)
- $F_p$ (Parallel Resonance frequency) = $f_s[1 + 1/(2\gamma)]$ (*Eq. 9)
- $k$ (Capacitance ratio) = $2\pi f_s C_0 / C_1$ (*Eq. 10)
- $Q$ (Quality factor) = $2\pi f_s L_1 / R_1$ (*Eq. 11)
- $M$ (Figure of merit) = $Q / \gamma$ (*Eq. 12)

The displacement of the crystal penetrates the fluid on top only to a known depth, which is quite small. As the crystal vibrates, the fluid on the electrode disperses, covering the majority of the metal. However, if the fluid does not cover the entire electrode, the results will be inaccurate. Thus, to combat this problem, the top electrode, where the fluid sample is deposited, is made larger than the bottom electrode. This eliminates any errors due to insufficient fluid dispersion, as the fluid is allowed to occupy an area larger than that which is being measured. A signal is applied to the crystal through one of these electrodes, thus causing the piezoelectric ceramic to expand and contract. The physical displacement on the x, y, and z-axis produces shear waves, which propagate as transverse waves in liquid media.

The customer has requested a rheometer that functions over a range of frequencies, from approximately 3 to 8 MHz. Thus, this “sweeping” mechanism will be employed using both the computer and the MAX038 waveform generator. The method for creating this sweep will be further discussed in the circuits section. The range over which the frequency should be evaluated for is obtained by estimating the resonant frequency of the specific piezoelectric crystal in use. The resonant frequency, or series frequency, is the frequency at which the ceramic most readily
vibrates in response to applied voltage, and is the point where it and most efficiently converts electrical energy to mechanical energy. This frequency is represented as a dip in the log impedance versus frequency curve, and occurs at the minimum impedance, or maximum admittance, for the system, as seen below \[^{7[6]}\]:

![Figure 1.8 Impedance as a function of frequency](image)

Figure 9: Impedance as a function of frequency\[^6\]

The term “\(f_s\)” is the series resonance frequency and is roughly equal to “\(f_m\)” the resonance frequency. “\(f_p\)” is the parallel resistance in the crystal and is roughly equal to “\(f_n\)” the maximum impedance frequency.

Once this resonant frequency is established, a range about that point can be established, so as to ensure accurate findings. The resonant frequency is an intrinsic characteristic of each quartz crystal. The crystal is cut in very precise and specific ways so as to ensure a resonant frequency at the point which the buyer desires. Not only is there an intrinsic resonant frequency, but there also exist harmonic overtones of this frequency that are inherent to each individual crystal. These overtones are essentially multiples of the resonant frequency. That is, if the resonant frequency is represented as \(F\), the overtones will be \(3F\), \(5F\), \(7F\), and so on, and appear as similar wave functions with a decreased change in impedance. A physical representation of harmonic overtones is demonstrated here by vibration direction:

\[^{7[6]}\] [www.americanpiezo.com](http://www.americanpiezo.com)
The typical range of frequencies for crystals used for QCM, or quartz crystal microbalance, applications is between 3 and 200 MHz. This range will adequately fulfill the requirements of the design specifications.

For this design, the piezoelectric crystal will receive a sinusoidal signal from a waveform generator. The initial sine-wave signal will be established, and the phase and admittance for this signal will be calculated. The crystal will then be loaded with the sample liquid, and the phase and admittance for this impeded waveform will be calculated. Loading the crystal simply means that a drop of the sample liquid will be placed atop of the gold plated electrode on the crystal. It should be noted that the electrode is composed of gold plated chromium, ensuring that the fluid in question will come in contact with only inert metals, thus no destruction of the sample should occur. The phase shift between the signal of the unloaded crystal and the loaded crystal will be analyzed by the LabVIEW program. The admittance will be sent via the tRMS chips and the data acquisition device to the computer to be analyzed. From this data, as well as a calibration constant “A” and the known density of the fluid in question, loss and storage modulus can be found. This device can be tested in a
pharmaceutical laboratory, with a fluid of known loss and storage modulus and a previously designed piezoelectric rheometer, to determine if the calculated values are accurate.

