Impact of ingredient selection on rheological properties of a semi-liquid syrup model for use in pulp/paste candy

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Abstract

Pulp/paste candy is popular in Mexico and is gaining popularity around the world. Pulp/paste candy is characterized as a soft, semi-fluid candy with particles in a colloidal suspension, typically squeezed out of the package for consumption. It is made by hydrating gums in water and mixing them with corn syrup, acid, powdered/pulverized sugar, and other minor ingredients yielding a product around 80°Brix and pH between 2-3. Over time the sucrose in the candy tends to invert, causing two types of failure: package leakage and solidification in package. Based on the findings of previous work from Molina-Rubio et al. (2010), a modified semi-liquid syrup model system was created with corn syrup, sugar, gums, and water. The model system was used to identify the influence these ingredients had on viscosity and texture since these factors are linked to the typical modes of failure in pulp/paste candy. An oscillatory sweep was used on a controlled force rheometer to identify the linear viscoelastic range. Oneway ANOVA with Tukey HSD was used to compare % total solids levels with complex viscosity at 0.1 rad/sec (there was a significant difference between all levels) and minimum tan (delta) (mid and high level were similar). Using a stepwise method, ANOVA models were generated that showed statistically significant effects on complex viscosity for gum level and sugar level as well as interactions ($p < 0.05$) between invert syrup-water, gum-water, and sugar type-water. The type of gum and the amount of corn syrup used didn't significantly impact on the viscosity of the system. Using probe tests helped to analyze samples that were too thick for the rheometer. Analysis showed an inflection point for exponentially increased hardness (85-93%TS) that should be further investigated. The stepwise regression model generated for stickiness showed that the invert syrup-water interaction was significant along with gum type. These results are applicable to the confectionery industry and can help companies test and create a candy that

meets the packaging and shelf life constraints that they desire. Targeting invert syrup-water levels and the hydration of the gum will have the most impact on the final product's viscosity and stickiness, which are important for primary package filling and storage. To create an easy to eat candy, no invert syrup should be used in formulation and gellan gum would be better to use than xanthan. Higher solids (> 85%) should also be avoided since it would create a candy that is harder to squeeze out of the package due to higher viscosity and textural hardness.

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Chapter 1 - Pulp/Paste Candy Background, Background of Rheological Properties, and a Literature Review of Past Studies Applicable to a Semi-liquid Pulp/paste Candy System

1: Background on Pulp/Paste Type Candy

Paste/pulp candy is generally described as a semi-liquid candy that may or may not have real fruit with particles in a colloidal suspension that is squeezed out of a package. The candy may or may not be spicy and is very popular in Mexico. Examples of this type of candy are: Crazy Hair's "Gooey Fruit Candy" (USA), Sonric's "Volcano" (Mexico), De la Rosa's "Pulparindo" (Mexico), Lucas' "Pelucas" (Mexico), and Hershey's "Pelon Pelo Rico" and "Crayón" (Mexico). In 2001, $166.6 million worth of candy was imported into the United States from Mexico and much of the candy is spicy and appeals to the growing Latino population's tastes (Zúñiga, 2002). Despite the popularity of the candy, not much literature about pulp/paste candy has been published, but several studies on sucrose, glucose, fructose-hydrocolloid mixtures, sucrose nucleation, and acid catalyzed inversion in other candy products have been published. The complex nature of pulp/paste candy brings unique challenges to product developers when trying to determine what the driving factors are for shelf life failure. Since pulp/paste candy is a sugar based colloidal suspension, literature about sugar syrups, gum networks, and sucrose crystallization might be relevant since this complex candy shares all of these aspects. This literature review will provide an overview of processing, typical shelf life conditions, and potential defects of pulp/paste candy before reviewing other studies that looked at similar aspects in other candy products or model systems, with the intent of applying that knowledge to pulp/paste candy systems in the future.

Pulp/paste candy is generally made by hydrating a gum in water and then adding that solution to a mixture of corn syrup, acid, buffer, and salt. This mixture is then heated and mixed. During mixing, sugar is added as a bulking agent, along with the color, flavor, flour/chili powder, and any preservatives that might be used. The final solids are around 80-90°Bx (Molina-Rubio et al., 2010). For filling and packaging the pulp/paste candy is kept at or below 45°C for less than 1.5 hours to limit inversion. The reducing sugars should be kept to less than 5% to prevent excess moisture pick up, which is a similar parameter used for hard candy (Edwards, 2000). Over the typical shelf life of 12 months, the candy tends to undergo inversion and the more hygroscopic glucose and fructose are created from sucrose. Typically, pulps have a low pH between 2-3 and a water content between 10-12.5% (Molina-Rubio et al., 2010). The average storage temperature for paste candy is at ambient temperatures between 22-27°C in Mexico and the US, with an average relative humidity between 50-80%. Over time the sucrose in the pulps tends to invert and lose moisture causing two modes of failure—an inability to be squeezed from the package or package leakage. These modes of failure are often studied in industry by dynamic mechanical analysis (DMA) and mechanical texture analysis.

2: Background of Rheological Properties

Typically, viscoelastic materials, including most food products, use DMA and are first measured with a strain or amplitude sweep to determine the strain amplitude dependence of the storage modulus (G') and the loss modulus (G") (Gunasekaran & Ak, 2000). This amplitude sweep shows if there is independence between these two moduli and whether or not there is linearity observed (i.e. the Linear Viscoelastic Region, or LVR). If the strain becomes too much the critical strain is reached. At this point the material's structure is broken and the line begins to descend. It is important for further characterization of the material to be done with a strain that is within the LVR prior to the critical strain to allow for comparison between the methods, since at this point G' is nearly independent of frequency. The next test typically run to try and characterize viscoelastic materials is a frequency sweep. In this, measurements are made across various frequencies, while holding the strain constant. More fluid like behavior is identified with G" > G' and more solid like behavior is identified with G"< G'. In Figure 1.1 it is evident that tan (δ) is equal to G"/G' and can be used to quantify the relationship between the storage and loss moduli. In addition to G" and G' complex viscosity, η*, can also be examined. Complex viscosity is equal to the shear modulus divided by frequency ($\eta^* = G^*/\omega$). The Cox-Merz rule indicates that frequency and shear rate can be considered synonymous in a steady state region and that the complex viscosity from oscillation can be used interchangeably with the shear-viscosity (Rao, 2007). Since shear-rate experiments are not possible at extremely high viscosities, this provides a convenient means to obtain the same information using oscillation experiments within the LVR instead (Kulicke & Porter, 1980). Tan (δ) can also be used to characterize viscoelastic behavior as illustrated with Figure 1.1. These types of analysis can also be applied to a model system for determining viscosity in a semi-liquid model system.

The Texture Analyzer is an instrument that is typically used to measure hardness on many food samples (Foegeding & Steiner, 2002). Kilcast and Roberts (1998), characterized hardness as a relationship between the force of the probe penetrating and the cohesiveness between the molecules within the treatment. This test mimics how hard it is to squeeze candy from a package. Adhesive force was characterized as the peak force withdrawn from the sample and was the relationship between the intermolecular bonding of the treatment and the probe and stringiness was the distance between the sample surface and the point where the force dropped to zero. (Kilcast & Roberts, 1998).

A relevant study by Molina-Rubio et al. (2010) successfully used a TA-XT2 Texture Analyzer with a 2.54 cm diameter acrylic cylinder, traveling at 2mm/s, and traveled 5mm into the sample surface to test the hardness of a semi-liquid syrup system.

3: Literature Review of Past Studies Applicable to A Semi-liquid Pulp/paste Candy System

Molina-Rubio et al. (2010) examined a system similar to pulp/paste candy. They examined the rheological and textural properties of a semi-liquid syrup by creating a model system composed of sucrose, high fructose corn syrup, dextran or carrageenan, citric acid, and water. This system was chosen to mimic the common composition of some Mexican candies (Molina-Rubio et al., 2010). Probe tests, the most common tests for measuring mechanical texture, were used to measure the textural properties of hardness and adhesiveness. Rheology was measured by using a stress control rheometer for low viscosity samples and a shear control rheometer with parallel plates for very viscous samples. Since low and high stirring rates were tested, Molina-Rubio et al. (2010) chose to examine turbidity as well. All measurements were repeated three times. The results showed the gum concentration was too low to modify the viscosity of the samples. Stirring velocity and acid concentration also did not have a significant effect on viscosity. The two factors that influenced the viscosity of the samples were gum type (whether dextran or carrageenan was used) and sugar concentration. Molina-Rubio et al. (2010) proposed that the viscosity differences were due to differences in the water availability between samples. High sucrose levels preferentially bound the water and prevented the gums from becoming hydrated, thus, limiting an increase in viscosity. Gum type and sugar concentration were also the only two factors that caused significant differences in hardness and adhesiveness. Gum type was the only variable that caused significant differences in turbidity. For all rheological and textural tests, the carrageenan samples had a higher mean value compared to the dextran samples. This means that if product developers choose carrageenan for their syrup, they can use less and potentially save money. Gum type was the only variable to exhibit significant differences in turbidity, with carrageenan exhibiting less turbidity than dextran. The data from this study suggest that the sugar concentration and the type of gum used can modify the textural and viscosity properties of confectionery semi-liquid syrups.

Quintas et al. (2006) examined supersaturated, 70-85% (w/w) sucrose solutions and measured their rheological behavior using a controlled stress rheometer. Since there was crystal growth in the metastable samples, a secondary creep experiment was performed under a constant stress of 5 Pa. Sucrose solutions tend to behave as Newtonian fluids and the results from this study showed this. The relationship between temperature and viscosity was also investigated and supersaturated sucrose solutions between 69.97-85.21 w/w % were studied over a temperature range of 0-90°C. The results from this experiment were then applied to two popular models, the Arrhenius model and the WLF model. In 1937, Eyring and Hirschefelder used the Arrhenius equation to predict the movement of molecules in the liquid state and it tends to work well for food samples that are not near the glass transition point; however, substances near the glass transition point, T_g, tend to deviate from this model (Quintas et al., 2006). A second model developed by Williams, Landel, and Ferry, known as the WLF equation, helps to explain behavior around the T_g of a substance (Quintas et al., 2006). Quintas et al. (2006) found that the supersaturated sucrose solutions were better represented by the WLF equation and the results tended to have less error if they used 0°C as the reference temperature in the equation.

When gums are added to samples, the rheology of the samples tends to follow the WLF equation (Kasapis et al., 2004; Rcondo, 2006). Kasapis et al. (2004) looked at the addition of gelatin, gellan, pectin, galactomannans, carrageenan, and agarose to either corn syrup (agarose, carrageenan) or sucrose solutions (galactomannans, gellan, gelatin, and pectin) and applied the free volume/WLF approach using 0°C as the reference temperature. By applying the free volume theory this approach used the irregularities in molecular packing caused by the polymers in the mixture to generate a shift factor in the WLF equation. This was then used to explain the temperature dependence of the rheology of the solutions during the glass transition area. When Kasapis et al. (2004) looked at potential gelatin replacements for ice cream and confections, they showed that although the free volume/WLF approach was a valid, predictive method, the polysaccharides exhibited different characteristics from gelatin. The polysaccharides exhibited synthetic polymer tendencies in supersaturated sugar solutions not observed in gelatin, which promoted chain association and made for a more rubbery gel. Tang et al. (2001) showed that sucrose and gellan concentrations had an impact on gel strength, but fructose concentration had no impact on gel strength. This is similar to what Papagerorgiou et al. (1994) found. Tang et al. (2001) looked at both sucrose and fructose in solutions of 0-35% w/w in the formation of gels made with gellan gum. Their study used compression tests to look at gel strength and visible light absorption to examine gel clarity. The gel strength of sucrose was tested by compressing the samples between two parallel plates at a constant speed until they failed. The gels with higher sucrose concentrations tended to have a higher gelling temperature. Tang et al. (2001) theorized that this was due to the ability of sucrose to pack into, and stabilize, the gellan double helix chains. Similar results were found with a high-solute corn syrup-sucrose-gellan mixture (Papageorgiou et al., 1994). Tang et al. (2001) found that the cooling method had an impact on gel strength and clarity. More rapid cooling methods created a weaker, clearer gel, independent of the amount of sucrose or fructose added. In an unsaturated system of sucrose and fructose, less absorbance was indicated at 490nm and, thus, indicated a clearer gel than what was observed in samples without the sugars.

Altay and Gunasekaran (2012) investigated a different hydrocolloid combination, gelatin and xanthan, and analyzed the rheology of the resulting gel mixtures by using the WLF equation. In addition to the WLF approach, they used the principle of time-temperature superposition (TTS), which is commonly used with synthetic polymers. In order that this principle may be applied, the shapes of the adjacent curves must match exactly, the same shift factor must be used for all functions, and the WLF equation can be used to show that the temperature is consistent with the shift factor. Roos and Karel (1993) stated that during a food product's shelf life, the changes in moisture content and temperature that the product is subjected can decrease the stability of some glass-like compounds by increasing the temperature difference (Altay & Gunasekaran, 2012). This approach builds on the previous approaches of Quintas et al. (2006) and Kasapis et al. (2004). The combinations of gelatin and xanthan gum were prepared at two moisture contents (20% and 25%) and three different gelatin-to-xanthan gum ratios (5:0, 9:1, and 4:1), and three corn syrup-to-sugar ratios for each moisture content. All samples were run in duplicate. To determine the dependance of viscoelastic behavior on temperature and time, a controlled-stress rheometer was used (Altay & Gunasekaran, 2012). The data showed that the TTS method could be employed because all of the conditions were met within the experimentation temperature range. The successful application of TTS indicated that the cooling process did not promote morphological changes in the samples. Altay and Gunasekaran showed that the polysaccharide network formation accelarated as the gelatin levels decreased, which was expected. In general, when the concentration of gum/gelatin in mixture was higher, the network became stronger and the glass transition point temperature increased. Even at high temperatures

(60°C) the combination of gelatin and xanthan gum maintained an elastic tendency. This was a surprise to the researchers, since gelatin's rubbery characteristic is known to degrade at higher temperatures. However, samples with only gelatin experienced a faster collapse of their free volume than samples with xanthan gum or a combination of gelatin and xanthan. Altay and Gunasekaran (2012) cautioned that the concentrations of xanthan gum were rather low and with greater concentrations of gums the TTS method has not been proven.

The two common modes of failure in paste candy are due to crystallization and inversion. Crystallization of a sugar matrix is affected by additives, water content, and temperature (Levenson & Hartel, 2009). Sucrose nucleation is the foundation for making sucrose fondant candies and much research has been devoted to this area. Since one of the forms of failure in pulp/paste candy is crystallization, research on fondants might help to identify possible culprits for what causes pulp/paste type candy to crystallize. Levenson and Hartel (2009) examined how different dextrose equivalents in corn syrup affected crystallization. They showed that nucleation rates increased as temperature increased, but the type of corn syrup used did not play a large role in crystallization unlike what was observed in another study by Tjuradi and Hartel (1995).

Acid inversion is also a mode of failure in pulp/paste candies. The general reaction is $C_{12}H_{22}O_{11}$ (sucrose) + H_2O (water) + H^+ (from an acid)$\rightarrow$ $C_6H_{12}O_6$ (glucose) + $C_6H_{12}O_6$ (fructose) + H^+. This is especially problematic, since many of the pulp/paste candies are sold in tropical countries. The acid splits the sucrose into the more hygroscopic glucose and fructose, resulting in cold flow (Jarrett, 2012). Shalaev et al. (2000) examined the factors that affect the acid-catalyzed inversion of amorphous sucrose in the presence of citric acid with less than 0.1% residual water over a two-week period. They measured the glass transition temperatures of the samples, the residual moisture, and the amount of inversion. At 50°C, the sucrose underwent significant inversion at a 1:10 ratio of citric acid to sucrose and pH of 2.43. Shalaev et al. (2000) showed that even with much less moisture than what is typically found in pulp/paste candy, due to the fairly high acid content and low pH of 2-3 in pulp/paste candy, acid inversion will likely occur and produce reducing sugars.

While much work has been done, using both DMA and mechanical texture studies, to evaluate the rheological properties of sucrose and the impact of the syrup type in low moisture/hard candy systems, no definitive study has focused on pulp/paste candy. Although Molina-Rubio et al. (2010) studied the concentration of sucrose in a semi-liquid syrup or pulp/paste candy-type system, they did not examine whether the particle size or morphology of the sugar had an impact on rheology and texture of the model system. The polysaccharides examined in Molina-Rubio et al. (2010) were limited to dextrans and carrageenan, but as Kasapis et al. (2004), Tang et al. (2001), Papageorgiou et al. (1994), and Altay & Gunasekaran (2011) showed other gums that are typically used in pulp/paste candy can affect the rheological properties of sucrose and corn syrup systems. The type of corn syrup was also not evaluated in the Molina-Rubio et al. (2010) system, but based upon other studies, such as Tjuradi & Hartel (1995) and Levenson & Hartel (2009) the type of syrup might have an effect on the crystallization of fondants. Since the results of studies on fondant candy have been inconsistent, a study that examines the effects on pulp/paste type candy would be beneficial to see if corn syup type has an effect on the rheology and texture properties of that system. Based on the findings of the previous work, future avenues for research can modify the semi-liquid syrup system proposed in Molina-Rubio et al. (2010) to make it more focused on the ingredients and processing used in paste/pulp candies. This future research should focus on the particle size and morphology of the sugar used, the corn syrup type used, and the type of gum used. The research hypothesis for such a project would be that the ingredient factors of the model pulp system (particle size of sucrose, gum type, and corn syrup type) will all impact the rheology and texture of the modified semi-liquid syrup model system, and by extension, pulp/paste candy.

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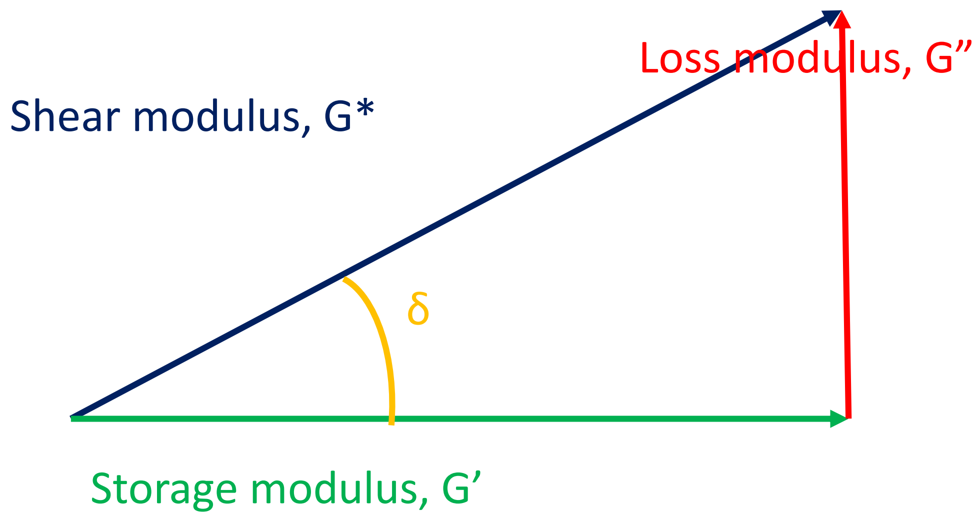

Figure 1.1 Relationship of Moduli

Chapter 2 - Viscosity Studies on a Semi-liquid Model System for Use On Pulp/paste Candy

1: Introduction

Paste/pulp candy is generally described as a soft fluid candy that may or may not have real fruit with particles in a colloidal suspension that is squeezed out of a package. In 2001, $166.6 million worth of candy was imported into the United States from Mexico and much of the candy is spicy and appeals to the growing Latino population's tastes (Zúñiga, 2002). The complex nature of pulp/paste candy brings unique challenges to product developers when trying to determine what the driving factors are for shelf life failure, typically caused by crystallization or inversion. Both of these types of failures create rheological changes to the candy and cause issues with leakage or an inability to squeeze the candy from the package. There has been little research completed on the formulation and processing of this type of candy. Therefore, since pulp/paste candy is a sugar based colloidal suspension, recent research about the rheological properties of sugar syrups, gum networks, and sucrose crystallization can provide insight into creation of a model system for this complex candy, since it shares all of these aspects.

Though Molina-Rubio et al. (2010) studied the concentration of sucrose in a semi-liquid syrup or pulp/paste candy-type system, they did not examine whether the particle size or morphology of the sugar had an impact on the rheology of the model system. Although it was hypothesized that particle size might make a difference because if the sugar particles are smaller there would be more surface area for the liquid portion to coat on a w/w basis. The same authors also hypothesized the type of gums used would impact rheology in their model system. The polysaccharides examined by Molina-Rubio et al. (2010) were limited to dextrans and carrageenan, but as Kasapis et al. (2004), Tang et al. (2001), Papageorgiou et al. (1994), and

Altay & Gunasekaran (2011) showed other gums typically used in pulp/paste candy can affect the rheological properties of sucrose and corn syrup systems. The syrup type was also not evaluated in the Molina-Rubio et al. (2010) system, but based upon other studies, such as Tjuradi & Hartel (1995) and Levenson & Hartel (2009) the type of syrup might have an effect on the rheological properties of the system. Since inversion in pulp candy can occur during processing and over the shelf life the addition of invert syrup to the model system would be beneficial. This research focused on the development of a model system, expanding on the one described by Molina-Rubio et al. (2010) by modifying the ingredient components, and using the model system to determine the impact of the type and amount of key ingredients on the viscoelastic properties of the system by using dynamic mechanical analysis (DMA). This broad study also examined the interaction of the key components of the model system to impact formulations for improved shelf life of pulp paste candy. It was the objective of this study to show that the key factors of particle size of sucrose, gum type, and syrup type will impact the rheology of the pulp/paste candy semi-liquid model system created. A custom mixture, D-optimal design was created since it could use both categorical and continuous factors (JMP, 2007).