A benefit to this design is that the circuit board that was designed, which will serve as the crystal stimulation and primary form of data acquisition, is able to function over a wide range of frequencies, due to the frequency sweep function. Thus, a variety of crystals can be used with this device with little adjustments necessary. However, since several crystals must be tested to find the range, a larger amount of sample fluid is wasted during testing, and the user loses a degree of ease when experimenting. Also, the raw data graphs of impedance versus frequency and phase versus frequency will allow the user to determine if the resonant frequency is being applied, or a harmonic overtone of that frequency. Although the harmonic overtones could theoretically be used as secondary resonant frequencies, which would allow for a greater variety of usable scans, the use of overtones greater than the fifth multiple will leave the user with amplitudes and phase shifts that are too small to record, thus the integrity of the experimental values is questionable.

Piezoelectric crystals are manufactured in two different cuts, AT and SC. Their main manufacturing difference is that AT crystals are singly rotated cut, while SC crystals are doubly rotated cut, as demonstrated below.
The AT and SC cuts are each capable of a resonance frequency range from 1 MHz to 250 MHz. The AT crystals are super-sensitive to stresses in the body of the resonator that are caused by outside forces, however they can be cut to ensure a low temperature coefficient, or low shift in frequency due to temperature, and thus will be used with this device. While the SC crystal has a slightly higher performance reliability under perfect conditions, it can show a significant frequency shift due to temperature gradient, and thus would not be ideal for this application.

The crystal is connected to the circuit from the outside of the device through alligator chips. This will not only allow for interchangeable crystals, but will also allow for the crystal to be placed within the refrigeration system that the client currently utilizes. The crystal is connected to the two alligator clips, with one clip feeding in the signal from the waveform generator, and the other clip sending the output signal from the crystal to the rest of the board.

**The Circuit**

The basic design of the board employs the use of the MAX038 waveform generator to create sine waves on the order of 7 MHz. This frequency can be adjusted to create a sweep, as previously mentioned, by hitting pin 8 of the chip with a voltage proportional to the desired frequency. The voltage is sent to the chip from the
computer, via the data acquisition device. The voltage can be calculated by the following equation: voltage required to alter frequency = (center frequency of the chip – desired frequency) / (0.2915 x center frequency of the chip); where the center frequency of the chip is the frequency of the output signal when no outside voltage is employed. This signal is sent through a power op-amp, which essentially shields the MAX038 chip and protects it from potential current overload by the crystal as well as amplifies the signal, which in turn sends the signal to the piezoelectric crystal. The signal from the crystal is then sent through two inverting op-amps, which convert the original voltage to a positive, which is proportional to the current. The signal is then taken from a line directly before the crystal and sent to one t-RMS chip and the phase detector. The signal from after the second op-amp is taken from a line and sent to the other t-RMS chip and the same phase detector. The output from the two t-RMS chips and the phase detector is then sent via the data acquisition device to the computer. The board operates on a +5/-5 volt power supply, and thus a +5 volt regulator and a -5 volt regulator were incorporated into the design to ensure that the correct voltage output was always sent to the board. These also protect the board from voltage overload, however do not guard against an “underload”. A schematic for this circuit appears as follows:
Figure 13 - Final Schematic

Where the MAX038 is the waveform generator; IC03, IC04 and IC05 are AD9631AR op-amps; IC10 is the -5 volt regulator UCC284; IC09 is the +5 volt regulator ADP3367; IC07 and IC08 are AD637 tRMS chips, and IC06 is the AD8302 phase detector. The printed circuit board appears as follows:
Figure 14a - Final PCB 1

Where “C” indicates a capacitor; “R” indicates a resistor; “XT” indicates the crystal input and output; “pot” indicates a potentiometer resistor; and the various in and out lines are for the input and output of the voltage, tRMS and phase signals.

The first prototype of the printed circuit board appears as follows:
This board, however, required redesigning, as the right half of the board was completely remodeled and the red +5 voltage plane had to be eliminated due to shortage problems.