2: Materials and Methods

2.1 Ingredients

The Amerfond® crystallized fondant sugar (120 µm mean grown-to-size sugar), #11 Nulomoline® partial invert syrup, and 6x Confectioners' sugar (a 25 µm mean pulverized-to size sugar) were obtained from Domino Foods, Inc. (Yonkers, NY). The Clearsweet® 43/43 Corn Syrup used was from Cargill, Inc. (Minneapolis, MN). The Kelcogel F® (low acyl gellan gum) and Keltrol® (xanthan) gum were provided by CP Kelco (Atlanta, GA) and all water was deionized. All ingredients were donated and the same batches/lots were used in all treatments.

2.2 Experimental Design

A Design of Experiments (DOE) was devised using JMP 12 (from SAS Institute, Inc.) custom design tool since there were both categorical (gum type and sugar type) and continuous factors (levels of gum, invert syrup, corn syrup, and water). A custom mixture, D-optimal design of experiments was created since it allowed for the use of both categorical and continuous factors and had built in replicates (JMP, 2007). D-optimal designs also allow for the use of multiple levels for categorical factors and are used for screening experiments (Jones, Lin, & Nachtsheim, 2008). This custom mixture design type also minimizes material usage, since the screening can be augmented and done sequentially, thus, honing in on the important factors and minimizing material waste on non-essential factors. JMP 12 was chosen since it is a leader in creating custom mixture D-optimal designs.

Figure 2.1 shows a ternary plot developed in JMP 12 with the initial design chosen for the semi-liquid syrup/pulp candy model. Treatment codes for each sample were created, assigning an alphanumeric level for each of six potential ingredient levels (ex. treatment 1gs 1cs 0is 1g 5w used level 1 of grown sugar, level 1 of corn syrup, no invert syrup, level 1 of gellan gum, and level 5 of water), see Table 2.1. A sample number was assigned by JMP indicating the order that the treatments were run for analysis. The initial design had 17 treatments, 18-22 were then added sequentially to increase the focus of the viscosity testing. Limitations on the minimum amount of water were introduced, since it was determined with initial testing that some water was needed to hydrate the gums. As part of the design there was one other linear constraint, formulations with less than fifty percent sugar were not examined since the sugar would fully dissolve in the matrix (Mageen, Kristott, & Jones, 1991).

2.3 Sample Preparation Methods

For each run identified in the design (Table 2.2), a 500g sample was prepared. Each treatment was prepared by using five grams of water (subtracted from the total amount) to hydrate the gum. After mixing and leaving to hydrate for at least 30 min, the hydrated gum was then added to a stainless steel pot containing the corn syrup and/or invert syrup with the additional water (if any) added. This pot was then heated to 75°C, measured by using a temperature probe (Cole Parmer, type K, 10” stainless steel) and thermometer (Fluke 52II), then removed from heat. Sifted pulverized sugar or sifted grown sugar was then added and mixed by hand. The soluble solids were measured by using a hand refractometer (ATAGO Master-100H), similar to the Molina-Rubio et al. (2010) methodology. However, since some treatments did not produce a clear refractometer reading, such as the one shown in Figure 2.2 it was decided to use calculated total solids (without including the solids from the gum, since that would be hydrated in the treatment) in the analysis. The calculated percent total solids no gum (%TS) for each treatment is determined by multiplying the percent of each component by the solids of that component. The following equation was applied, %TS = (% sugar) 0.99 + (% invert syrup) 0.757 + (% corn syrup) 0.81

The sample was then stored in a glass Mason jar at ambient conditions until ready to read on the AR-G2 rheometer (TA Instruments) (Figure 2.3).

2.4 Viscosity Testing

Parallel plate viscosity testing was performed on a stress controlled rheometer (TA Instrument, AR-G2) for all treatments. A serrated 40mm spindle (part. No. 513400.905, plate AL

ST 40mm, serial no. 992164) and Peltier plate was used. The data was then collected with the TRIOS software from TA Instruments. Before the treatments were run the rheometer was calibrated and oscillatory mapping was done at the frequency of 1.0 Hz. and a range of $1.0e^{-3}$ to 0.01 rad. displacement.

After calibration, an amplitude sweep or strain ramp was done to identify the LVR used for frequency sweep testing. The run parameters for the amplitude sweep were to equilibrate at 25°C, then sweep from 0.01% to 10% strain at a frequency of 1.0Hz, with 5 points taken per decade. The generated Lissajous/torque v. displacement curve from the TRIOS software was used to pick a stress point in the LVR for each treatment. Points were chosen from areas within the LVR that generated a smooth curve.

A frequency sweep was conducted within the LVR at 25°C, between the angular frequency range of 0.1 to 100rad/s. This sweep was then conducted two more times with all treatments held at 25°C during the testing. This setup and software was used because it is typically used in industry to characterize rheology.

2.5 Viscosity Analysis

The η^* slope for the first repetition was for further regression analysis since, using the Cox-Merz rule (Rao, 2007), it is synonymous to the slope of shear viscosity. A simple linear regression line was fitted to all of the complex viscosity vs. frequency plots generated from the treatments using TRIOS. Statistics were calculated using JMP 12. ANOVA was used to identify any significant differences in the slope of the first repetition of the complex viscosity vs. frequency plots as well as differences and interactions between model system ingredients and the complex viscosity at 0.1rad/s for the first repetition of the frequency sweep. Complex viscosity at 0.1 rad/s and %TS were compared using oneway ANOVA with Tukey HSD.

Relationships between G' and G" were also analyzed using tan (δ) vs. frequency plots generated in TRIOS. The raw data for tan (δ) was scrubbed to remove zeroing errors, with negative values changed to 0.0000001(See Appendix A for raw data and B for scrubbed data). Comparisons of tan (δ) curves based upon treatment %TS were done to characterize the impact of total solids on the solid/liquid behavior of the treatment across various frequencies. Minimum tan (δ) and %TS were also compared using oneway ANOVA with Tukey HSD.

3: Results and Discussion

3.1 Overall Observations on Treatments

Figure 2.3 illustrates the different clarities and some of the visual differences between treatments. Treatments 1gs 1cs 0is 1g 5w and 1gs 1cs 0is 3x 5w (Samples 2 and 15) appear to visually have very little, if any, particulates suspended in the samples. Treatment 4ps 2cs 0is 1x 1w (Sample 16) was not visually homogenous and did not visually have enough liquid to fully disperse the solid sugar particles. Prepared treatments 4ps 2cs 0is 1x 1w, 4ps 1cs 2is 1x 1w, 4ps 1cs 1is 1x 2w (samples 16, 19, and 22) were too thick and crumbly to test the viscosity. It is likely that these treatments did not contain enough of a liquid to fully coat or suspend the sugar particles. Prepared treatments 1gs 1cs 0is 1g 5w, 1ps 1cs 0is 3g 5w, 1gs 1cs 0is 3x 5w, 1ps 1cs0is, 1x 5w (samples 2, 9, 15, and 17) were very thin and almost water-like in consistency (see Figure 2.3). This observed visual range of consistencies indicated that the desired breadth of experimental design was accomplished prior to viscosity analysis. Overall, this study showed that measured °Bx (using a hand held refractometer) was still a good predictor of solids with an adjusted R^2 value of 0.96 (Table 2.3), but was not as accurate as using the calculated %TS in predicting the total solids from corn syrup, invert syrup, and sugar. This is because the refractometer measures °Bx and different types of sugars (glucose, fructose, polysaccharides) register different °Bx for the same solids level on the hand held refractometer since the different sugars refract differently.

3.2 Results and Discussion of Amplitude Sweep

The design of experiments succeeded in providing a varied array of data. The majority of the treatments had a well-defined and large LVR (one exemplified in Figure 2.4) while others, such as seen in Figure 2.5, had a very small one (if any). Treatments in the mid-total solids-not gum range of 76-82% (samples 1, 3, 4, 6, 10, 11, and 13) had very large LVRs and did reach critical strain or have a crossover point. Treatments with lower %TS (samples 2, 9, & 15) generally had fairly large LVRs, but exhibited some material breakdown towards the end of the plot, see Figure 2.4.

3.3 Overall Results and Discussion of Frequency Sweep

Due to the broad range of sample viscosities, not every treatment in the design had a frequency sweep performed. For some of the treatments (treatments with >92% TS) the stress identified in the LVR was outside of the range of the instrument and could not be run, (Figure 2.5). Figures 2.6-2.8 exemplify the different types of rheological behavior found with the frequency sweeps. Figure 2.6 shows a representative example of a low %TS treatment with a less than 1-log decrease in η^* with increase in frequency and with a continuously decreasing tan (δ) with increase in frequency. Figure 2.7 shows a representative example of a high %TS treatment with a 2-log decrease in η^* with increase in frequency and a decreasing, then increasing tan (δ) with increase in frequency. For the majority of the treatments, the three repetitions were overlaid, indicating that the sample structure did not change much throughout the three repetitions of the frequency sweep (Figure 2.7). Figure 2.8 exemplifies some of the most structure breakdown that occurred between repetitions (the triangles are the 1st repetition, the squares are the 2nd repetition, and the circles are the third repetition of the frequency sweep on the treatment).

3.4 Analysis of Complex Viscosity (η*) versus Frequency

All of the treatments, except 1ps 1cs 0is 3g 5w, exhibited a decrease in complex viscosity (η*) with an increase in frequency (Figure 2.9). A decrease in η* with an increase in frequency is typical of viscoelastic materials with biopolymer dispersions (Rao, 2007). The treatments with lower (A) %TS seem to have less negative η* slopes compared to medium level (B) and high level (C) %TS (Figure 2.9), indicating that the increase in frequency oscillation might be causing the particles suspended to line up and create resistance to shear. Figure 2.9 also shows the complex viscosity was much higher overall for treatments with high %TS. For high % TS treatments (C), the complex viscosity goes from 1.0×10^7 Pa*s at low frequencies down to about 100 Pa*s at high frequencies. This complex viscosity was much higher than the treatments at low % TS treatments (A) that range from around 0.1 Pa*s to 10 Pa*s, indicating that the treatments which have higher %TS are much more viscous than the low %TS treatments. High, med, and low %TS treatment levels all had different means (high to low %TS: $p < 0.001$, high to med %TS: $p = 0.0078$, med to low %TS: $p = 0.109$) and therefore, viscosities (Figure 2.10). This shows that at higher solids levels it is much harder to squeeze pulp/paste candy from the package. It also agrees with the findings from Perry and Green (1998) and Quintas et al. (2005) that, in a simple sucrose-water system, viscosity also increases with increased sucrose concentration. Treatment 3ps 1cs 3is 3g 1w, Figure 2.8, uniquely had G' > G", which showed that it was more solid vs. the majority of the samples measured, which had G" > G', showing that they are more fluid. This was expected for treatments 1gs 1cs 0is 1g 5w, 1ps 1cs 0is 3g 5w, 1gs 1cs 0is 3x 5w, 1ps 1cs 0is 1x 5w (samples 2, 9, 15, and 17) since they all have low levels of corn syrup and invert syrup. However, it was unexpected for the thicker, more dough-like samples. In Steffe (1996) it is theorized that this is caused by the presence of a yield stress in the matrix. If there was a yield stress in this model system, it could mimic the impact in pulp/paste type candy for the ease of both pumping candy into containers as well as squeezing candy out.

A stepwise technique was used to create an ANOVA model for the log transformation of η^* at the low oscillation rate of 0.1 rad/s. This showed that the interaction between invert syrup and water was significant, along with the main factor of gum level see Table 2.5. The type of gum used was just above the 95% confidence level ($p = 0.0597$). Cakebread (1970) found that when invert sugar was exchanged for a portion of other components at a constant total solids level in a system composed of sucrose, corn syrup solids, water, and invert sugar, the viscosity of the system was lower. These findings reaffirm the results from Cakebread (1970). Also, since %TS when broken out into levels was significant, the factor effects from the model generated might have been diluted due to the many factors included in the model.

Having a greater portion of invert syrup and water (B) in the system creates a lower predicted viscosity (Figure 2.11). At lower levels of water, having more invert syrup creates a predicted higher complex viscosity for treatments with gellan gum (A), Figure 2.11. This might be explained by the preference of fructose over sucrose to associate with water and the preference of sucrose over polysaccharides to associate with water since there is less steric hindrance (Rao, 2007). In this screening study, since the water available to hydrate the gellan gum was held constant as the level of gum varied, prior to the addition to the syrups, the competition of free water with the syrups could have resulted in the gum using some of the water from the invert syrup to hydrate since it was the ingredient with the next highest moisture level (76%). Treatment 3ps 1cs 3is 3g 1w exemplifies a treatment with a high complex viscosity

(Figure 2.9), a high invert syrup level, low water, and gellan gum. Since gellan gum is known to form gels in aqueous solutions at 20°C (Funami, et al., 2008) and that gellan networks are strengthened with sucrose (Papageorgiou et al., 1994) this treatment could be at the sol-gel interface and have a stronger gel network formed than treatments with lower %TS which have the gum too dispersed to form a network.. This is also suggested by the tan (δ) of treatment 3ps 1cs 3is 3g 1w (Figure 2.12) since at 0.1 rad/s it is at 1, the borderline between sols and gels (Funami, et al., 2008). With higher levels of water in treatments 1ps 1cs 0is 1g 5w and 1ps 1cs 0is 3g 5w, the surplus of water could have interrupted the network by dispersing the gum too far so that it became a sol, as shown in Figure 2.12 since the tan (δ) is much greater than 1. This network interruption contributed to the decrease in viscosity since with this and the use of more in the formulation over sugar resulted in an increase in small molecules. These small molecules could also be acting as plasticizers and contributing to the decrease in viscosity. Foegeding and Steiner (2002) theorized that in caramel, the increase of low molecular weight saccharides in syrups helped to act as plasticizers, similar to the other small molecules in the system (water) and these small molecules acted as diluents, decreasing the viscosity. This interaction could impact how easily the pulp/paste candy is squeezed out and formulation-wise it would be better to have more water and less invert syrup to yield a lower viscosity product (Figure 2.11). Since product viscosity is also important in processing pulp/paste type candy, future testing at processing temperatures should be done to gain insights if this impact of invert syrup still holds at higher processing temperatures. However, since this was a screening study with only one treatment, 3ps 1cs 3is 3g 1w, to support this theory it is possible that it could be a coincidence that higher invert syrup was part of the treatment with the least amount of water that allowed the formation of a gel. The model could have confounded the higher invert syrup level with the gel network formation and higher viscosity and the concept that invert syrup increased complex viscosity at high %TS could just be coincidental. Additional testing in this area should be done to confirm that this interaction between invert syrup and water was real.

The interaction between gum level and water showed that with more water in the system the predicted viscosity at 0.1 rad/s shifts higher (Figure 2.11). This might be because at lower levels of water, there might not have been enough water in the system to fully hydrate the gum and allow it to be fully functional. Molina-Rubio et al. (2010) suggests that for carrageenan and dextran 0.1-0.3% gum is too low to detect a difference in viscosity, however, in this modified semi-liquid model system the interaction between gum and water at only 0.0005-0.001% gum suggests otherwise. The borderline gum type significance showed that gellan gum at 0.1rad/s had a higher complex viscosity than xanthan gum (Table 2.5; Figure 2.11). This could indicate that the low acyl gellan gum has created a gel structure. Since xanthan gum cannot create a gel, the gel network formed by gellan might have contributed to the increased viscosity difference between gum types. Also, since low acyl-gellan gels are known to be brittle, testing the more flowable high acyl-gellan in future research might be helpful to further explore differences in gum type (Tang et al., 2001). This would expand on the existing knowledge base for pulp/paste candy and could identify the optimal gum for pulp/paste candy formulation that builds a network to suspend sugar particles, but is still flowable and easily squeezed from the package.

Since Molina-Rubio et al. found that soluble solids had a significant effect on viscosity, this study showed that it might indeed have been the inversion of sucrose that occurred in Molina-Rubio et al. (2010) which contributed to this effect. Also, since %TS was significant for complex viscosity when broken out into levels (Figure 2.10), the effect might have been diluted due to the many factors included in the model. This is due to sucrose having more power than the other factors. Since there were 2 types of sugar + 4 levels of sugar tested, there was more power to differentiate differences over, for example, the factor of corn syrup where there were just 4 levels tested.

The result that sugar level is significant, is in agreement with, and improved upon the study by Molina-Rubio et al. (2010) and their finding that °Bx is an important factor by examining corn syrup/invert syrup and sugar instead of just °Bx (or soluble solids). Although the model system had individual treatments that contained a lower sucrose level with higher invert syrup levels (treatments 1ps 1cs 4is 1g 3w, 3ps 1cs 3is 3g 1w, 2ps 2cs 2is 3x 1w), mimicking inversion, the overall ANOVA for the components looked across all treatments as a whole and not just those individual treatments. In the study by Molina-Rubio et al. (2010) it was likely the invert generated from the acid and sugar concentration that contributed to the significance of °Bx had a significant effect on their system. However, since the increase in invert syrup level can only definitively explain the use of invert/high fructose corn syrup in formulation examining the impact of the increase of invert syrup due to inversion deserves further research.

To further quantify the variation in viscosity of the samples the slope of the η^* vs. frequency plots was examined. Figure 2.9 indicates that the slope of the majority of the η^* vs. frequency plots are flat at low frequencies and then start to curve with higher frequencies (analogous to apparent viscosity vs. shear rate plots with the Cox-Merz rule) and this indicates Newtonian behavior, followed by the start of a power law region. This type of behavior is typical for gums, since it is thought that the dispersed biopolymer molecules rearrange due to an increase in shear. With little shear, there is little rearrangement of polymer chains and Newtonian flow is exhibited (slope of 0), but with higher shear the biopolymer chains undergo gradual rearrangement that results in power law behavior, which is typically shear thinning (negative slope) (Rao, 2007). Further examination on the slope of plots was then conducted. A simple linear regression was run on all of the η* vs. frequency plots. Generally, this regression line had an R^2 value of around 0.9 and was therefore a good fit for analysis. The slopes of the η* simple linear regression line for each sample repetition were then averaged to yield the average η* slope (Table 2.4). The regression analysis was performed for only the first of the three repetitions to eliminate sample breakdown (Table 2.4). The η* slope for the first repetition was then used for further regression analysis since, using the Cox-Merz rule (Rao, 2007), it is synonymous to the slope of shear viscosity. The treatment slopes ranged from -0.74 to 0.32, which were all right around 0, though this range could indicate some slight shear thickening/thinning with the extreme treatments. When creating the model using stepwise regression for complex viscosity slope, total solids was not included as a factor since it could be explained fully by the factors of sugar, corn syrup, and invert syrup. Upon analysis from JMP 12 of the η* slope, the model generated showed at a 95% confidence level that the effect of sugar impacts the slope of (complex) viscosity, but that the type of sugar (grown or powdered) does not. Invert syrup is just over the significance level of 0.05 ($p = 0.0528$) and if the level was changed to a 90% confidence level would be considered to have an impact on the slope of complex viscosity. The amount of corn syrup used does not have any impact. At a confidence level of 95%, the interactions between invert syrup-water, gum-water, and sugar type-water had an impact on the viscosity slope, but not for gum type or corn syrup amount (Table 2.6). Having more invert syrup and water in the system creates a more positive (complex) viscosity slope, showing by extension, that at higher frequencies the treatments have a higher viscosity (Table 2.6). This might be explained by the preference of fructose over sucrose to associate with water and the preference of sucrose over polysaccharides to associate with water since there is less steric hindrance (Rao, 2007). This interaction could impact pumping of product. Therefore, if pumping at low oscillation or squeezing the product out of the package at room temperature, it would be better to have more water and less invert syrup, but if pumping is done at higher frequencies, it would be better to have more invert syrup and less water to yield a lower viscosity product.

The interaction between gum level and water shows that with more water in the system the predicted viscosity slope shifts higher (Table 2.6). This might be because at lower levels of water, there might not have been enough water in the system to fully hydrate the gum and allow it to be fully functional. If this was the case, it shows how critical proper gum hydration is, since a developer might be able to use less gum if more water is available to hydrate. Although the gum was hydrated prior to the addition to the syrups to minimize competition for the free water, at the lower water formulation levels, there wasn't enough water included in the model to fully hydrate the gum. Molina-Rubio et al. (2010) suggests that for carrageenan and dextran 0.1-0.3% gum is too low to be tested, however, in this modified semi-liquid model system the interaction between gum and water at only 0.0005-0.001% xanthan and gellan gum suggests otherwise.

The result that sugar level is significant (Table 2.6), with increased sugar increasing the complex viscosity slope (and by extension viscosity) of the treatment, is in agreement with, and improved upon the study by Molina-Rubio et al. (2010) and their finding that °Bx is an important factor by examining corn syrup/invert syrup and sugar instead of just °Bx (or soluble solids). It was likely the invert generated from the acid and sugar concentration that contributed to the significance of °Bx had a significant effect on their system. However, this deserves further research, since citric acid inversion was not specifically examined in this study and since citric acid is typically an ingredient in pulp/paste type candy the levels used and processing conditions could impact the viscosity of the candy based upon this study. The identified sugar type-water interaction shows that the larger particle size (grown sugar) has a lower viscosity with more water than the smaller (powdered) sugar type. This difference in viscosity between the two types of sugar was likely because there was less surface area for the liquid phase to coat for the larger particle size sugar and that with increased water levels the minimum level of the liquid phase required to coat all of the particles was met, resulting in a decrease in viscosity. Although larger particles would make the candy flow out of the package easier making it easier to eat, sensory testing should be done to confirm that the 'grittier' paste is still acceptable.

3.5 Analysis of Tan (δ) versus Frequency

Tan (δ) was also analyzed to see if the treatments were becoming more liquid or solid over the increase in frequency. Nine of the treatments had a tan (δ) vs. frequency plot that moved from a high to low tan (δ) with increasing frequency (Table 2.7). Seven of the treatments had a lower tan (δ) at lower frequencies, then an increase at higher frequencies before leveling off, indicating that there was a disruption of the gel matrix. This behavior is exemplified in Figure 2.7. Up until this inflection point in the tan (δ) frequency sweep, the plot had a negative slope indicating that the treatment was becoming more solid-like due to the larger storage modulus, G'. However, once past the inflection point in Figure 2.7, tan (δ) increased and the sample became more liquid-like. Treatments 1ps 2cs 2is 1x 3w, 3ps 1cs 0is 1x 4w, 1gs 1cs 4is 3x 3w, 3gs 1cs 3is 1x 1w, 1gs 4cs 0is 3x 3w, 2ps 2cs 2is 3x 1w, 2gs 3cs 2is 1g 1w, 3ps 2cs 0is 3x 3w all had a positive slope overall. Figure 2.6 shows treatment 1ps 1 c 0is 1x 5w, sample 17 (a low %TS treatment), with a different trend in the tan delta vs. frequency plot—continuously decreasing with higher frequencies, thus exhibiting more solid like behavior with increasing frequency. The lower tan (δ) trend, with minimum tan (δ)s ranging from 1-3 at 1-3 rad/s found in treatments with 91.59-92.18% TS might be explained by the additional sugars increasing the strength of the internal bonds within the system, creating a more solid-like material at that low oscillatory rate. Rao (2007) has shown that with the increase of either sucrose or fructose in a starch dispersion, an increase of G' and lower G" resulted and theorized that this might be the cause. ANOVA of log [minimum tan (δ)] confirmed that with gellan gum and water there was an interaction (Table 2.8 and Figure 2.13). The log transformation of tan (δ) was chosen to normalize the residuals (Figure 2.14). The minimum point of the tan (δ) curve for each treatment was selected since it represents the most solid aspect of the plot. At this point, gum type, corn syrup amount, and invert syrup amount are all significant as main effects and also as interactions with water. Since corn syrup and invert syrup both are composed of about 20% moisture, with an increased addition of these syrups, it contributed to higher minimum tan (δ) or a more liquid like system, especially at lower water levels (Figure 2.13).

3.6 Analysis of Viscosity and Total Solids using Tan (δ)

It becomes apparent when the treatments are organized by %TS that the total solids percent seems to effect the viscosity, see Table 2.7. The treatments with 59% TS had a larger tan (δ) observed range (the difference between the maximum and minimum tan (δ) value measured in the tested frequency sweep) and a smaller minimum tan (δ) when gellan gum was used over xanthan gum. This is also true when the treatment contains more gum (Figure 2.12). Within the 59% TS treatments, there is a general trend of the tan (δ) for the two gellan treatments (indicated by triangles) behaving similarly and the two xanthan gum treatments (indicated by circles) behaving similarly, though this was not true with the system overall (Figure 2.12). While all 59% TS treatments appear to have a decreasing tan (δ), the gellan gum treatments seem to have a larger slope or decrease more with higher frequencies. This behavior, however, was not significant across all treatments.

When comparing the tan (δ) of the treatments with lower solids vs. the treatments with higher solids, it was found that at higher frequencies the behavior was opposite. Treatments with lower solids behaved more like solid materials at high frequencies and treatments with higher solids behave more like liquids at higher frequencies (Figure 2.12). Treatments in the mid-range (B) have the typical solids range for pulp candy (Figure 2.12). The plots are relatively flat, but are starting to show a curvature similar to the high %TS treatments. Figure 2.15 indicates that the means between treatments with 75-90%TS are not significantly different than treatments with >90%TS confirming that it is only treatments with <60%TS that have different solid-like characteristics (from the minimum tan (δ) based on %TS level).

Examples of near-ideal viscoelastic liquid behavior could be found in the treatments with 59.48 % TS (treatments 1gs 1cs 0is 1g 5w, 1ps 1cs 0is 3g 5w, 1gs 1cs 0is 3x 5w, 1ps 1cs 0is 1x 5w). This near-ideal viscoelastic liquid behavior of treatments at lower solids is generally the opposite of treatments with a high % TS (Figure 2.12). Examples of higher solids behavior can be found in treatments 3gs 1cs 3is 1x 1w & 2ps 2cs 2is 3x 1w (samples 12, 20). Although treatment 3ps 1cs 3is 3g 1w (sample 5) has higher solids, it behaves very similarly to a perfect viscoelastic material since tan (δ) decreases as the frequency increases throughout the observed frequency range. This is illustrated in Figure 2.8. This apparent anomaly is likely due to the higher gellan gum level along with the higher invert syrup used in this treatment, thus allowing the gum to more fully hydrate and exhibit the viscoelastic functionality.

At 59.48 % TS the two types of gums have similar curves, with a slight shift in tan (δ) for the ones that have a higher concentration. The samples made with xanthan gum had a much lower tan (δ) across most of the frequency range, whereas the gellan gum samples had a tan (δ) that dropped off sharply around 1-5 rad/s (G' increases and meets G" ; Appendix C, Figures C. 9,15,17). This indicates that the two xanthan samples behaved more like solids overall at 59.48% TS, whereas the two gellan treatments only behaved more like solids at higher frequencies. At the lower frequencies which would mimic eating conditions, it would be better to use the low acyl gellan gum, since it would be easier to squeeze out of the package at room temperature. This finding was supported by Altay & Gunasekaran (2012) where they found that at 60°C (vs. 25°C) a sucrose/corn syrup/gelatin solution had a larger G' than G" across a DMA frequency sweep when xanthan gum was increased, indicating agreement that xanthan gum might be contributing to solid-like behavior in the model system. The increasing solid-like characteristic at lower total solids percentage might be due to the fast deformation process occurring during the frequency sweep. Since with increasing frequencies, the relaxation time is kept constant. This relationship is common in the behavior of dispersions and is quantified with the dimensionless Deborah (De) number or De = (1/dynamic frequency)/relaxation time (Rao, 2007). With the higher solids treatments, there appears to be an inflection point around 5 rad/s where the tan (δ) changes from low to high. This behavior of a decreasing, then increasing tan (δ) reaffirms the findings of Kasapis et al. (2004), where they found this to be the case in a sugar/biopolymer mixture. They theorized that this behavior was explained in relationship to the mixture's T_g. They theorized the initial decrease in tan (δ) was due to predominant free molecular flow at low frequencies, with the increase of tan (δ) occurring when stable gel networks are formed at higher frequencies. This behavior does not appear in the lower % solids samples and might be indicative of the gel structure breaking down, since sucrose provides strength to some gel networks (Papagerorgiou et al., 1994). Although 59% total solids is lower than what is typically used for pulp type candy, this information is useful if trying to formulate a lower sugar pulp/paste candy option or a water based crème while still targeting the same viscosity for pumping the candy in manufacturing (though this would still need to be tested to see if it holds true at temperatures other than 25°C).

4: Conclusion of Viscosity Study

Since this was a study using a broad range of treatments, the DOE provided a wide array of treatment viscosities. ANOVA of the model generated showed that there were interactions ($p < 0.05$) between invert syrup-water, gum-water, and sugar type-water. At room temperatures, to facilitate easier squeezing/lower viscosity of pulp/paste candy it would be better to have more water and less invert syrup/high fructose corn syrup in the formulation. Also, having more water and a larger particle size of sugar helped to lower the viscosity of the system, though sensory testing should be done to verify that having a 'grittier' sugar would be acceptable. Sugar and gum levels as factors also had a statistically significant effect. Oneway ANOVA using Tukey HSD showed that high %TS, when broken into levels had a significant impact on complex viscosity at 0.1rad/s, with a much higher complex viscosity than the low %TS level. This shows that at higher solids levels it would be much harder to pour/squeeze pulp/paste candy from the package. The amount of corn syrup used does not appear to have an impact on the viscosity of the system. Examining the tan (δ) confirms along with the other tests for η^* that invert syrup and gum type are important factors that contribute to the viscosity of the semi-liquid syrup model system. Based upon the differences in gum type tested, low acyl-gellan gum would be more optimal than xanthan gum, but high acyl-gellan gum should be tested in the future since it has more flowable properties than the low acyl version. The significant impact of invert syrup and sugar on soluble solids/°Bx is in agreement with several studies (Molina-Rubio et al. (2010), Cakebread (1970), Perry and Green (1998), and Quintas et al. (2005)) and provides further clarity to the conclusion from Molina-Rubio et al. (2010) that °Bx is an important factor in the determination of viscosity in a pulp/paste semi-liquid syrup model system.

5: References for Chapter 2

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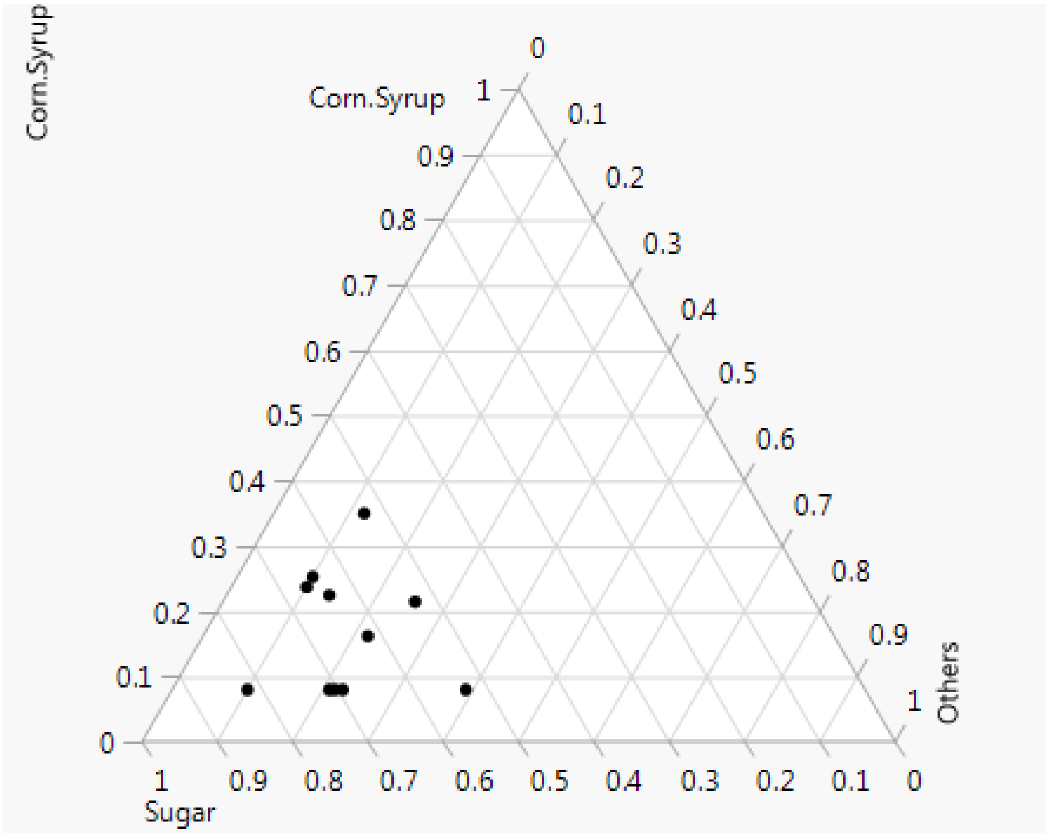


Figure 2.1 Ternary Plot of Initial Samples

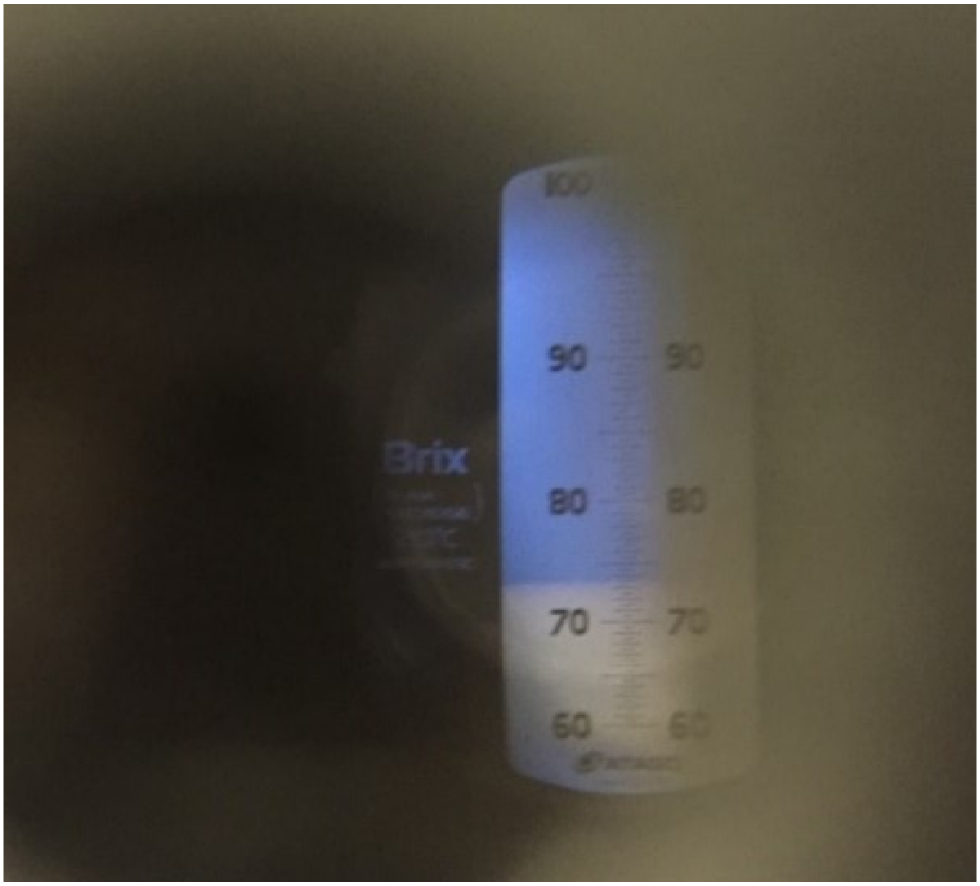


Figure 2.2 View from Refractometer

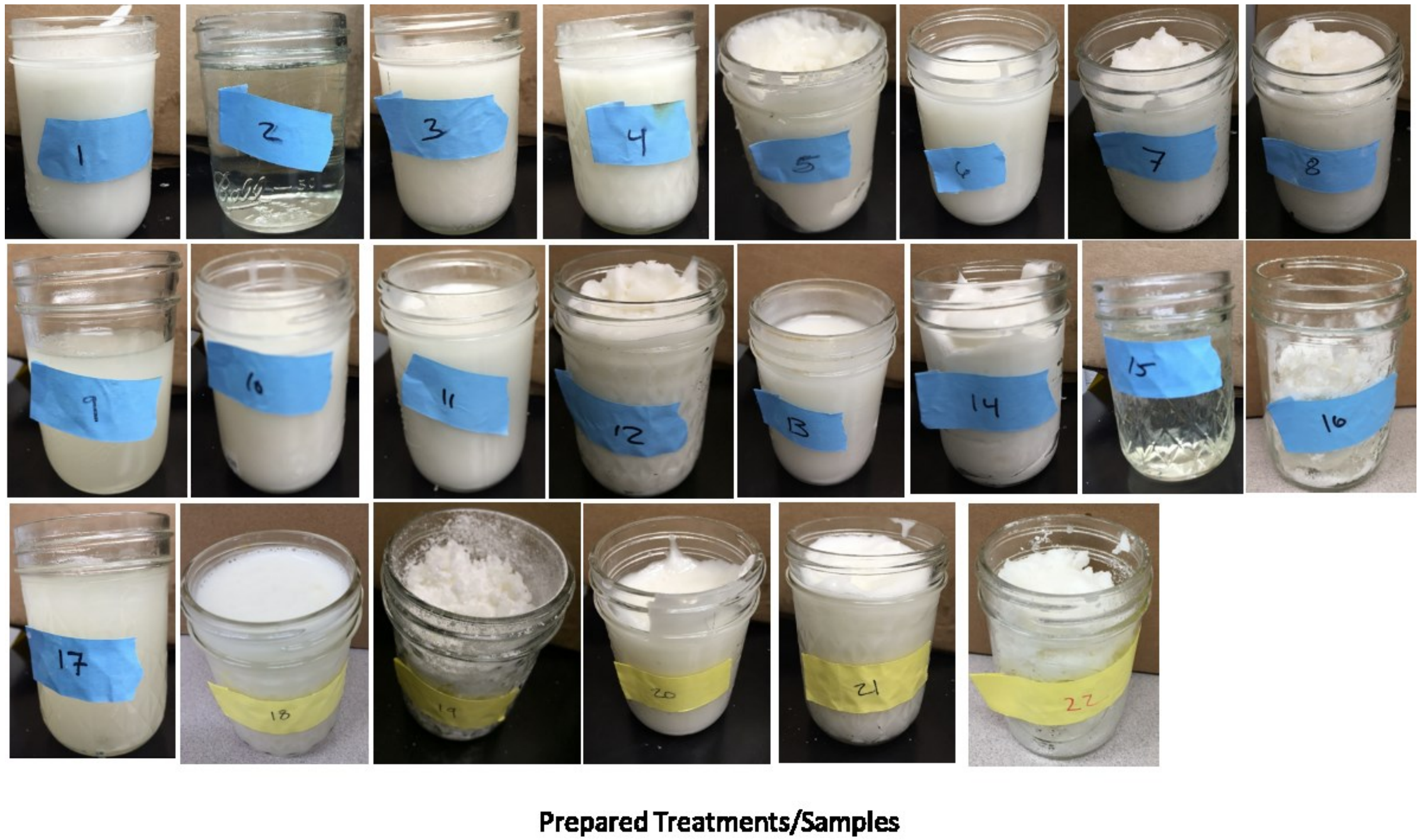


Figure 2.3 Prepared Treatments for Design of Experiments

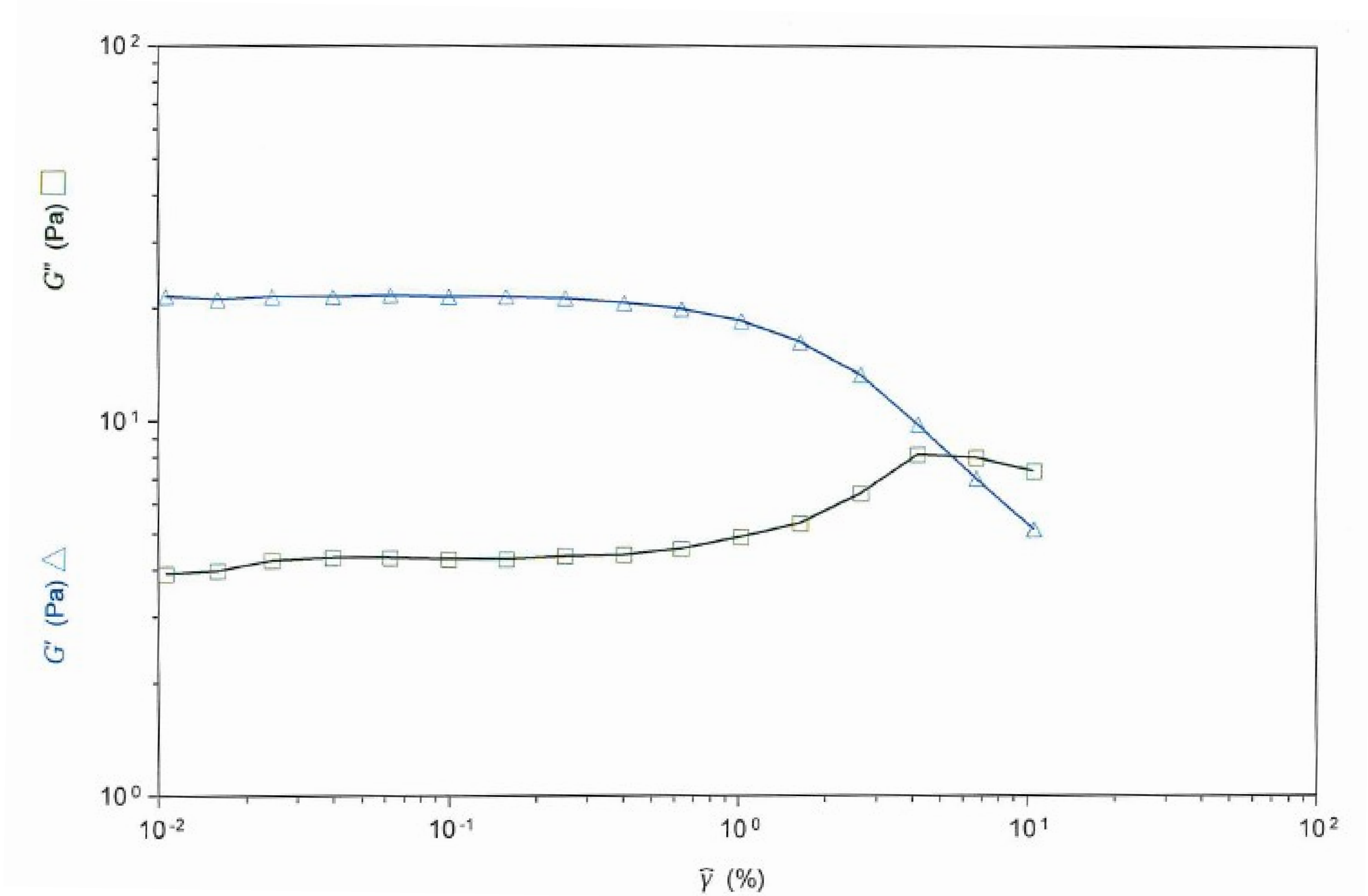


Figure 2.4 Amplitude Sweep for Treatment 1gs 1cs 0is 1g 5w (sample 2) Showing a LVR with a Critical Strain