**Analysis of the Phase Shift**

A phase shift results from the change in phase between the input signal entering the crystal and the output signal from the crystal. The phase difference is caused due to the damping mechanism of the crystal causing a lag of the output frequency. This shift does not change the frequencies before and after the crystal. The two signals are then fed into an AD8302 phase shift detector which analyzes a dc output for the phase shift between the two input signals. This is done by taking one input as a reference, and multiplying both sine waves together using digital logic algorithm. The basic idea behind finding the gain factor is taking one reference input and sweeping the other input in amplitude, while measuring the voltage magnitude of the amplitude. Providing these sine wave input signals a dc output is provided that ranges between 0 and 1.8 volts. A zero degree change in phase shift gives off an
output of 1.8 volts and a 180° shift is a 0 volt dc output. This chip also covers the range between 0° to -180° but with a negative slope. The dc output from the phase detector is then sent to the computer for analyzing the crystal impedance.

Figure 1 demonstrates the measurement mode for the phase and output gain.

![Phase versus Voltage gain](image)

The pin configuration and circuit setup for the AD8302 is shown in figure16, and figure 17 respectively.

![Phase Detector Pin Configuration](image)
Wide Band RMS to DC Converter

The true rms to dc converter evaluates the root mean square of sinusoidal inputs. This IC chip computes true rms values up to 8MHz, which is compatible with the design specifications. In this design there are two rms to dc converters used, one for the signal coming before and the other after the crystal. The first converter gives a dc output for the voltage supplying the crystal, while the second converter gives a dc output proportional to the current through the crystal. The dc outputs are then sent to the computer with the output from the phase detector. Figure 18 is a pin diagram for the AD637 rms to dc converter.
The pin configuration shown in figure 19 is for an SOIC surface mount board. The circuit connections for the rms to dc converter are shown in figure 5.

![Figure 19: Circuit connection for TRM to dc Converter](image)

**Power Connections**

The circuit is supplied with a voltage of ± 5 volts for proper operation. A voltage regulator is used for acquiring the voltage needed within a narrow band for circuit protection. Two different IC chips are used one supplies the + voltage, while the other supplies the – voltage supply.

The negative voltage supplier has a safety feature that limits the amount of current. At overload this chip will automatically turn off for a delay period, and then it will turn back on after a period that is forty times longer than the off period. This chip has a wide input voltage range between -3.2 to -15 volts with a steady voltage output. Figure 6 shows a pin configuration for the UCC284 negative voltage supply IC chip.
The positive voltage regulator works similar to the negative voltage regulator. The ADP3367 is used to give a +5 voltage output, and can supply current up to 300mA. The chip operates at a wide voltage range between +2.5 to +16.5 volts, and has a high accuracy output voltage with a ±2% error. The ADP3367 has a very easy circuit configuration that is shown in figure 21.

A medical desktop adaptor is used as a power source for easy device mobility. The adapter operates at a voltage range between 88 to 264 volts, with a low noise output regulation of 0.5%. This allows the device to take power supply from any wall outlet and can be used according with European standards.

**Sending the Signal to the Computer**

Our client wants the final fluid properties as well as the raw data to be displayed on a personal computer. This will allow our client the ability to have more
thorough documentation of the measurements and more freedom in evaluating the sample’s properties.

The phase, voltage and current from the circuit will be transmitted to the computer through a NI 6008 data acquisition device. This device is ideal for our project. It allows up to 8 12-bit analog input channels, 2 analog output channels, 12 digital input/output channels and one counter. With the NI DAQ device, we are able to take in data for analysis as well as control the device with output from the DAQ. This device was chosen to allow the rheometer to be controlled through the computer. It communicates with the computer through a USB connection and is also powered by that connection. Figure 22 depicts the NI 6008 DAQ device.

![Figure 22: National Instruments 6008 Data Acquisition Device](image)

**Background on Determining Fluid Properties from Impedance Measurement**

Figures 14 and 15 are circuit representations of the crystal when it is unloaded and loaded with the sample fluid. Where $C_0$ is the static plus stray capacitance, $R_1$ is the losses in the crystal due to friction or heat loss, $C_1$ is the energy stored in the crystal during each oscillation, $L_1$ is the inertial mass of the crystal, $R_2$ is the real component of the motional impedance due to the load, and $X_2=\omega L_2$ is the imaginary
component of the motional impedance due to the load.