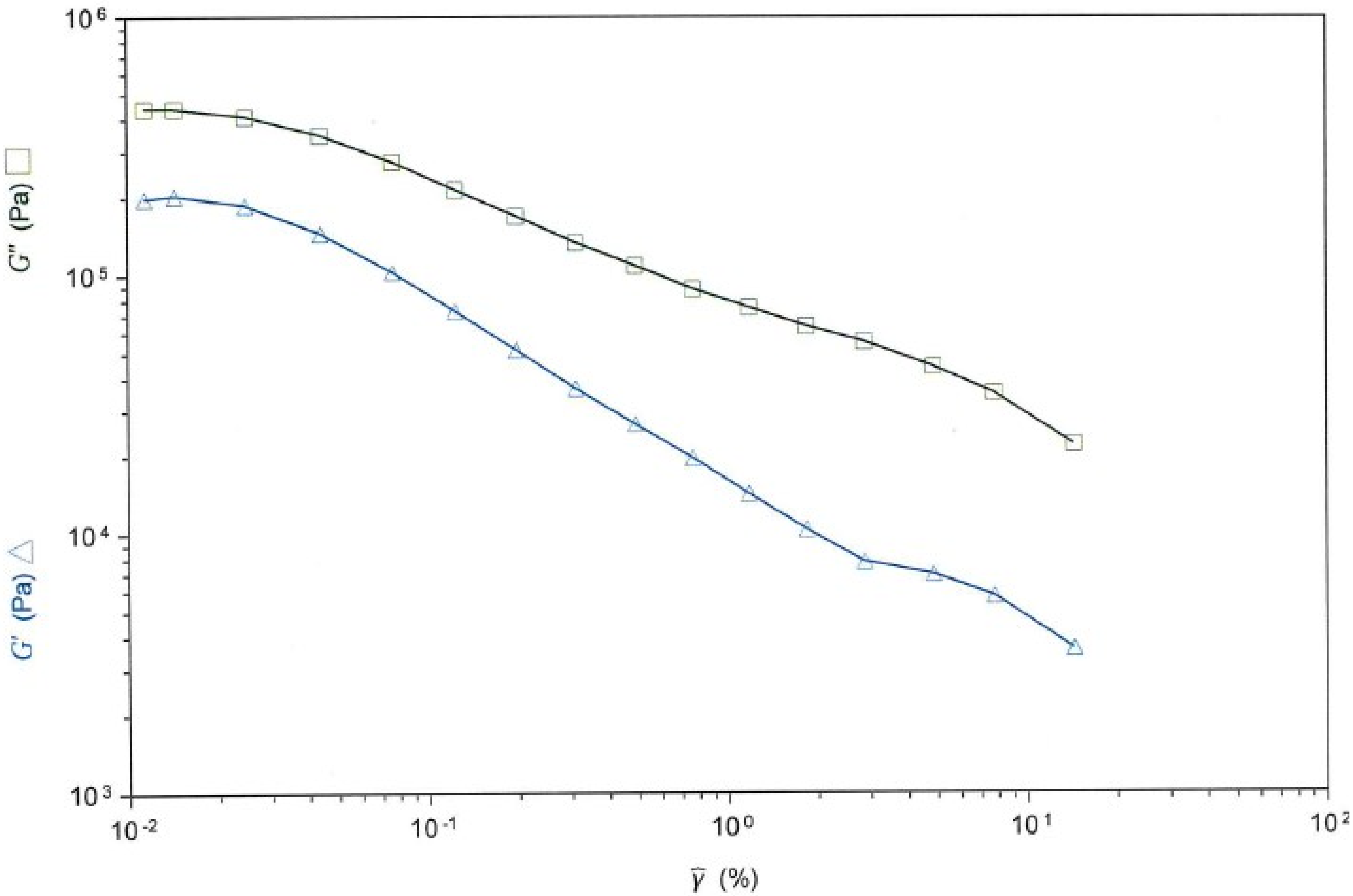


Figure 2.5 Amplitude Sweep for Treatment 3ps 3cs 0is 3x 1w Showing No LVR within Instrument Range

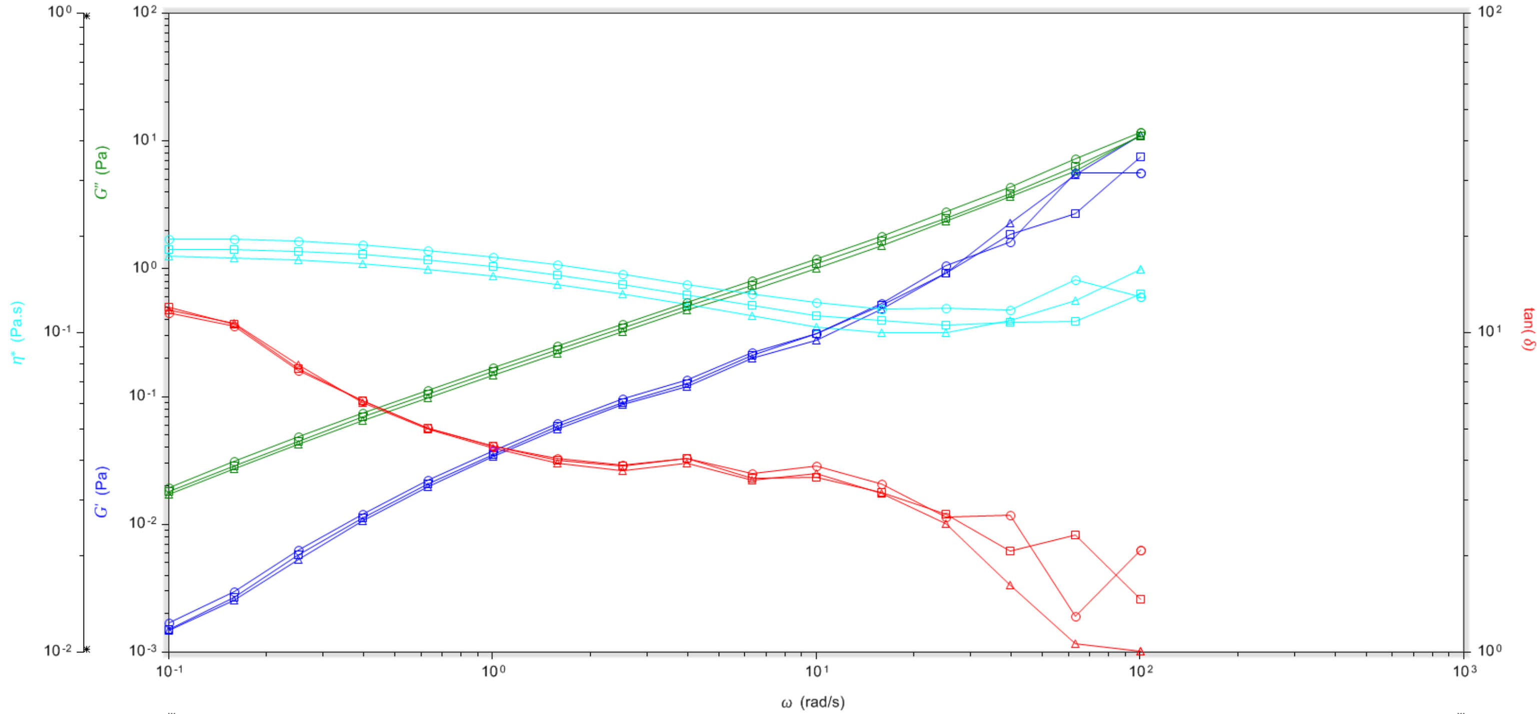


Figure 2.6 A Typical Frequency Sweep for Low %TS Treatments (Treatment 1ps 1cs 0is 1x 5w)

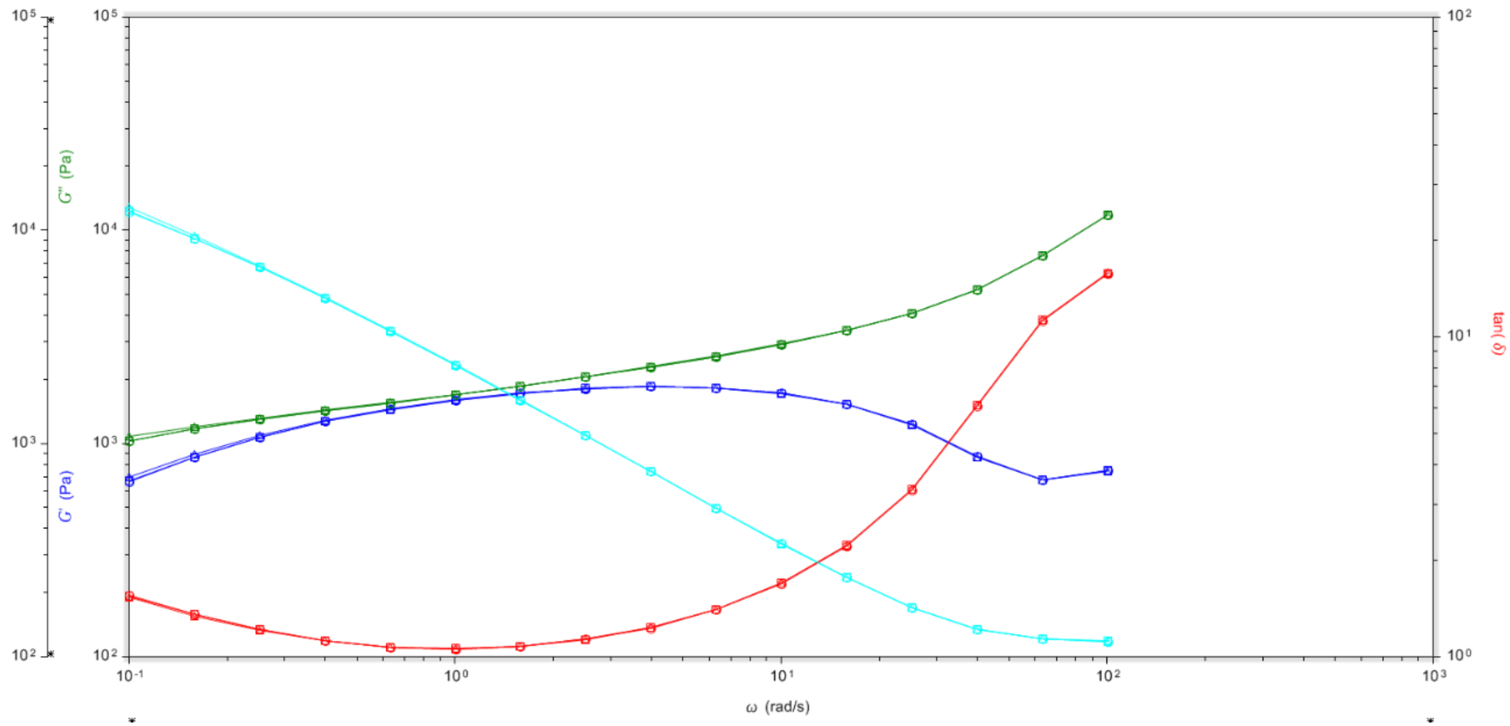


Figure 2.7 A Typical Frequency Sweep for High %TS Treatments (Treatment 3gs 1cs 3is 1x 1w)

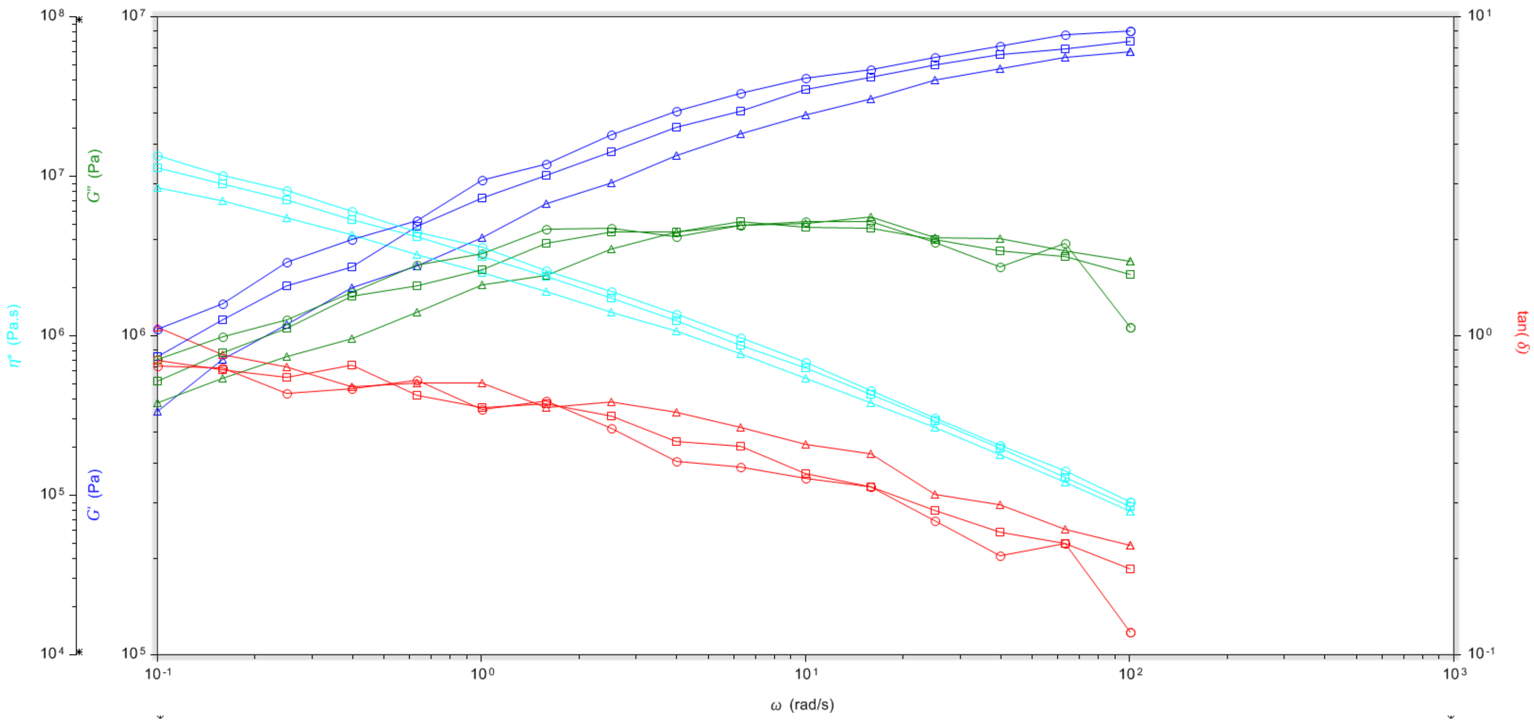
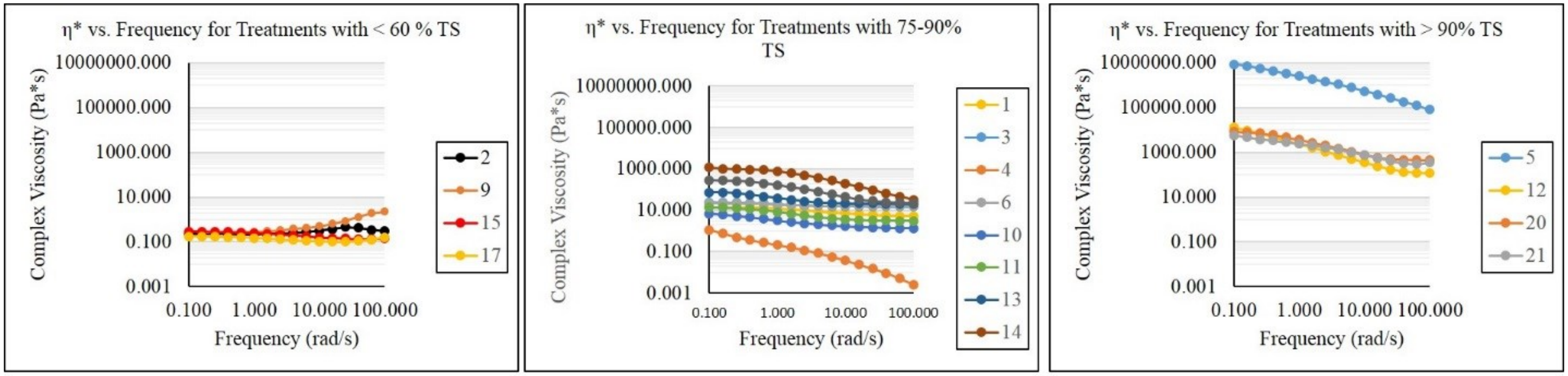


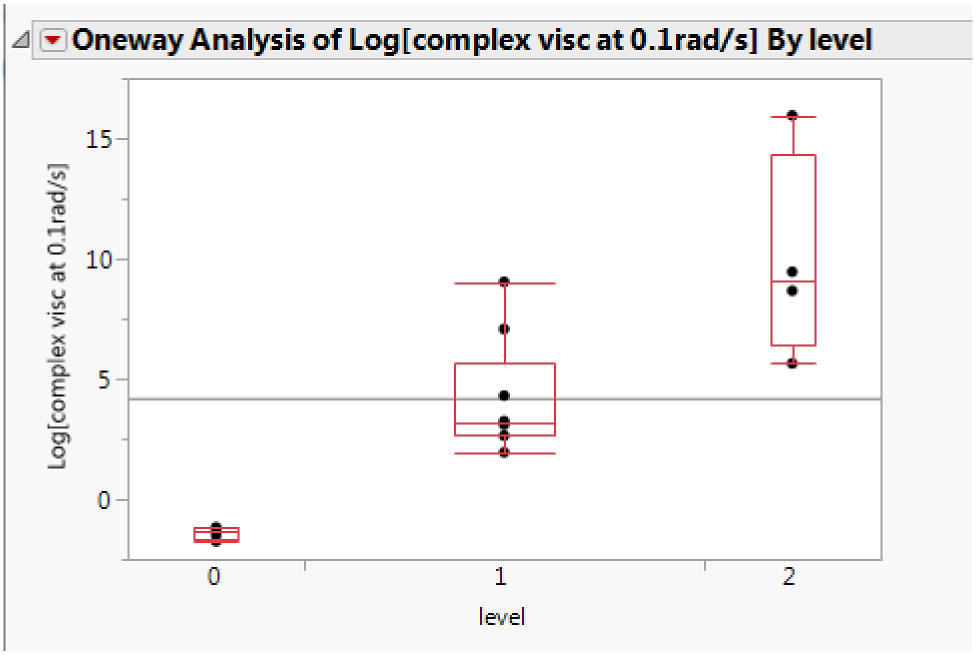
Figure 2.8 Frequency Sweep for Treatment 3ps 1cs 3is 3g 1w (sample 5) Showing Treatment Breakdown over Subsequent Frequency Sweep Repetitions



A B C

Comparison of η* as a function of frequency for Low (A), Medium (B), and High (C) % TS Levels

Figure 2.9 Comparison of Complex Viscosity as a Function of Frequency broken into %TS Levels



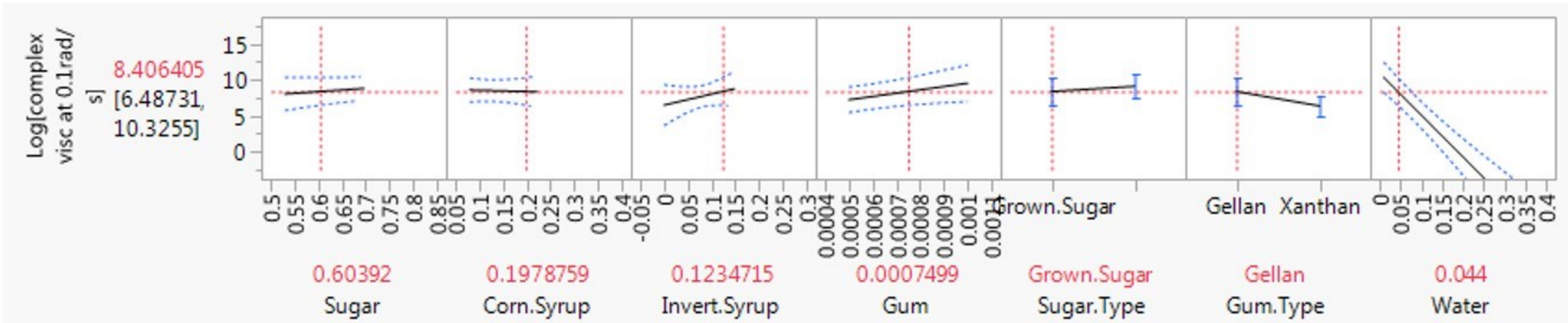
Connecting Letters Report

Level		Mean
2	A	9.929956
1	B	4.125225
0	C	-1.404442

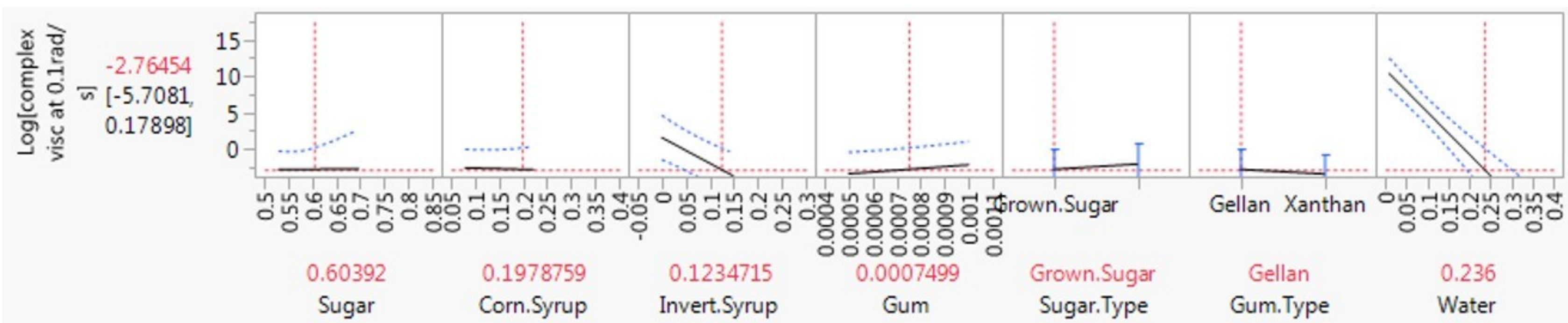
Levels not connected by same letter are significantly different.

Levels: 0 = < 60%TS, 1 = 75-90%TS, 2 = > 90% TS

Figure 2.10 Oneway ANOVA of log [η*] at 0.1 rad/s by %TS Level w/ Tukey HSD Comparison



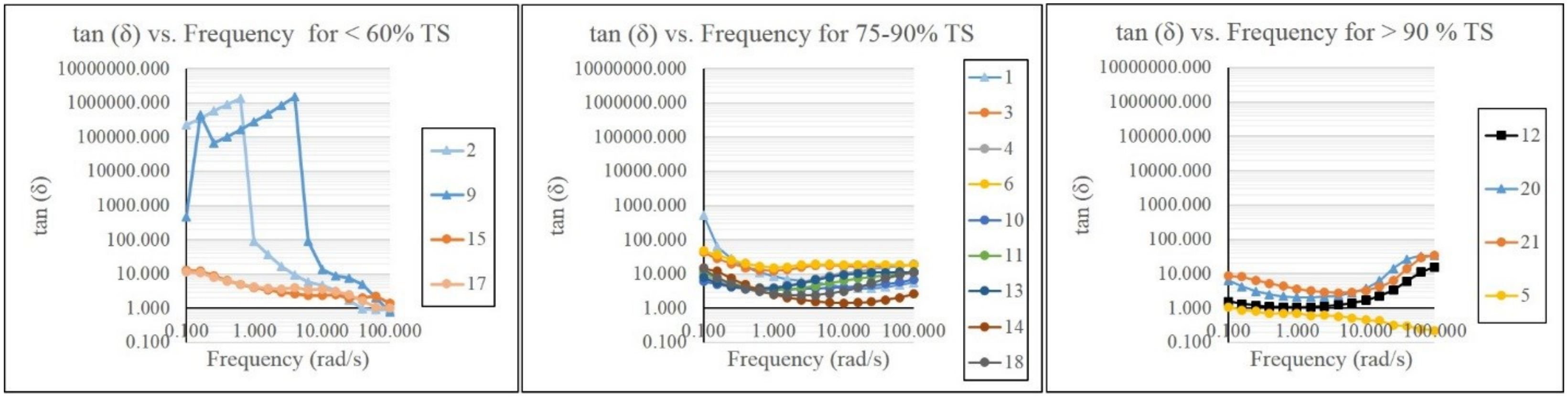
A



B

Predicted Complex Viscosity at 0.1 rad/s for Factor Interactions: Low Water (A); High Water (B)

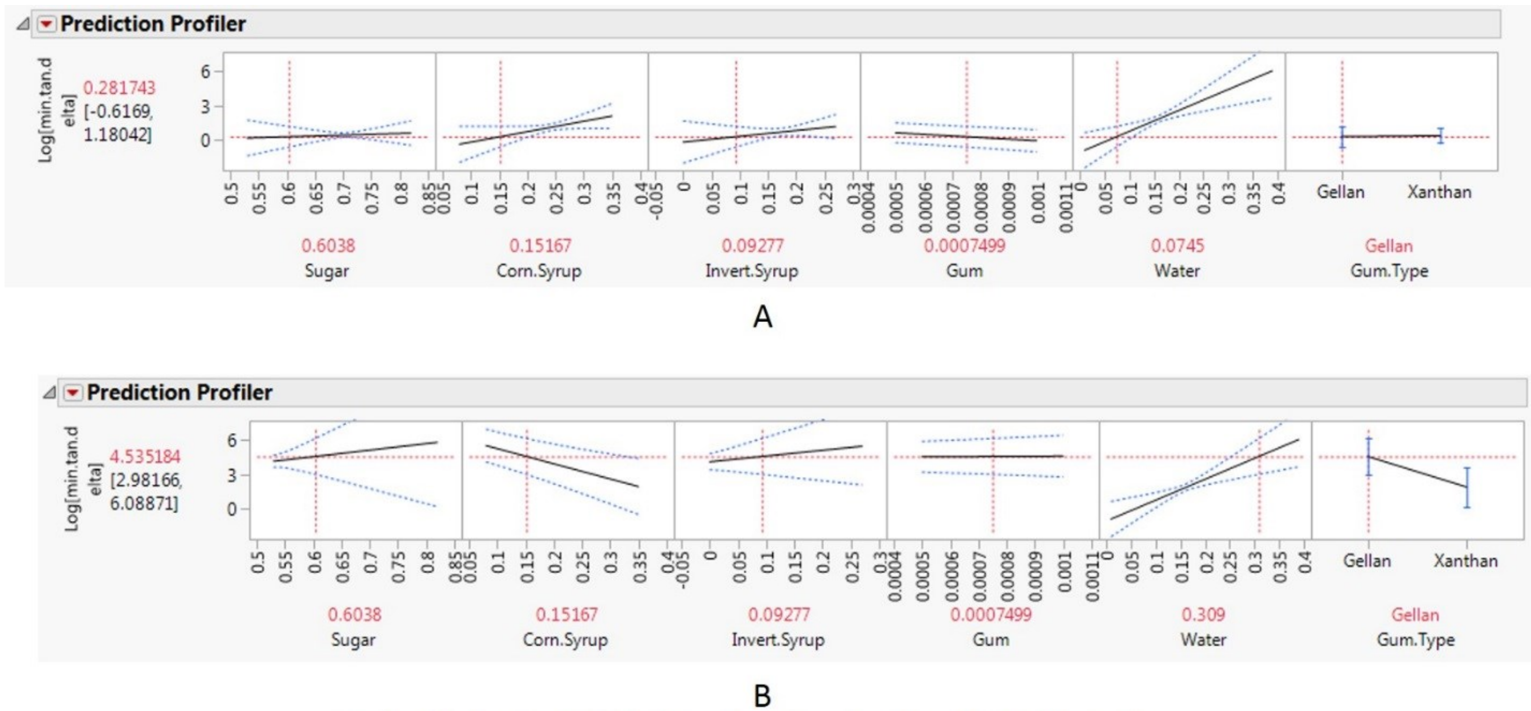
Figure 2.11Water Interactions from Predicted Complex Viscosity at 0.01 rad/s (Generated in JMP 12)



A B C

Comparison Between tan (δ) vs. Frequency (rad/s) for Low (A), Medium (B), and High (C) % TS Levels

Figure 2.12 Frequency vs. tan (δ) for < 59% TS Treatments (A), 75-90%TS Treatments (B), and >90%TS Treatments (C)



Predicted log[min tan(delta) for Factor Interactions: Low Water (A); High Water (B)

Figure 2.13 Prediction profiler for low water (A) and high water (B) levels to show water interactions for log [min tan (delta)] from JMP 12

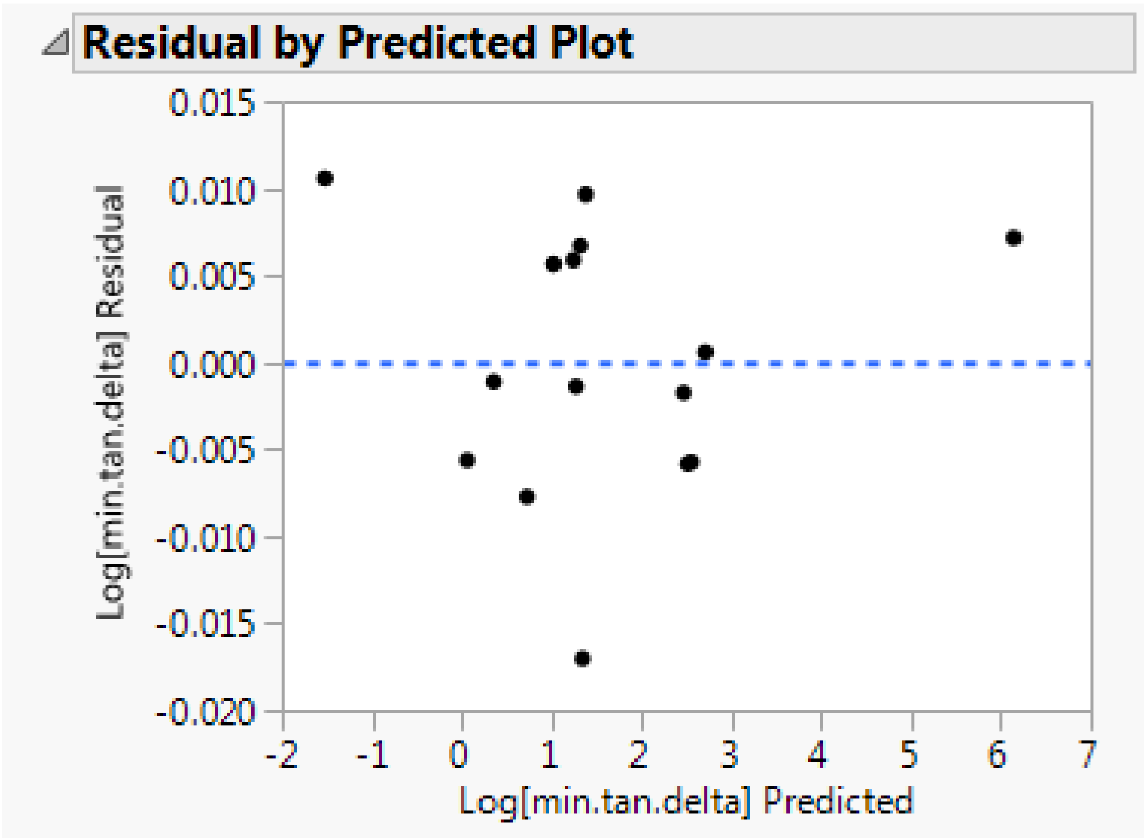
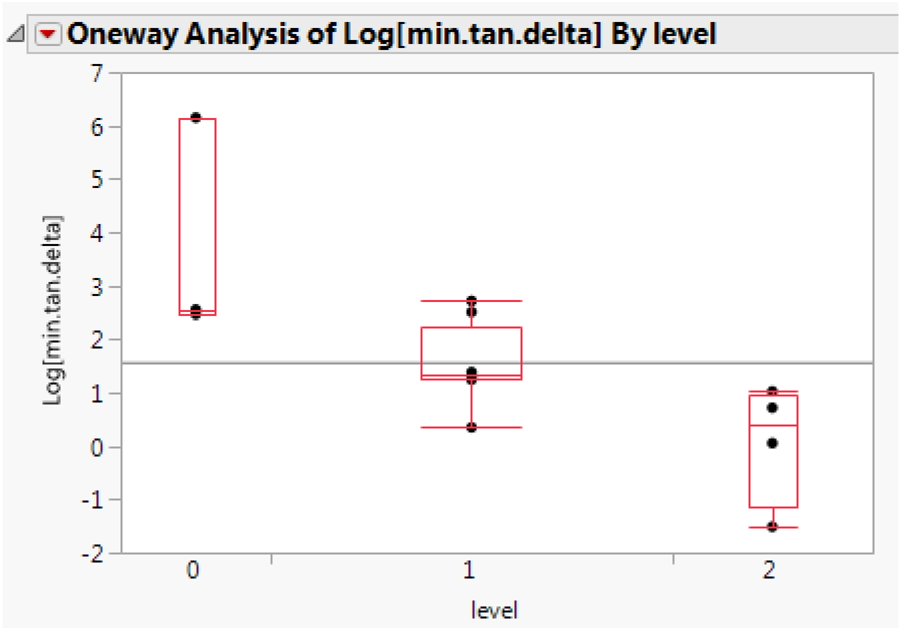


Figure 2.14 Residual Plot of Log (min tan delta) Transformation



Connecting Letters Report

Level			Mean
0	A		3.7230723
1		B	1.5152281
2		B	0.0730623

Levels not connected by same letter are significantly different.

Levels: 0 = < 60%TS, 1 = 75-90%TS, 2 = > 90% TS

Figure 2.15 Oneway ANOVA of log [min tan delta] by %TS Level w/ Tukey HSD Comparison

Table 2.1 Treatment Coding

	level					
variable	**0**	**1**	**2**	**3**	**4**	**5**
sugar (gs (grown)/ps (powdered))	*	0.5	0.6	0.7	0.8	*
corn syrup (cs)	*	0.1	0.2	0.3	0.4	*
invert syrup (is)	0	0.05	0.1	0.2	0.3	*
gum (g (gellan)/x (xanthan))	*	0.0005	0.0007	0.001	*	*
water (w)	*	0.01	0.05	0.1	0.2	0.4

* = no treatments at this level were made for this factor

Table 2.2 Design of Experiments with Sample Number and Treatment Code

Sample	TREATMENT CODE**	Sugar	Corn.Syrup	Invert.Syrup	Gum	Sugar.Type	Gum.Type	Water	%TS (not gum)
1	2gs 2cs 2is 2g 1w	0.6	0.2	0.09	0.0007	Grown.Sugar	Gellan	0.13	81.73
2	1gs 1cs 0is 1g 5w	0.5	0.1	0.00	0.0005	Grown.Sugar	Gellan	0.39	59.48
3	1ps 4cs 0is 1g 3w	0.5	0.4	0.00	0.0005	Powdered.Sugar	Gellan	0.12	81.35
4	1ps 2cs 2is 1x 3w	0.5	0.2	0.14	0.0005	Powdered.Sugar	Xanthan	0.12	80.63
5	3ps 1cs 3is 3g 1w	0.7	0.1	0.20	0.0010	Powdered.Sugar	Gellan	0.01	92.58
6	1ps 1cs 4is 1g 3w	0.5	0.1	0.27	0.0005	Powdered.Sugar	Gellan	0.12	79.92
7	2gs 3cs 0is 3x 1w	0.7	0.3	0.00	0.0005	Grown.Sugar	Gellan	0.01	92.99
8	3ps 3cs 0is 3x 1w	0.7	0.3	0.00	0.0010	Powdered.Sugar	Xanthan	0.01	92.59
9	1ps 1cs 0is 3g 5w	0.5	0.1	0.00	0.0010	Powdered.Sugar	Gellan	0.39	59.48
10	3ps 1cs 0is 1x 4w	0.7	0.1	0.00	0.0005	Powdered.Sugar	Xanthan	0.22	76.88
11	1gs 1cs 4is 3x 3w	0.5	0.1	0.27	0.0010	Grown.Sugar	Xanthan	0.12	79.92
12	3gs 1cs 3is 1x 1w	0.7	0.1	0.22	0.0005	Grown.Sugar	Xanthan	0.01	92.18
13	1gs 4cs 0is 3x 3w	0.5	0.4	0.00	0.0010	Grown.Sugar	Xanthan	0.12	81.35
14	4gs 1cs 0is 3g 3w	0.8	0.1	0.00	0.0010	Grown.Sugar	Gellan	0.10	88.48
15	1gs 1cs 0is 3x 5w	0.5	0.1	0.00	0.0010	Grown.Sugar	Xanthan	0.39	59.48
16	4ps 2cs 0is 1x 1w	0.8	0.2	0.00	0.0005	Powdered.Sugar	Xanthan	0.01	95.73
17	1ps 1cs 0is 1x 5w	0.5	0.1	0.00	0.0005	Powdered.Sugar	Xanthan	0.39	59.48
18	3ps 2cs 0is 3x 3w	0.7	0.2	0.00	0.0010	Powdered.Sugar	Xanthan	0.10	85.49
19	4ps 1cs 2is 1x 1w	0.8	0.1	0.09	0.0005	Powdered.Sugar	Xanthan	0.01	95.26
20	2ps 2cs 2is 3x 1w	0.6	0.2	0.13	0.0010	Powdered.Sugar	Xanthan	0.01	91.59
21	2gs 3cs 2is 1g 1w	0.6	0.3	0.09	0.0005	Grown.Sugar	Gellan	0.01	91.97
22	4ps 1cs 1is 1x 2w	0.8	0.1	0.05	0.0005	Powdered.Sugar	Xanthan	0.05	92.19

** ps= powdered sugar, gs = grown sugar, cs= corn syrup, is = invert syrup, g = gellan, x= xanthan, w = water

Table 2.3 ANOVA and Effect Tests for Sugar, Corn Syrup, and Invert Sugar vs. °Bx

Summary of Fit

Rsquare	0.968998
Rsquare Adjusted	0.963185
RMSE	1.812754
Mean of Response	76.625
Observations	20

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F
Model	3	1643.3602	547.787	166.6992	<0.0001*
Error	16	52.5773	3.286		
C. Total	19	1695.9375			

Parameter Estimates

Term	Estimate	Std Error	t ratio	prob > ltl
Intercept	20.4618	2.968328	6.89	<0.0001*
Sugar	64.13175	4.311546	14.87	<0.0001*
Corn Syrup	70.85428	4.194572	16.89	<0.0001*
Invert Syrup	61.9046	4.574353	13.53	<0.0001*

Table 2.4 Treatments with Average Complex Viscosity Slope for Average of all Repetitions and 1st Repetition Only to Remove Changes due to Treatment Breakdown

		gs/ps	cs	is	g/x			w	60-96% solids		1st run only	1st run only
Sample	**TREATMENT CODE****	**Sugar**	**Corn.Syrup**	**Invert.Syrup**	**Gum**	**Sugar.Type**	**Gum.Type**	**Water**	**%TS (not gum)**	**AVG η^* slope**	**η^* Slope**	**η^* at 0.1 rad/s**
1	2gs 2cs 2is 2g 1w	0.6	0.2	0.09	0.0007	Grown.Sugar	Gellan	0.13	81.73	-0.17156	-0.169246	13.8796
2	1gs 1cs 0is 1g 5w	0.5	0.1	0.00	0.0005	Grown.Sugar	Gellan	0.39	59.48	-0.590723667	0.09171	0.229806
3	1ps 4cs 0is 1g 3w	0.5	0.4	0.00	0.0005	Powdered.Sugar	Gellan	0.12	81.35	-0.252853333	-0.0846946	24.4358
4	1ps 2cs 2is 1x 3w	0.5	0.2	0.14	0.0005	Powdered.Sugar	Xanthan	0.12	80.63	-0.201789	-0.212267	25.431
5	3ps 1cs 3is 3g 1w	0.7	0.1	0.20	0.0010	Powdered.Sugar	Gellan	0.01	92.58	-0.699968667	-0.674315	8458950
6	1ps 1cs 4is 1g 3w	0.5	0.1	0.27	0.0005	Powdered.Sugar	Gellan	0.12	79.92	-0.048672767	-0.0538737	21.9893
7	2gs 3cs 0is 3x 1w	0.7	0.3	0.00	0.0005	Grown.Sugar	Gellan	0.01	92.99	*	*	*
8	3ps 3cs 0is 3x 1w	0.7	0.3	0.00	0.0010	Powdered.Sugar	Xanthan	0.01	92.59	*	*	*
9	1ps 1cs 0is 3g 5w	0.5	0.1	0.00	0.0010	Powdered.Sugar	Gellan	0.39	59.48	0.319119667	0.298795	0.312536
10	3ps 1cs 0is 1x 4w	0.7	0.1	0.00	0.0005	Powdered.Sugar	Xanthan	0.22	76.88	-0.252853333	-0.253007	6.92356
11	1gs 1cs 4is 3x 3w	0.5	0.1	0.27	0.0010	Grown.Sugar	Xanthan	0.12	79.92	-0.269207333	-0.263694	13.8263
12	3gs 1cs 3is 1x 1w	0.7	0.1	0.22	0.0005	Grown.Sugar	Xanthan	0.01	92.18	-0.739469333	-0.738138	12837.6
13	1gs 4cs 0is 3x 3w	0.5	0.4	0.00	0.0010	Grown.Sugar	Xanthan	0.12	81.35	-0.205624	-0.21444	73.6419
14	4gs 1cs 0is 3g 3w	0.8	0.1	0.00	0.0010	Grown.Sugar	Gellan	0.10	88.48	-0.531288667	-0.535376	1180.75
15	1gs 1cs 0is 3x 5w	0.5	0.1	0.00	0.0010	Grown.Sugar	Xanthan	0.39	59.48	-0.130166333	-0.133592	0.29108
16	4ps 2cs 0is 1x 1w	0.8	0.2	0.00	0.0005	Powdered.Sugar	Xanthan	0.01	95.73	*	*	*
17	1ps 1cs 0is 1x 5w	0.5	0.1	0.00	0.0005	Powdered.Sugar	Xanthan	0.39	59.48	-0.0773367	-0.0656011	0.173764
18	3ps 2cs 0is 3x 3w	0.7	0.2	0.00	0.0010	Powdered.Sugar	Xanthan	0.10	85.49	-0.437725	-0.440808	281.732
19	4ps 1cs 2is 1x 1w	0.8	0.1	0.09	0.0005	Powdered.Sugar	Xanthan	0.01	95.26	*	*	*
20	2ps 2cs 2is 3x 1w	0.6	0.2	0.13	0.0010	Powdered.Sugar	Xanthan	0.01	91.59	-0.512477667	-0.514444	8428.81
21	2gs 3cs 2is 1g 1w	0.6	0.3	0.09	0.0005	Grown.Sugar	Gellan	0.01	91.97	-0.461811	-0.479666	5813.85
22	4ps 1cs 1is 1x 2w	0.8	0.1	0.05	0.0005	Powdered.Sugar	Xanthan	0.05	92.19	*	*	*

** ps= powdered sugar, gs = grown sugar, cs= corn syrup, is = invert syrup, g = gellan, x= xanthan, w = water

* not able to be run

Table 2.5 Summary of Fit, ANOVA, and Effect Tests for Log [η*] at 0.1 rad/s Model Generated

Summary of Fit

Rsquare	0.978168
Rsquare Adjusted	0.941782
RMSE	1.142224
Mean of Response	4.189946
Observations	17

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F
Model	10	350.7356	35.0736	26.883	0.0003*
Error	6	7.82805	1.3047		
C. Total	16	358.5636			

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Sugar	1	1	1.023815	0.7847	0.4098
Corn Syrup	1	1	0.087211	0.0668	0.8046
Invert Syrup	1	1	3.868318	2.965	0.1359
Gum	1	1	8.44123	6.47	0.0439*
Sugar Type	1	1	1.836794	1.4079	0.2803
Gum Type	1	1	7.004747	5.369	0.0597
Water	1	1	0.001708	0.0013	0.9723
Sugar*Water	1	1	0.079475	0.0609	0.8133
Invert Syrup * Water	1	1	11.102	8.5094	0.0267*
Gum * Water	1	1	2.28575	1.752	0.2338
Gum Type*Water	1	1	3.768422	2.8884	0.1401

Parameter Estimates

Term	Estimate	Std Error	t ratio	prob > |t|
Sugar	5.526261	6.23838	0.89	0.4098
Corn Syrup	-1.73278	6.702079	-0.26	0.8046
Invert Syrup	26.97271	15.66444	1.72	0.1359
Gum	5116.135	2011.362	2.54	0.0439*
Sugar Type (grown)	-0.35667	0.300598	-1.19	0.2803
Gum Type (gellan)	1.161536	0.501288	2.32	0.0597
Water	-1.38492	38.27423	-0.04	0.9723
Sugar*Water	-20.1972	81.8327	-0.25	0.8133
Invert Syrup * Water	-263.941	90.48109	-2.92	0.0267*
Gum * Water	-11261.3	8507.957	-1.32	0.2338
Gum Type(gellan)*Water	-3.5654	2.097875	-1.7	0.1401