![Figure 23: A circuit representation of the unloaded crystal.](image)

Our client is interested in measuring two fluid properties, loss modulus $G''$ and storage modulus $G'$. In equation 1 and 2, $R_2$ is the change of the real part (resistance) of the impedance when the crystal is loaded. $X_2$ is the change in the imaginary part of the impedance when the crystal is loaded ($X_2 = \omega L_2$). $A$ is a constant associated with each crystal. $\rho_{\text{Liq}}$ is the density of the sample.

$$G' = \frac{R_2^2 - X_2^2}{A^2 \rho_{\text{Liq}}} \quad \text{(Eq. 1)}$$
\[ G'' = \frac{2R^2X^2}{A^2 \rho_{liq}} \] (Eq. 2)

Determining both \( G'' \) and \( G' \) are important in studying Non Newtonian fluids, which are fluids that store some of the energy that is applied to them. Energy storage occurs in fluids with large molecules at high concentrations where molecular movement is inhibited by neighboring molecules. The intermolecular interaction that causes energy storage is an important fluid property that must not be confused with viscosity. However, in many viscometers, the storage modulus is ignored and causes the measured viscosity to deviate from actual rheometric characteristics. This is not a problem with Newtonian fluids because with Newtonian fluids \( X_2 = R_2 \) which causes \( G' \) to go to zero. The following equation is normally used when calculating Newtonian viscosity.

\[ G'' = \eta_{liq} = \frac{2R^2X^2}{A^2 \rho_{liq}} \] (Eq. 3)

Each crystal must be measured in order to determine the constant \( A \) before any fluid properties can be obtained. A fluid with known density and loss and storage modulus will be used to determine the constant \( A \), which is equal to \(^1\):

\[
A = \frac{N \pi}{4K^2 \omega_s C_0 Z_q} \] (Eq. 4)

Where \( N \) is the overtone number, \( K \) is the piezoelectric coupling constant, \( \omega_s \) is the series resonant frequency, \( C_0 \) is the static plus stray constant, and \( Z_q \) is the quartz mechanical impedance. \( A \) is necessary because it allows us to calculate the mechanical impedance from the measured electrical impedance (where \( Z_{Elec} = A*Z_{Mech} \)). Therefore, the electrical values (\( X_2 \) and \( R_2 \)) can be used in determining \( G' \)
and $G''$ when $A$ is included in the equations.

Fluids with known $G'$ and $G''$ values will be used to calibrate the device (determine the constant $A$).

**Details of the LabVIEW Program**

Through the graphical user interface, the user can choose from four different tasks: unloaded, loaded, find loss and storage modulus, and find $A$. Each will be discussed in detail below.

Unloaded: The unloaded function records the phase, voltage, current and frequency of the crystal when it does not have a liquid load applied to it. The data is saved to excel sheets.

Loaded: The loaded function does exactly the same thing as the unloaded function, but the data is saved to a different file. For both the unloaded and loaded functions, the user can select the frequency sweep range and the step size. Below is the block diagram for the unloaded and loaded functions.

![Figure 25: Unloaded and Loaded Labview block diagram](image)

Find Loss and Storage Modulus: This function calculates the loss ($G'$) and
storage (G") modulus through the following equations\(^8\). The user must input the value \(A\), and the density of the sample liquid.

\[
L_1 = \frac{1}{C_o \left( (2\pi f_p)^2 - (2\pi f_s)^2 \right)}
\]

\[
C_1 = \frac{1}{L_1 (2\pi f_s)^2}
\]

\[
L_m = \frac{1}{C_1 (2\pi f_s^l)^2}
\]

\[
R_2 = \frac{1}{G^l} - R_1
\]

\[
L_2 = L_m - L_l
\]

\[
X_2 = 2\pi f_s^l u L_2
\]

\[
G' = \frac{R_2^2 - X_2^2}{p_{liq} A^2}
\]

\[
G'' = \frac{2R_2 X_2}{p_{liq} A^2}
\]

Where \(L_1\), \(C_1\), \(C_o\), \(R_1\), \(L_2\), \(R_2\) are defined in the following model\(^9\) of the crystal and variables with \(m\) subscripts refer to the motional arm of the circuit. Where \(L_m = L_1 + L_2\), \(R_m = R_1 + R_2\). \(X_2\) is the imaginary component of the impedance of the liquid. The

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\(^8\) Obtained with help from Atul Saluja, University of Connecticut

values $f_s$ and $f_p$ are the resonant frequency and the parallel frequency of the crystal.

![Circuit representation of piezoelectric quartz crystal](image)

**Figure 26:** Circuit representation of piezoelectric quartz crystal

The loss and storage modulus are calculated from the data saved in the unloaded and loaded functions. This function also graphs all the relevant data.

![Resulting graph from the Labview user interface](image)

**Figure 27:** Resulting graph from the Labview user interface

The following is the block diagram of the Find Loss and Storage Modulus function.

This part of the program uses MATLAB script to perform the calculations and to plot...
the graph.

Figure 28: Find Loss and Storage Modulus Labview block diagram

Find A: The find A function calculates the A value found in the $G'$ and $G''$ equations. This constant is dependant on the crystal and the circuit. This is done by using a sample of known $G'$ and $G''$. The user will first have to run the crystal unloaded and loaded and then use this function. The user will have to provide the $G'$, $G''$, A, and density of the known liquid. The value of A must be found before running tests on any unknown samples. Below is the block diagram of the find A program. It is essentially the same as the Find Loss and Storage Modulus except it requires different inputs from the user.
Figure 29: Find A labview block diagram

Enclosure

This device calls for an enclosure with the following properties:

* RF shielding - since this device operates at high frequencies and is affected by radio transmissions
* Easily machinable – since we need to make holes for the power supply, the crystal, and the USB cable
* Approximate dimensions of 4 X 3 X 5 inches
* PCB mounts

Pomona (distributed by Newark for $54.86 with part number 3742; dim: 4.13 X 2.68 X 6.16 inches) makes a product that meets these requirements. Below is the layout of the enclosure. There are slots for PCBs and room for the data acquisition device. This enclosure will provide room for expansion. If more crystals, and therefore more circuit boards, are needed there are plenty of slots and enough room for additions.
Figure 30: Dimensions of the enclosure
The crystals are located on the outside of the enclosure. They are connected to the printed circuit board through alligator jacks. Alligator jacks were decided upon because our client currently uses a water jacket to obtain a desired temperature and therefore needs to have good mobility with the crystal.

**Optimal Design**

It was decided after careful consideration of the three possible designs that the piezoelectric crystal method is the most appropriate for this project. The crystal method utilizes two equations derived from the Stokes - Einstein equation, equations 1 and 2, of which both have been proven to find loss and storage modulus from impedance shifts. These equations are reliable and precise, essential for accurate
rheological property modeling.

This design has been considered to be optimal by not only the designers, but by the client as well. Many devices on the market today measure the loss and storage modulus of sample fluids with shear forces. With the majority of these rheometers, the forces are applied directly to the sample to find these fluid properties. Often, when this force acts as a stress on the sample, that sample will begin to degrade. A relationship exists between the force applied to the sample and the storage modulus, which these devices do not account for. Because of this, the values of the fluid properties are skewed and not completely accurate. The client proposes to use this device for the study of protein concentrations that are intended for in vivo injection. Errors in measurements, even slight, could completely alter the expected outcome of experiments on these concentrations. For this reason, the piezoelectric crystal is the optimal design. The crystal method uses shear waves to measure the impedance of the liquid without applying the force directly to that sample. Thus, the risk of degradation of the molecules due to stress is virtually eliminated. As such, the impedance measured for the liquid will be accurate, as will the results from the equations for the measurement of loss and storage modulus.