Table 2.6 ANOVA and Effect Tests for η* Slope Model Generated by Stepwise Regression

Summary of Fit

Rsquare	0.924953
Rsquare Adjusted	0.849905
RMSE	0.103647
Mean of Response	-0.30967
Observations	17

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F
Model	8	1.059229	0.132404	12.3249	0.0009*
Error	8	0.085942	0.010743		
C. Total	16	1.145171			

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Sugar	1	1	0.06622	6.1642	0.0379*
Corn Syrup	1	1	0.007959	0.7408	0.4114
Invert Syrup	1	1	0.05538	5.1551	0.0528
Gum	1	1	0.007886	0.7341	0.4165
Water	1	1	0.038157	3.5519	0.0962
Sugar Type	1	1	0.000879	0.0818	0.7821
Invert Syrup * Water	1	1	0.135224	12.5875	0.0075*
Gum * Water	1	1	0.097212	9.049	0.0169*
Water * Sugar Type	1	1	0.104814	9.7568	0.0142*

Parameter Estimates

Term	Estimate	Std Error	t ratio	prob > |t|
Sugar	-0.55957	0.225382	-2.48	0.0379*
Corn Syrup	0.243866	0.28333	0.86	0.4144
Invert Syrup	-1.23731	0.54495	-2.27	0.0528
Gum	-151.048	176.2935	-0.86	0.4165
Water	-1.09091	0.578838	-1.88	0.0962
Sugar Type	0.012075	0.042219	0.29	0.7821
Invert Syrup * Water	15.50329	4.369734	3.55	0.0075*
Gum * Water	2399.873	797.7871	3.01	0.0169*
Water * Sugar Type	-0.61934	0.198278	-3.12	0.0142*

Table 2.7 Treatments Showing Effect on tan (δ) Organized From Lowest to Highest Total Solids

Sample	TREATMENT CODE**	%TS (not gum)	tan(delta) data range	min tan(delta)	freq.at min tan(delta) rad/s	max tan(delta)	freq.at max tan(delta) rad/s
2	1gs 1cs 0is 1g 5w	59.48	1351639.101	0.899083	100	1351640	0.630957
9	1ps 1cs 0is 3g 5w	59.48	1550249.218	0.782305	100	1550250	3.98105
15	1gs 1cs 0is 3x 5w	59.48	11.47464	1.36436	100	12.839	0.1
17	1ps 1cs 0is 1x 5w	59.48	10.77609	1.00731	100	11.7834	0.1
10	3ps 1cs 0is 1x 4w	76.88	3.34842	3.47123	1	6.81965	100
6	1ps 1cs 4is 1g 3w	79.92	31.8253	15.084	1	46.9093	0.1
11	1gs 1cs 4is 3x 3w	79.92	7.59527	3.54733	1	11.1426	100
4	1ps 2cs 2is 1x 3w	80.63	15.5016	4.0092	0.630957	19.5108	100
3	1ps 4cs 0is 1g 3w	81.35	29.9325	12.2696	1	42.2021	0.1
13	1gs 4cs 0is 3x 3w	81.35	7.69024	3.75756	0.630957	11.4478	63.0957
1	2gs 2cs 2is 2g 1w	81.73	528.5343148	3.76507	15.849	532.2993848	0.1
18	3ps 2cs 0is 3x 3w	85.49	11.88309	2.38181	2.51189	14.2649	0.1
14	4gs 1cs 0is 3g 3w	88.48	13.53616	1.42224	10.0001	14.9584	0.1
20	2ps 2cs 2is 3x 1w	91.59	31.90518	2.05292	1.5849	33.9581	100
21	2gs 3cs 2is 1g 1w	91.97	32.17654	2.79456	3.98105	34.9711	100
12	3gs 1cs 3is 1x 1w	92.18	14.87155	1.05835	1	15.9299	100
22	4ps 1cs 1is 1x 2w	92.19	*	*	*	*	*
5	3ps 1cs 3is 3g 1w	92.58	0.844589	0.220601	100	1.06519	0.1
8	3ps 3cs 0is 3x 1w	92.59	*	*	*	*	*
7	2gs 3cs 0is 3x 1w	92.99	*	*	*	*	*
19	4ps 1cs 2is 1x 1w	95.26	*	*	*	*	*
16	4ps 2cs 0is 1x 1w	95.73	*	*	*	*	*

** ps= powdered sugar, gs = grown sugar, cs= corn syrup, is = invert syrup, g = gellan, x= xanthan, w = water

* not able to be run

Table 2.8 ANOVA and Effect Tests for log [min tan (delta)] Model Generated by Stepwise Regression

Rsquare	0.999979
Rsquare Adjusted	0.999709
RMSE	0.028679
Mean of Response	1.572219
Observations	15

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F
Model	13	39.56861	3.04374	3700.7792	0.0129*
Error	1	0.000822	0.00082		
C. Total	14	39.56943			

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Sugar	1	1	0.061846	75.1966	0.0731
Corn.Syrup	1	1	0.277398	337.2793	0.0346*
Invert.Syrup	1	1	0.720149	875.6051	0.0215*
Gum	1	1	0.073536	89.4103	0.0671
Water	1	1	0.057028	69.3384	0.0761
Gum.Type[Gellan]	1	1	0.602314	732.3331	0.0235*
Sugar*Invert.Syrup	1	1	0.027494	33.4291	0.109
Sugar*Gum	1	1	0.048388	58.8329	0.0825
Sugar*Water	1	1	0.020788	25.2748	0.125
Corn.Syrup*Invert.Syrup	1	1	0.321175	390.5058	0.0322*
Corn.Syrup*Water	1	1	0.637953	775.6651	0.0228*
Invert.Syrup*Gum	1	1	0.014985	18.2198	0.1465
Gum*Water	1	1	0.128276	155.9669	0.0509
Water*Gum.Type[Gellan]	1	1	5.563228	6764.1403	0.0077*

Parameter Estimates

Term	Estimate	Std Error	t ratio	prob > |t|
Sugar	-4.70787	0.542907	-8.67	0.0731
Corn.Syrup	20.17925	1.098779	18.37	0.0346*
Invert.Syrup	21.37652	0.722409	29.59	0.0215*
Gum	-6621.72	700.2884	-9.46	0.0671
Water	11.00667	1.32181	8.33	0.0761
Gum.Type[Gellan]	-0.48049	0.017755	-27.06	0.0235*
Sugar*Invert.Syrup	-13.9377	2.410623	-5.78	0.109
Sugar*Gum	8160.011	1063.85	7.67	0.0825
Sugar*Water	17.97589	3.57558	5.03	0.125
Corn.Syrup*Invert.Syrup	-43.8204	2.217496	-19.76	0.0322*
Corn.Syrup*Water	-94.6715	3.399242	-27.85	0.0228*
Invert.Syrup*Gum	-1753.83	410.8801	-4.27	0.1465
Gum*Water	6392.784	511.8867	12.49	0.0509
Water*Gum.Type[Gellan]	5.842707	0.071041	82.24	0.0077*

Chapter 3 - Texture Study on A Semi-liquid Model System for Use On Pulp/paste Candy

1: Introduction

In 2001, $166.6 million worth of candy was imported into the United States from Mexico to satiate the US' growing love of spicy candy (Zúñiga, 2002). One typical type of spicy candy is paste/pulp candy. This is a soft, fluid candy with particles suspended in syrup and is typically squeezed out of a package by the consumer. Crystallization and inversion are normally blamed for shelf life failure in pulp/paste candy, though there has been little research completed. Crystallization and inversion create rheological changes to the candy and cause issues with leakage or an inability to squeeze the candy from the package. Therefore, recent research about the textural properties of sugar syrups, gum networks, and sucrose crystallization in similar types of confections can provide insight into creation of a model system for this complex candy.

One study that examined pulp/paste candy was by Molina-Rubio et al. (2010). It studied the concentration of sucrose in a semi-liquid syrup or pulp/paste candy-type system, but did not examine whether the particle size or morphology of the sugar had an impact on rheology and texture of the model system. Molina-Rubio et al. (2010) only studied dextrans and carrageenan for their gum component, but as Kasapis et al. (2004), Tang et al. (2001), Papageorgiou et al. (1994), and Altay & Gunasekaran (2011) showed other gums typically used in pulp/paste candy can affect the rheological properties of sucrose and corn syrup systems. Based upon other studies, such as Tjuradi & Hartel (1995) and Levenson & Hartel (2009) the type of syrup may have an effect on the rheological properties of the system. A modifcation to remove the acid component used in the system proposed by Molina-Rubio et al and instead use invert syrup could help to model the inversion that occurs when pulp candy is processed and still model the shelf life with more control. This research focused on the development of a model system based on the one suggested by Molina-Rubio et al. (2010) and using the system to determine the impact of the type and amount of key ingredients on the textual properties of the system by using probe tests. This broad study also examined the interaction of the key components of the model system to impact formulations for improved shelf life of pulp paste candy. It was the objective of this study to show that the key factors of particle size of sucrose, gum type, and syrup type will impact both the hardness and stickiness of the pulp/paste candy semi-liquid model system created. A custom mixture, D-optimal design was created since it could use both categorical and continuous factors (JMP, 2007).

2: Materials and Methods

2.1 Ingredients

As previously mentioned in Chapter 2, the ingredients used in this study were all donated and the same lots were used in all treatments. The sugar used was Amerfond® crystallized fondant sugar (120 µm mean grown-to-size) and 6x Confectioners' sugar (5 µm mean pulverized-to size) obtained from Domino Foods, Inc. (Yonkers, NY). The syrups use were Clearsweet® 43/43 Corn Syrup from Cargill, Inc. (Minneapolis, MN) and #11 Nulomoline® partial invert syrup from Domino Foods, Inc. (Yonkers, NY). The gums used were Kelcogel F® (low acyl gellan) and Keltrol® (xanthan) provided by CP Kelco (Atlanta, GA). The water used was deionized.

2.2 Experimental Design

Previously mentioned in Chapter 2, JMP 12 (from SAS Institute, Inc.) was used to create a Design of Experiments (DOE) using custom design tools since there were categorical (gum type and sugar type) and continuous factors (levels of gum, invert syrup, corn syrup, and water). A D-optimal design of experiments was created and had built in replicates (JMP, 2007). D-optimal designs also allow for the use of multiple levels for categorical factors, have built in replicates, are used for screening experiments, and minimize material usage, since the screening can be augmented and done sequentially (Jones, Lin, & Nachtsheim, 2008). JMP 12 was used because it is a leader in creating custom mixture D-optimal designs.

A ternary plot developed in JMP 12 with the initial design chosen for the semi-liquid syrup/pulp candy model is shown in Figure 3.1. Formulations with less than fifty percent sugar were not examined since the sugar would fully dissolve in the matrix (Mageen, Kristott, & Jones, 1991). Treatment codes for each sample were created, see Table 3.1. Sample numbers were assigned indicating the order that the treatments were run with the initial design including 17 treatments, but then was augmented to 22 treatments.

2.3 Sample Preparation Methods

As previously mentioned in Chapter 2, a 500g sample was prepared for each run identified (Table 3.2). Five grams of water (subtracted from the total amount) were used to hydrate the gum for each treatment. This then was mixed and left to hydrate for a half hour. Corn syrup and/or invert syrup with any required additional water were added to a stainless steel pot along with the hydrated gum. This pot was then heated to 75°C, measured by using a temperature probe (Cole Parmer, type K, 10” stainless steel) and thermometer (Fluke 52II), then removed from heat. The specified type of sifted sugar was then added and mixed. Soluble solids were measured by using a hand refractometer (ATAGO Master-100H) similar to the Molina-Rubio et al. (2010) methodology, but since not all treatments read as clearly as Figure 3.2, it was decided to use calculated total solids. The calculated percent total solids no gum (%TS) for each treatment was determined by using the same formula stated in Chapter 2.

The sample was then stored into a glass Mason jar at ambient temperatures until ready to read on the TX-XT2 Texture Analyzer (Texture Technologies Corp., New York).

2.4 Texture Testing

A TA-XT2 Texture Analyzer (Texture Technologies Corp., New York) was used to measure the hardness of the pulp samples, see Figure 3.3. This instrument is typically used in industry to measure texture. Since the probe that Molina-Rubio et al. (2010) used was not available, a method for caramel was selected because it is similar in texture and generates acceptable curves from typical pulp/paste candy products (Kilcast & Roberts, 1998).

The method selected was a modified version of the method for caramel, “Properties of caramel using a 0.75” spherical probe” from the Exponent Stable Micro Systems Software applications guide. This method had the probe travel at 5.0mm/s, traveled 2.0mm into the sample surface to test hardness and withdrew at 10mm/s to test stickiness. The modification on this method was the use of a 0.75” stainless steel spherical probe vs. a 0.75” acrylic probe. This test was run five times on each of the DOE treatments.

2.5 Texture Analysis

A macro calculating average peak force and average negative peak force was run using Exponent Stable Micro Systems software. Statistics were calculated using JMP 12. ANOVA was used to identify any differences and interactions between model system ingredients and the average peak force and the negative average peak force. To try and normalize the data, a log transformation was done on the average peak force data. Log10 [Avg peak force] and average negative peak force were compared with %TS using oneway ANOVA with Tukey HSD.

3: Results and Discussion

3.1 Analysis of Hardness

The texture-gram for treatments showed that the 5 repetitions of probe tests per treatment were similar and could be averaged together (Figure 3.4). There was a general trend that as the solids increase, the average peak force increases (Table 3.3). Treatments 1gs 1cs 0is 1g 5w, 1ps 1cs 0is 3g 5w, 1gs 1cs 0is 3x 5w, 1ps 1cs 0is 1x 5w with 59.48% TS all have a peak force around 12 N, where treatments 2gs 3cs 2is 1g 1w,2gs 3cs 2is 1g 1w, 3gs 1cs 3is 1x 1w, 4ps 1cs 1is 1x 2w, 3ps 1cs 3is 3g 1w, 3ps 3cs 0is 3x 1w with about 92%TS all have over 1,000 N of peak force required for sample penetration. The larger mean peak force for treatments with >90%TS is shown in Figure 3.5. This higher peak force required is likely because with higher concentrations of sucrose the water would bond with additional sucrose in the treatments. This hydrogen bonding between the hydroxyl groups in the sucrose and the hydroxyl groups in the gums created a strong, cohesive, network and requiring more force from the probe to disrupt it. Treatment 4ps 2cs 0is 1x 1w was not measured, since it was a non-homogenous sample, due to insufficient moisture. For treatments 2gs 3cs 0is 3x 1w and 4ps 1cs 2is 1x 1w with > 93% TS, the peak force dropped. The lower force could be explained by lose cohesion in the treatments due to a scarcity of water to dissolve sugar particles or fully hydrate the gum, resulting in a more malleable treatment. To remove residual trends exponential and log10 transformed models were created using JMP 12 (Figure 3.6). From the data gathered (Figure 3.7 & Table 3.4), there appears an inflection point in hardness between 85-92 %TS. The force required for treatment penetration increases exponentially in this sweet spot, suggesting that this solids level be avoided for pulp type candy, since the candy is typically squeezed out of the package. The log10 transformed average peak force data explains about 54% of the variance in the data, but there might be some residual trends (Table 3.5). The log10 transformed data for average peak force does not show that gum level had a significant effect on hardness. For sugar level and water level, the increased peak force in the higher solids area might be due to the sucrose acting as a stabilizer on the hydrated gum helices, as suggested by Tang et al. (2001) and Kawai et al. (2008). A study by Papageorgiou et al. (1994) found that at higher sucrose concentrations (85% solutes) and a higher temperature (90°C), the gellan gum dramatically increased the mechanical strength in a sucrose-corn syrup system. While Papageorgiou et al. (1994), Tang et al. (2001), and Kawai et al. (2008) did not test xanthan gum and focused on temperature variation, the sucrose-gum interaction is likely a key factor in explaining this inflection point. The area of 85-92 % TS is within the typical soluble solids range of 80-90°Bx for pulp/paste candies and could result in issues extruding the product. Further experimentation should be done within this range to verify that there is indeed a hardness tipping point.

3.2 Analysis of Stickiness/Adhesion

Treatment 1ps 2cs 2is 1x 3w, stuck to the probe after it penetrated the sample and clung on when it backed out (cohesive failure) and treatment 4ps 1cs 2is 1x 1w did not stick to the probe at all (adhesive failure), showing that the DoE provided a wide range of textures. Figure 3.8 illustrates an example of cohesive failure from a sticky treatment. In Table 3.3, the average negative peak force did not change much until treatment 4gs 1cs 0is 3g 3w, 88 % TS. After this point the negative peak force became increasingly negative (or more sticky) before lowering again for samples with the highest %TS (Table 3.3 & Figure 3.9). If there was a tipping point, it was not as pronounced as for the average peak force. This stickiness inflection point is on the upper end of the %TS range for pulp/paste type candies. This could create problems filling primary containers if the candy solids fall into the 85-93%TS range. The average negative peak force declined past about a 93% TS level (Table 3.3) potentially because in order to form the strong hydrogen bonds in the cohesive network and with the probe, water is needed. Treatments lose cohesion when there is not enough water to fully dissolve sugar particles or fully hydrate the gum, thus creating a less sticky treatment. Identifying a model for both the positive and negative average peak forces proved difficult. There were residual trends shown in the predicted plots, (Figure 3.10) for average negative peak force. Log, exponential, absolute value, and natural log transformations did not help to randomize the residuals. JMP 12 was used to generate an ANOVA (analysis of variance) model. The best model for stickiness was over fit (adjusted R^2 = 0.59, R^2 = 0.73) (Table 3.6). The model generated shows that the interactions between invert syrup and water as well as invert syrup level and gum type have significant effects on the maximum stickiness of the treatments. The gum type as a significant determinant of texture (xanthan gum treatments were more sticky than gellan treatments) agrees with the study by Molina-Rubio et al. (2010), though a different probe material was used and different gums were tested in that study. Based upon the model system, using gellan gum over xanthan in formulation of pulp/paste type candy would be preferable due to the cleaner fill and less residue stuck in the containers after eating since it was less sticky.

The interaction of invert syrup and water caused the stickiness of the model system to increase with the increase of water (Table 3.6). This increase in stickiness could be caused by the increase in small molecules, from the glucose and fructose monosaccharides that form invert syrup and water. This larger amount of small molecules could be increasing the free volume of the treatment, leading to a thermodynamically favorable attraction between the treatment and the probe. Foegeding and Steiner (2002) stated this was the case for caramels, a similar matrix to the system tested. They theorized the increase of low molecular weight saccharides in the syrups with higher dextrose equivalents (versus the low DE corn syrups with high molecular weight polysaccharides) helped to act as plasticizers, similar to the other small molecules in the system (water and sugar). The increase of small molecules acted as diluents. This increased the free volume of the treatment due to the decrease in average molecular weight of the mixture. This increase of free volume could contribute to a lower T_g. Since it is thermodynamically favorable for materials with lower energy to adsorb to higher energy materials/surfaces to lower the surface energy of the system as a whole, it might explain why treatments with a lower T_g would stick to a wrapper (Foegeding & Steiner, 2002). To confirm that gum type and invert syrup-water interactions impact stickiness in a semi-liquid model system, more samples should be run, especially in the higher total solids (not gum) range, since this model is not optimal.

4: Conclusion of Texture Study

The texture study helped to analyze the samples that were too high of a viscosity for the ARG-2. When the treatments were ordered in order of ascending %TS, it showed a tipping point/ inflection point for hardness, where the treatment becomes exponentially harder after 85%TS, and then decreases once the solids level goes beyond 93%TS. The interaction of invert syrup and water caused the stickiness of the model system to increase with the increase of water. Since this explorative study focused on breadth of samples with the design of experiments, the relationships of invert syrup level, gum type, and water level in this semi-liquid syrup system should be explored further as they influence the pulp model texture. This indicates that to create an easy-to-eat candy with less filling/eating residue, no high fructose corn syrup/invert syrup should be used in formulation and gellan gum should be used over xanthan.

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Figures:

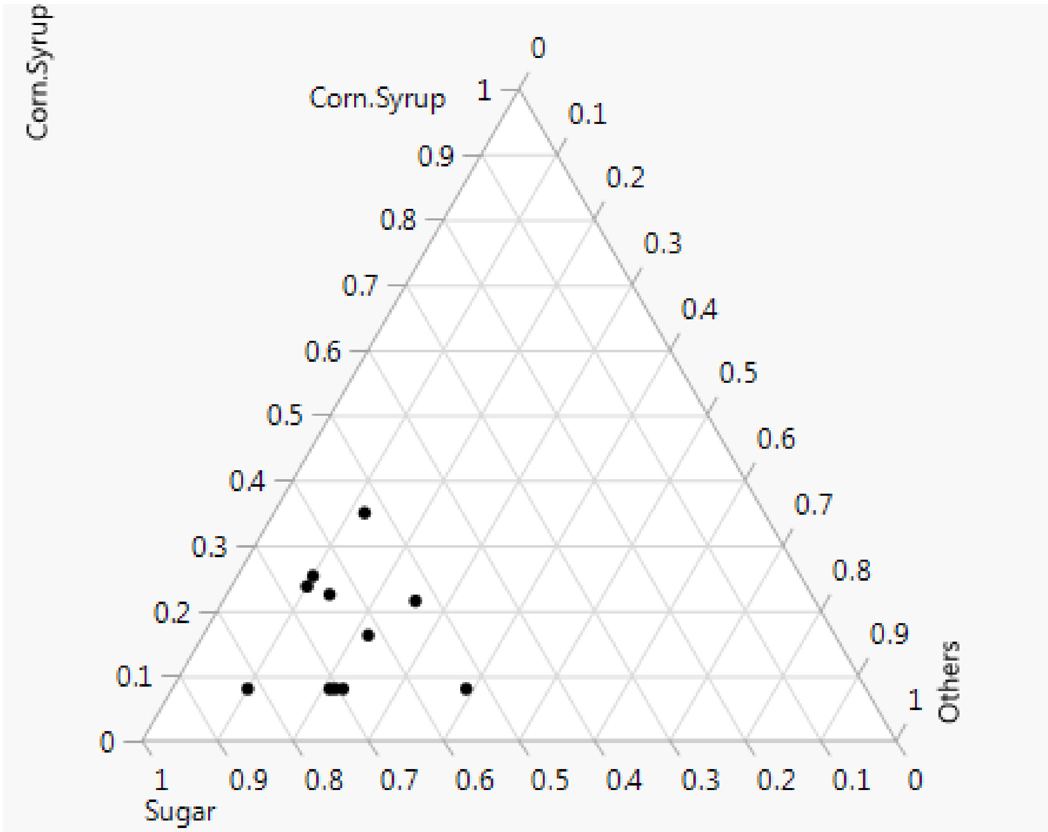


Figure 3.1 Ternary Plot of Initial Samples

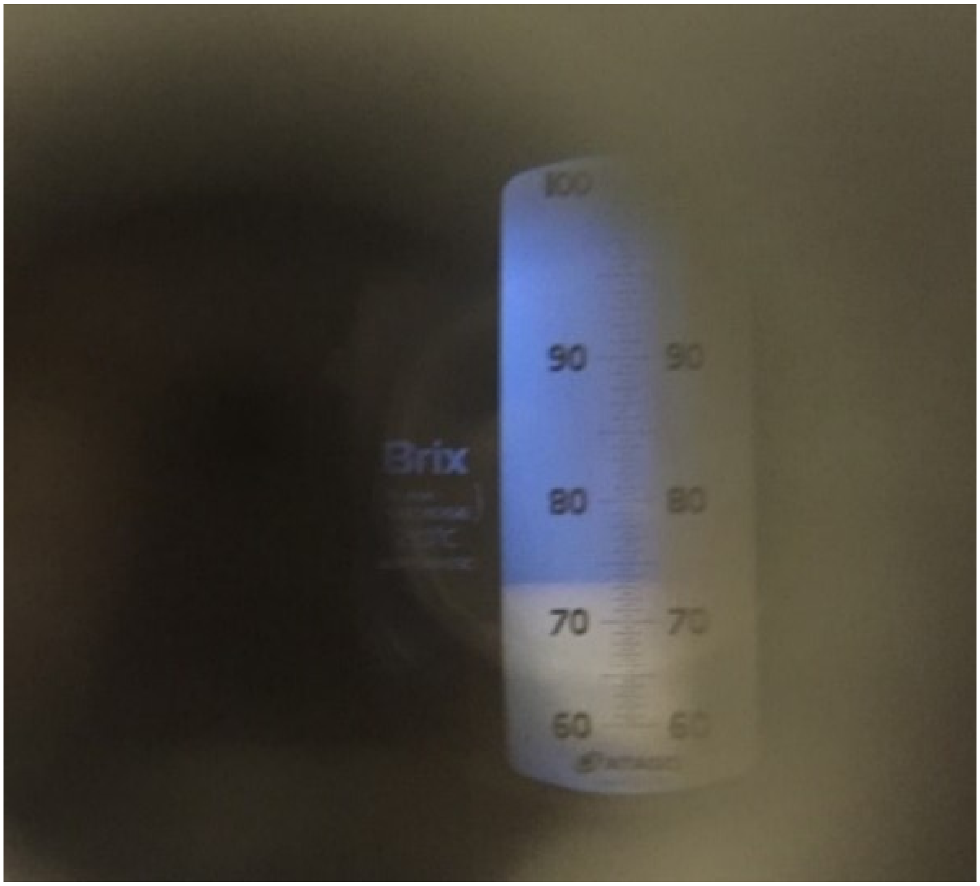


Figure 3.2 View from Refractometer

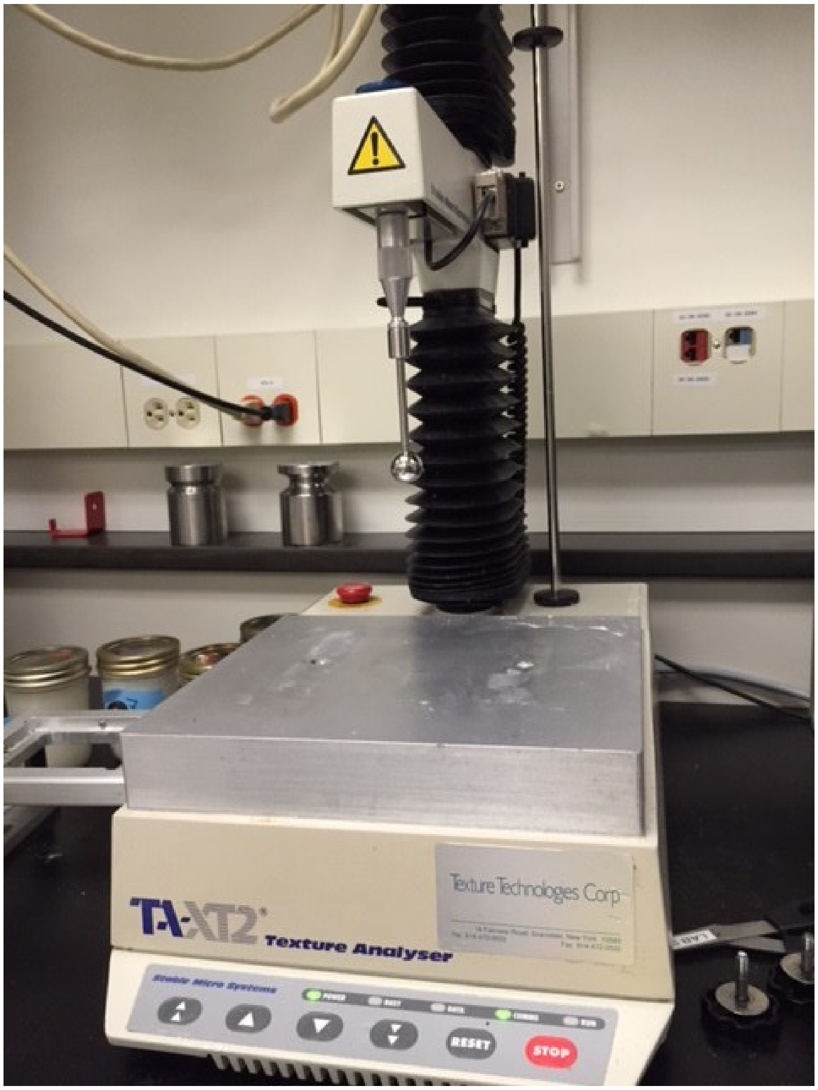


Figure 3.3 TA-XT2 with 0.75” Stainless Steel Ball Probe

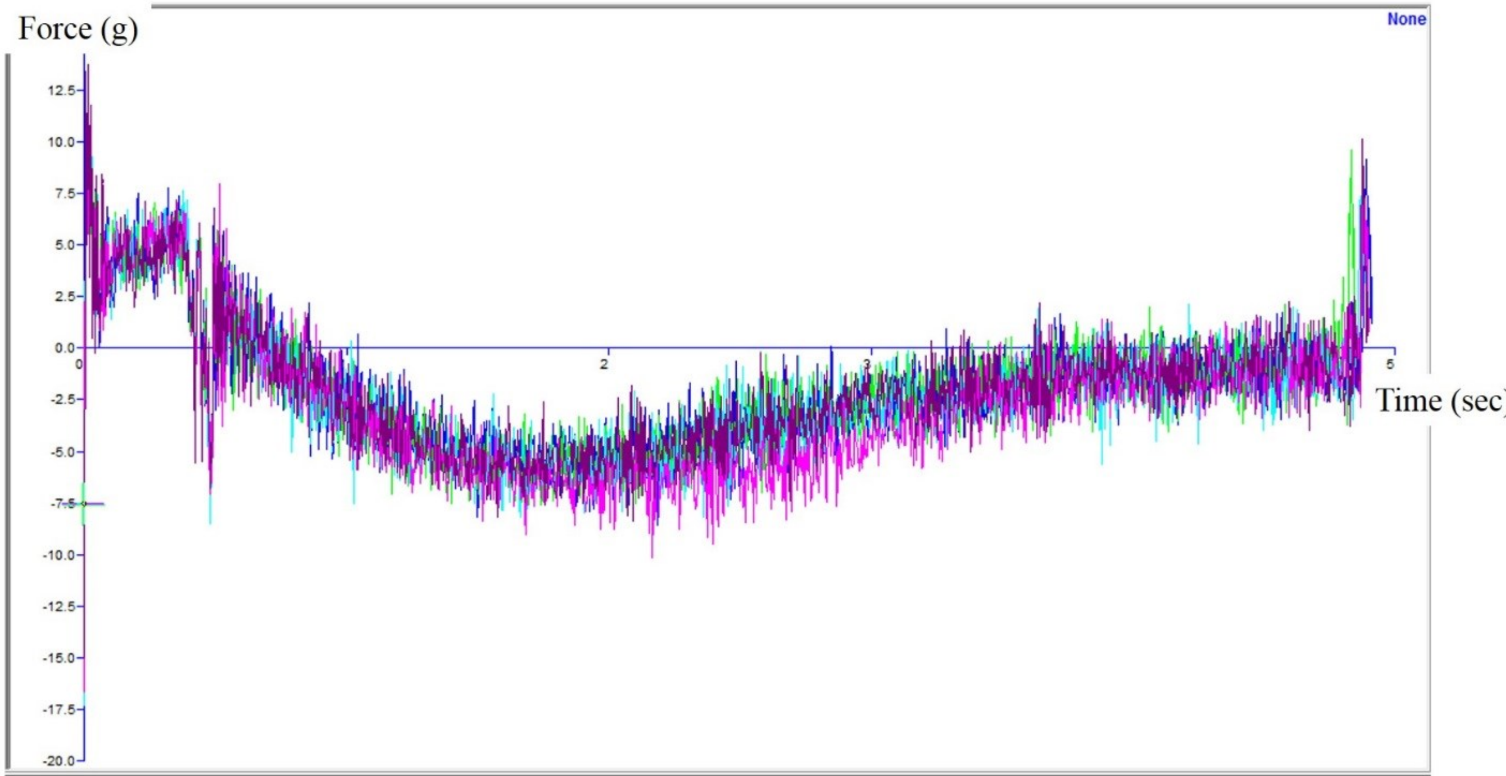
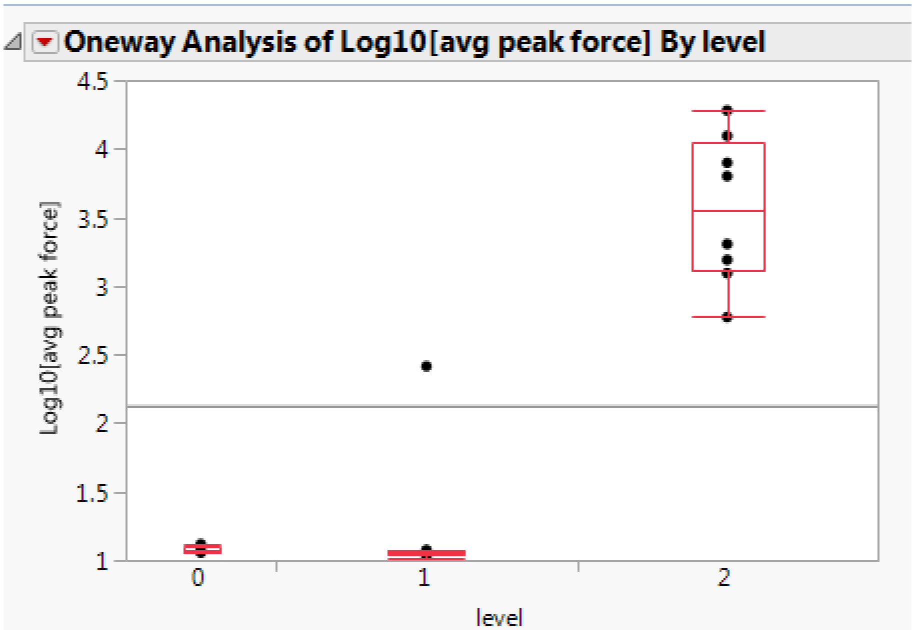


Figure 3.4 Texture-gram showing all 5 Repetitions for Treatment 1ps 2cs 2is 1x 3w (sample 4) with Typical Pulp/Paste Candy Solids (80.63%)

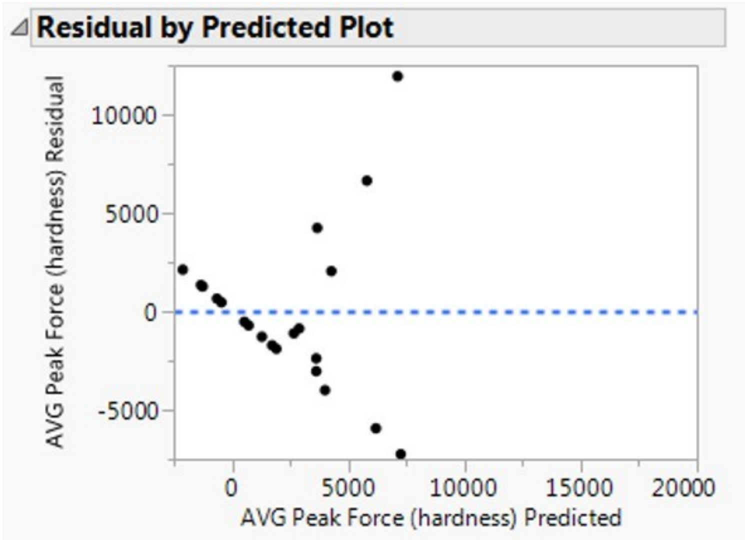


Connecting Letters Report

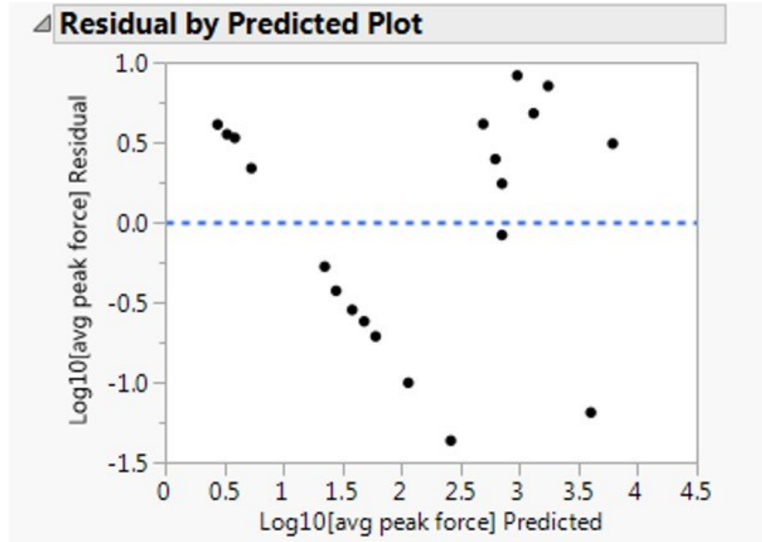
Level			Mean
2	A		3.5550233
1		B	1.2241995
0		B	1.0794875

Levels not connected by same letter are significantly different.

Figure 3.5 Oneway ANOVA of log 10 [AVG Peak Force] by %TS Level w/ Tukey HSD Comparison



Residual by Predicted Plot for AVG Peak Force



Residual by Predicted Plot for Log10[AVG Peak Force]

Figure 3.6 Residual by Predicted Plots for AVG Peak Force

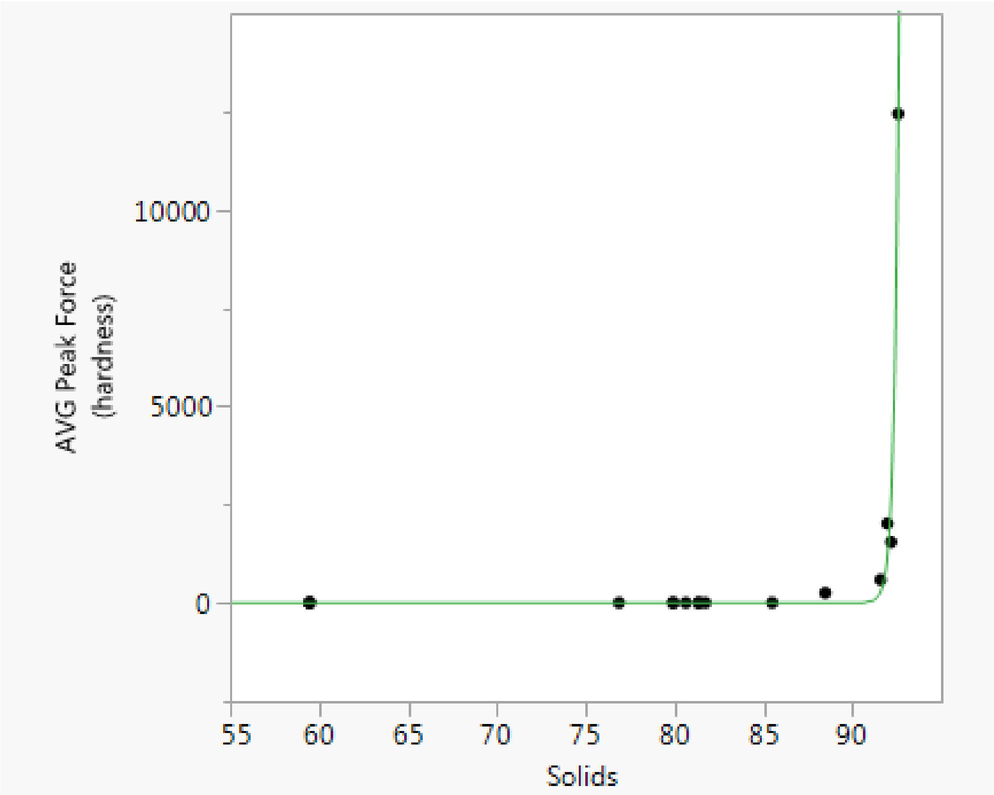


Figure 3.7 Average Peak Force (N) vs. Calculated Solids (%)

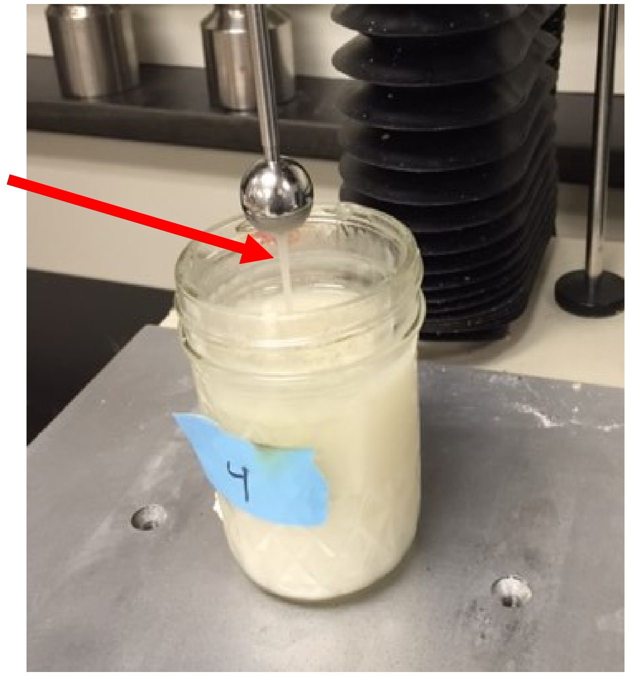
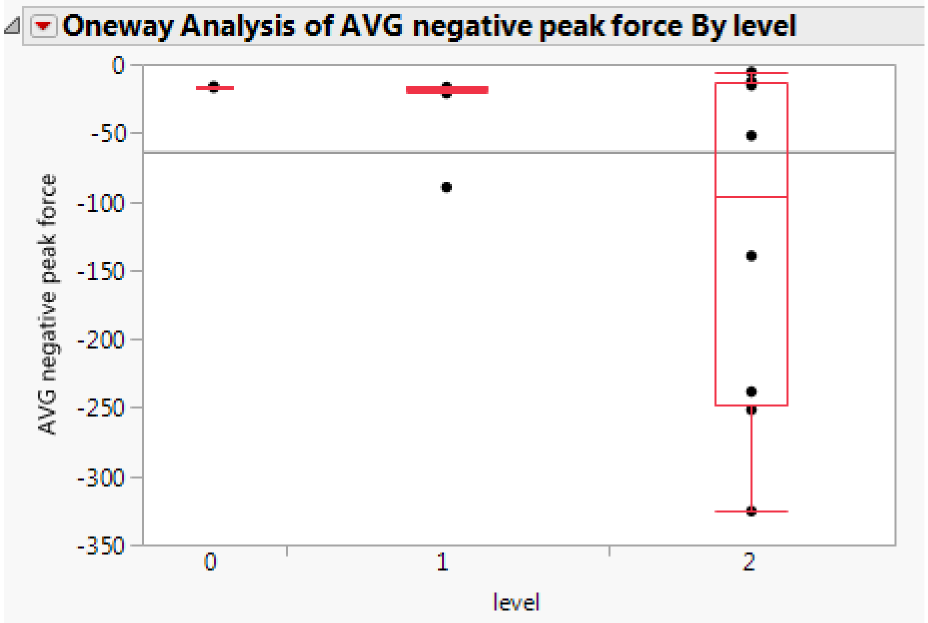


Figure 3.8 Sticky Treatment



Connecting Letters Report

Level			Mean
0	A	B	-16.6748
1	A		-25.4627
2		B	-130.0817

Levels not connected by same letter are significantly different.