Not only will this optimal design eliminate error due to sample degradation, it will improve accuracy by establishing a relationship between storage modulus and the frequency applied to the crystal. As the frequency of the signal exciting the piezoelectric crystal is increased and decreased, the storage modulus of the sample also changes accordingly. This design will function with a number of crystals, each resonating at a different given frequency; thus the sample loss and storage modulus can be found over a variety of frequencies. From this, a graphical analysis of these two factors could establish a relationship, and more accurate readings.
**Realistic Constraints**

The machine must be constrained to manufacturing and economic standards in order to be successfully mass-produced. Economically, the viscometer must be constructed of parts with a total value of no more than 1000 US dollars. To keep costs low, and to ensure that the machine will be correctly assembled, the overall design and assembly process will be specific enough for “non-engineers” to piece together. Also, for the sake of efficiency in manufacturing and budgeting, the machine will use widely available parts, when appropriate, as opposed to custom designed components. Schematics and directions will be available for manufacturers.

The viscometer will be designed for long-term use. To prevent the need for constant component replacement, durable, forgiving parts will be used during construction. The parts will be the most resilient pieces that fit to the budget. In the event that a user experiences difficulties operating the viscometer, an easily accessible “trouble-shooting” manual will be provided with the machine. This should guarantee proper usage, and thus a longer life to the device.

The design must be developed with environmental, ethical, social and political standards in mind. In the event that the machine must use a power supply, the voltage of the power supply should be high enough to run the machine, but should not be so high that it would be detrimental to the environment surrounding it. We do not foresee the device being in a position to violate any social, ethical or political standards, so long as it functions only as a small volume. However, due to the fact that we cannot monitor the usage of the viscometer, the device will be built such that its only use is to measure the loss and storage modulus of minute volumes of liquid. Though ideally the intentions of the buyer would be investigated, this procedure would be unrealistic in a business setting. Thus, the only protection from social, ethical and political deviants that we can supply is to ensure that this device is only
able to perform measurement tasks.

**Device Safety Features**

One of the most fundamental requirements to any mechanical operator is a device safety feature. It is of extreme importance for the consumer to operate the device without being at health risk. This is why safety features have been conducted into various components of the design which will ultimately reduce any risk factors.

Any device manufactured needs to be able to self shutdown safely without causing harm to the user. Since many devices are subjected to overheating due to the extensive power supply a method is conducted which will allow the circuit to disconnect which eventually terminates operation.

In this design voltage regulators are used to keep the voltage supplying the circuit within a narrow range. In the case of power overload the voltage regulator will turn off for a period of time then restart. In extensive power over load the voltage regulators will completely burn preventing power from reaching the rest of the printed circuit board.

Other included safety features are insulating bear wires that can ultimately lead to wire shorting. Uncovered wires can also produce the risk of electric shocks towards the user, with this design coax cables are used to help prevent any type of hazard.

The design will also be safe from sharp edges for a more comfortable usage. Sharp edges can be covered with a non conductive rubber material that will not affect measurement accuracy. They can even be replaced with curved edges were applicable, this will be determined with device progress.

The design includes a friendly user interface using lab view program that will unlikely have any safety risks of operation.
**Conclusion**

A rheometer can be used by a wide variety of research scientists to accurately calculate properties of sample liquids. Rheometers available for purchase on the scientific market today are designed to utilized relatively large volumes of fluids, thus limiting researchers to samples that are available in larger quantities. For fields of research that require very small volumes of test samples, such as protein studies in pharmaceuticals, rheometric measurements have thus far been quite difficult. However, with the advent of our prospective design of a minute volume rheometer, these difficulties will be eased. As a team biomedical engineers we have found that this device would satisfy the requirements requested by our client, which can also be used by other fellow scientists who need to acquire accurate data on physical properties of fluids.

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**Appendix**

Specifications
- Must ultimately display the storage and loss modulus of sample liquid of sample volumes of less than 20 μL

- User interface display on personal computer with raw and analyzed data

- Power supplied from wall outlet source

- Measurement accuracy within 1% of documented values for the loss and storage modulus (2-3% would be acceptable while >5% would be intolerable)

- Design for use in average research laboratory

- Operation at room temperature

- If shear waves are used for measurement, frequency must cover the range of 1kHz to 1MHz