Figure 3.9 Oneway ANOVA of AVG Neg. Peak Force by %TS Level w/ Tukey HSD Comparison

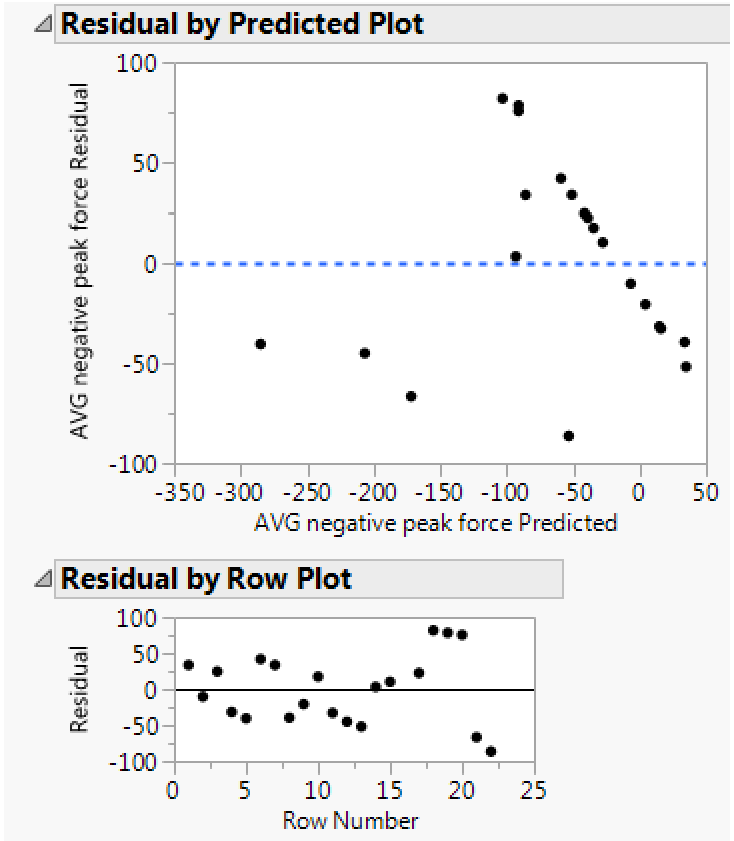


Figure 3.10 Average Negative Peak Force Model Residual Plot

Table 3.1 Treatment Coding

	level					
variable	**0**	**1**	**2**	**3**	**4**	**5**
sugar (gs (grown)/ps (powdered))	*	0.5	0.6	0.7	0.8	*
corn syrup (cs)	*	0.1	0.2	0.3	0.4	*
invert syrup (is)	0	0.05	0.1	0.2	0.3	*
gum (g (gellan)/x (xanthan))	*	0.0005	0.0007	0.001	*	*
water (w)	*	0.01	0.05	0.1	0.2	0.4

* = no treatments at this level were made for this factor

Table 3.2 Design of Experiments with Sample Number and Treatment Code

Sample	TREATMENT CODE**	Sugar	Corn.Syrup	Invert.Syrup	Gum	Sugar.Type	Gum.Type	Water	%TS (not gum)
1	2gs 2cs 2is 2g 1w	0.6	0.2	0.09	0.0007	Grown.Sugar	Gellan	0.13	81.73
2	1gs 1cs 0is 1g 5w	0.5	0.1	0.00	0.0005	Grown.Sugar	Gellan	0.39	59.48
3	1ps 4cs 0is 1g 3w	0.5	0.4	0.00	0.0005	Powdered.Sugar	Gellan	0.12	81.35
4	1ps 2cs 2is 1x 3w	0.5	0.2	0.14	0.0005	Powdered.Sugar	Xanthan	0.12	80.63
5	3ps 1cs 3is 3g 1w	0.7	0.1	0.20	0.0010	Powdered.Sugar	Gellan	0.01	92.58
6	1ps 1cs 4is 1g 3w	0.5	0.1	0.27	0.0005	Powdered.Sugar	Gellan	0.12	79.92
7	2gs 3cs 0is 3x 1w	0.7	0.3	0.00	0.0005	Grown.Sugar	Gellan	0.01	92.99
8	3ps 3cs 0is 3x 1w	0.7	0.3	0.00	0.0010	Powdered.Sugar	Xanthan	0.01	92.59
9	1ps 1cs 0is 3g 5w	0.5	0.1	0.00	0.0010	Powdered.Sugar	Gellan	0.39	59.48
10	3ps 1cs 0is 1x 4w	0.7	0.1	0.00	0.0005	Powdered.Sugar	Xanthan	0.22	76.88
11	1gs 1cs 4is 3x 3w	0.5	0.1	0.27	0.0010	Grown.Sugar	Xanthan	0.12	79.92
12	3gs 1cs 3is 1x 1w	0.7	0.1	0.22	0.0005	Grown.Sugar	Xanthan	0.01	92.18
13	1gs 4cs 0is 3x 3w	0.5	0.4	0.00	0.0010	Grown.Sugar	Xanthan	0.12	81.35
14	4gs 1cs 0is 3g 3w	0.8	0.1	0.00	0.0010	Grown.Sugar	Gellan	0.10	88.48
15	1gs 1cs 0is 3x 5w	0.5	0.1	0.00	0.0010	Grown.Sugar	Xanthan	0.39	59.48
16	4ps 2cs 0is 1x 1w	0.8	0.2	0.00	0.0005	Powdered.Sugar	Xanthan	0.01	95.73
17	1ps 1cs 0is 1x 5w	0.5	0.1	0.00	0.0005	Powdered.Sugar	Xanthan	0.39	59.48
18	3ps 2cs 0is 3x 3w	0.7	0.2	0.00	0.0010	Powdered.Sugar	Xanthan	0.10	85.49
19	4ps 1cs 2is 1x 1w	0.8	0.1	0.09	0.0005	Powdered.Sugar	Xanthan	0.01	95.26
20	2ps 2cs 2is 3x 1w	0.6	0.2	0.13	0.0010	Powdered.Sugar	Xanthan	0.01	91.59
21	2gs 3cs 2is 1g 1w	0.6	0.3	0.09	0.0005	Grown.Sugar	Gellan	0.01	91.97
22	4ps 1cs 1is 1x 2w	0.8	0.1	0.05	0.0005	Powdered.Sugar	Xanthan	0.05	92.19

** ps= powdered sugar, gs = grown sugar, cs= corn syrup, is = invert syrup, g = gellan, x= xanthan, w = water

Table 3.3 Treatment Average Peak Force & Average Negative Peak Force Organized by Increasing Total Solids (not gum)

Sample	TREATMENT CODE**	%TS	AVG Peak Force (N)	AVG Negative Peak Force (N)
2	1gs 1cs 0is 1g 5w	59.48	11.38	-16.93
9	1ps 1cs 0is 3g 5w	59.48	11.73	-16.17
15	1gs 1cs 0is 3x 5w	59.48	11.88	-17.27
17	1ps 1cs 0is 1x 5w	59.48	13.12	-16.33
10	3ps 1cs 0is 1x 4w	76.88	11.35	-17.07
6	1ps 1cs 4is 1g 3w	79.92	10.47	-17.24
11	1gs 1cs 4is 3x 3w	79.92	11.88	-16.65
4	1ps 2cs 2is 1x 3w	80.63	10.89	-16.72
3	1ps 4cs 0is 1g 3w	81.35	11.73	-16.75
13	1gs 4cs 0is 3x 3w	81.35	11.67	-16.83
1	2gs 2cs 2is 2g 1w	81.73	11.39	-17.14
18	3ps 2cs 0is 3x 3w	85.49	11.53	-21.10
14	4gs 1cs 0is 3g 3w	88.48	259.84	-89.66
20	2ps 2cs 2is 3x 1w	91.59	593.06	-15.38
21	2gs 3cs 2is 1g 1w	91.97	2,026.69	-238.32
12	3gs 1cs 3is 1x 1w	92.18	1,552.48	-251.64
22	4ps 1cs 1is 1x 2w	92.19	19,070.00	-139.72
5	3ps 1cs 3is 3g 1w	92.58	12,447.35	-325.42
8	3ps 3cs 0is 3x 1w	92.59	6,325.48	-5.57
7	2gs 3cs 0is 3x 1w	92.99	7,916.83	-52.04
19	4ps 1cs 2is 1x 1w	95.26	1,242.23	-12.56
16	4ps 2cs 0is 1x 1w	95.73	*	*

** ps= powdered sugar, gs = grown sugar, cs= corn syrup, is = invert syrup, g = gellan, x= xanthan, w = water

* not able to be run

Table 3.4 Fit of Exponential Model of Average Peak Force by Calculated Solids

Summary of Fit

AICc	255.4924
BIC	256.1459
SSE	2111196
MSE	140746.4
RMSE	375.1618
Rsquare	0.985424

Parameter Estimates

Term	Estimate	Std Error	Lower 95%	Upper 95%
Scale	9.43E-151	2.56E-149	-4.90E-149	5.11E-149
Growth Rate	3.833052	0.2931162	3.2585545	4.4075488

Table 3.5 ANOVA Model for log10 (AVG Peak Force) transformed data

Summary of Fit

Rsquare	0.68664
Rsquare Adjusted	0.542012
RMSE	0.862506
Mean of Response	2.127587
Observations	20

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F
Model	6	21.19106	3.53184	4.7476	0.009*
Error	13	9.67091	0.74392		
C. Total	19	30.86197			

Lack of Fit

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F	Max Rsquare
Lack of Fit	12	9.619357	0.801613	15.5492	0.1959	0.9983
Pure Error	1	0.051553	0.051553			
Total Error	13	9.67091				

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Sugar	1	1	17.62201	23.6882	0.0003*
Corn.Syrup	1	1	0.270444	0.3635	0.5569
Invert.Syrup	1	1	0.829028	1.1144	0.3103
Gum	1	1	0.053016	0.0713	0.7937
Water	1	1	9.719438	13.0652	0.0031*
Sugar.Type[Grown.Sug	1	1	0.146091	0.1964	0.6649
Gum.Type[Gellan]	1	1	0.004299	0.0058	0.9406

Parameter Estimates

Term	Estimate	Std Error	t ratio	prob > |t|
Sugar	5.003585	1.028053	4.87	0.0003*
Corn.Syrup	-1.20702	2.001877	-0.6	0.5569
Invert.Syrup	-2.44455	2.315663	-1.06	0.3103
Gum	215.193	806.0945	0.27	0.7937
Water	-5.50808	1.523848	-3.61	0.0031*
Sugar.Type[Grown.Sug	-0.08787	0.198296	-0.44	0.6649
Gum.Type[Gellan]	0.015268	0.200838	0.08	0.9406

Table 3.6 Model for Average Negative Peak Force/Maximum Stickiness

Summary of Fit

Rsquare	0.734357
Rsquare Adjusted	0.591318
RMSE	59.83623
Mean of Response	-63.6437
Observations	21

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F
Model	7	128670.7	18381.5	5.134	0.0055*
Error	13	46544.6	3580.4		
C. Total	20	175215.6			

Lack of Fit

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Ratio	Prob > F	Max Rsquare
Lack of Fit	12	46540.91	3878.41	980.7063	0.0249*	1
Pure Error	1	3.955	3.95			
Total Error	13	46544.86				

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Sugar	1	1	10041.59	2.8046	0.1179
Corn Syrup	1	1	1700.343	0.4749	0.5029
Invert Syrup	1	1	48066.7	13.4251	0.0029*
Gum	1	1	593.956	0.1659	0.6904
Water	1	1	520.934	0.1455	0.709
Gum Type	1	1	30026.87	8.3865	0.0125*
Invert Syrup * Water	1	1	50653.81	14.1476	0.0024*
Water * Gum Type	1	1	12540.44	3.5026	0.0839

Parameter Estimates

Term	Estimate	Std Error	t ratio	prob > |t|
Sugar	-111.296	66.45719	-1.67	0.1179
Corn Syrup	95.85393	139.0932	0.69	0.5029
Invert Syrup	-1019.45	278.2327	-3.66	0.0029*
Gum	22412.18	55026.41	0.41	0.6904
Water	44.34015	116.2438	0.38	0.709
Gum Type (gellan)	-54.1141	18.68613	-2.9	0.0125*
Invert Syrup * Water	8784.464	2335.465	3.76	0.0024*
Water * Gum Type	180.2959	96.3371	1.87	0.0839

Chapter 4 - Conclusions on the Impact of Ingredient Selection on the Viscosity and Texture of a Semi-liquid Syrup Model System for Use on Pulp/Paste Candy Including Inversion Calculations and Areas of Future Research

1: Inversion Calculations Based on the Study by Molina-Rubio et al. (2010)

While the pH of this study's model system was approximately 5 to intentionally limit the amount of inversion occurring when the treatments were tested, a typical pulp/paste candy has a lower pH. The interaction between invert syrup and water was found to have a significant impact on viscosity and texture showing the impact of high fructose as an ingredient. However, since invert syrup also is the result of inversion due to acid or salt hydrolysis, it would be interesting to see how the different levels of salt and acid typically found in pulp/paste/semi-liquid systems relate to inversion levels. Since sucrose hydrolysis can be catalyzed by salts and hydrogen ions/acids, the addition of these ingredients might have a significant impact on a finished pulp/paste candy's shelf life (Vukov, 1965). Although, with the ANOVA analysis in the present study, the impact of more invert syrup with less water and sucrose could not be evaluated to accurately evaluate the impact of inversion. It would be interesting to evaluate this impact in future studies, as well as the one by Molina-Rubio et al. (2010). The study by Molina-Rubio et al. (2010) looked at two levels of citric acid in a solution of 31.3°Bx (21% sucrose and 9% high fructose corn syrup) or 83°Bx (58.1% sucrose and 24.9% high fructose corn syrup). The pH of the 31.3°Brix samples ranged from 2.24-2.34 and the 83°Brix samples ranged from 2.09-2.18 (Molina-Rubio et al., 2010). This is within the typical range of 2-3 for paste/pulp candies. Cooking and processing temperatures used in Molina-Rubio et al. (2010) were between 88.5-

116°C and held for 17-31min. Equations published in Vukov (1965) show how the rate of sucrose hydrolysis relates to the pH, sugar concentration, salt concentration, and temperature of a solution. Calculations for acid hydrolysis on the Molina-Rubio et al. (2010) system were performed using the following equations, $\log(\mathrm{ka}) = 16.91 + \log(\mathrm{d} - \mathrm{c}) - \frac{5670}{T} - pH$ and $ka = \frac{dx}{dt} * \frac{1}{c}$, where ka is the reaction rate constant at a given concentration, c is the sucrose concentration in g/ml, d is the density in g/ml, and T is the absolute temperature in Kelvin. The results of the calculations can be seen in Table 4.1.

The general inversion rate for sucrose can be seen in Table 4.2 originally from the article "Challenges in Panning" (Gesford, 2002). In Table 4.2, the area that pulp/paste candy typically falls into is highlighted. This table illustrates that if held for an hour at typical processing conditions, 3.5% - almost 100% of sucrose would be inverted depending on the pH and temperature at which the candy was held. Based on the results from the DoE study and Tables 4.1 & 4.2 it is imperative to have consistent processing parameters and as low as possible hold times and temperatures for packaging. In addition to difficulties pumping the pulp/paste candy into the package the likelihood that the viscosity and texture of the candy will change over time could increase due to the additional inversion.

2: Overall Conclusions

Overall, this broad study showed that a model semi-liquid/pulp system could be used to examine the impact of ingredient selection on viscosity and texture. The results of this study do not support the theory that the particle size of sucrose, gum type, and corn syrup type will all impact the rheology and texture of a semi-liquid syrup model system, and by extension pulp/paste candy. ANOVA of the model generated for viscosity showed that there were interactions ($p < 0.05$) between invert syrup-water, gum-water, and sugar type-water. Sugar and gum level also had a statistically significant effect; larger additions increased the viscosity. The type of gum or the amount of corn syrup used did not appear to have an impact on the viscosity of the system (Table 4.3). The significant impact of invert syrup and sugar on soluble solids/°Bx is in agreement with several studies (Molina-Rubio et al. (2010), Cakebread (1970), Perry and Green (1998), and Quintas et al. (2005)) and provides further clarity to the conclusion from Molina-Rubio et al. (2010) that °Bx is an important factor in the determination of viscosity in a pulp/paste semi-liquid syrup model system.

The texture study provided additional treatment analysis, since it could include 4 treatments that were too high of a viscosity for the ARG-2. When the treatments were ordered in order of ascending % TS, it became apparent that there might be a tipping point at 85% TS for hardness, where the treatment becomes exponentially harder after a certain point, and then decreases around 93%TS once the solids level becomes too high. Since this study focused on breadth of samples more testing should be done with the TAX-T2 and additional treatments to verify.

The invert syrup/water interaction effects the semi-liquid pulp/paste model system in both texture (stickiness) and viscosity (Table 4.3). Targeting invert syrup-water levels and the hydration of the gum will likely have the most impact on the pulp/paste candy's viscosity and stickiness, which is important for both shelf life and primary package filling. These relationships should be explored further as they appear to significantly influence the rheology of pulp/paste candies based upon this semi-liquid pulp/paste system model.

3: Areas of Future Research

Since this was an explorative study, future research is recommended. It should focus on the area between 80-95%TS as well as exploring additional treatments with invert syrup or ingredients that create inversion/invert syrup as components (such as acid or salt). With treatments at 85-93%TS there is a likely tipping point where the hardness increases exponentially. Selling candy with solids past the tipping point could cause an increase in consumer complaints since pulp/paste candy is typically squeezed out of the package. Exponentially increased hardness of the candy would make squeezing difficult or impossible. Additional future research could look at processing conditions vs. storage conditions and include testing different sucrose hydrolysis conditions within the model semi-liquid/pulp system by using both different salt and acid conditions, to further test the impact of the interaction of invert and water on viscosity and texture, specifically stickiness. The sucrose hydrolysis conditions could also focus not only on the addition of acid or salt, but the impact of different processing parameters (i.e. held at high heat for a long time vs. low heat for a short time) with the added ingredient components.

4: References for Chapter 4

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Table 4.1 Calculated Acid Hydrolysis Rate for the Study by Molina Rubio et al. (2010)

		c		d	T			rate of hydrolysis	g/min	
acid (%)	**°Bx**	**sucrose conc (g/mL)**	**pH**	**Density**	**Temperature (K)**	**log(d-c)**	**5670/T**	**log(ka)**	**ka**	**g/min for sucrose conc.**
2.5	31.3	0.313	2.3	1.137	369.15	-0.084072788	15.35960991	-0.833682703	0.146661897	0.045905174
5	83	0.83	2.1	1.437	390.15	-0.216811309	14.53287197	0.060316719	1.148991243	0.953662732
5	31.3	0.313	2.3	1.137	369.15	-0.084072788	15.35960991	-0.833682703	0.146661897	0.045905174
2.5	83	0.83	2.1	1.437	390.15	-0.216811309	14.53287197	0.060316719	1.148991243	0.953662732

Table 4.2 Inversion Rate in Percent per Hour in Relation to pH and Temperature with Typical Pulp/Paste Candy pH and Processing Temperature Highlighted

Inversion Rate in %/hr in relation to pH and Temperature (Modified from Gesford, 2002)													
°C	**1 pH**	**1.5 pH**	**2 pH**	**2.5 pH**	**3 pH**	**3.5 pH**	**4 pH**	**4.5 pH**	**5 pH**	**5.5 pH**	**6 pH**	**6.5 pH**	**7 pH**
0	0.4	0.1	$4x10^{-2}$	$1x10^{-2}$	$4x10^{-3}$	$1x10^{-3}$	$4x10^{-4}$	$1x10^{-4}$	$4x10^{-5}$	$1x10^{-5}$	$4x10^{-6}$	$1x10^{-6}$	$4x10^{-7}$
10	1	0.3	0.1	$3x10^{-2}$	$1x10^{-2}$	$3x10^{-3}$	$1x10^{-3}$	$3x10^{-4}$	$1x10^{-4}$	$3x10^{-5}$	$1x10^{-5}$	$3x10^{-6}$	$1x10^{-6}$
20	3.5	1	0.35	0.1	$3.5x10^{-2}$	$1x10^{-2}$	$3.5x10^{-3}$	$1x10^{-3}$	$3.5x10^{-4}$	$1x10^{-4}$	$3.5x10^{-5}$	$1x10^{-5}$	$3.5x10^{-6}$
30	10	3	1	0.3	0.1	$3x10^{-2}$	$1x10^{-2}$	$3x10^{-3}$	$1x10^{-3}$	$3x10^{-4}$	$1x10^{-4}$	$3x10^{-5}$	$1x10^{-5}$
40	35	10	4	1	0.4	0.1	$4x10^{-2}$	$1x10^{-2}$	$4x10^{-3}$	$1x10^{-3}$	$4x10^{-2}$	$1x10^{-4}$	$4x10^{-5}$
50	98	30	10	3	1	0.3	0.1	$3x10^{-2}$	$1x10^{-2}$	$3x10^{-3}$	$1x10^{-3}$	$3x10^{-4}$	$1x10^{-4}$
60		98	35	10	3.5	1	0.36	0.1	$3.5x10^{-2}$	$1x10^{-2}$	$3.5x10^{-3}$	$1x10^{-3}$	$3.5x10^{-4}$
70			98	30	10	3	10	0.3	0.1	$3x10^{-2}$	$1x10^{-2}$	$3x10^{-3}$	$1x10^{-3}$
80				90	35	9	3.5	0.9	0.35	$9x10^{-2}$	$3.5x10^{-2}$	$9x10^{-3}$	$3.5x10^{-3}$
90					89	27	8.9	2.7	0.9	0.3	$9x10^{-2}$	$3x10^{-2}$	$9x10^{-3}$
100						62	21	6.2	2.1	0.62	0.21	$6.2x10^{-2}$	$2.1x10^{-2}$

Table 4.3 Summary of Model System Ingredients by Rheological Properties Impact

Model System Ingredients	Viscosity Impact	Texture Impact
Grown Sugar Powdered Sugar	Amount: yes-more sugar increases the viscosity slope Type: no unless interacting with water-grown sugar decreases viscosity slope	Amount: yes- more sugar increases hardness, especially within 85-93%TS Type: no
Invert Syrup	borderline as a main factor; significant as an interaction with water (at low frequencies more decreases viscosity, at high oscillitory frequencies more increases viscosity)	yes-with more water invert syrup increases stickiness
43/43 Corn Syrup	no	no
Deionized Water	no, unless part of sugar type, invert syrup, and gum interactions	yes- interacting with invert syrup-more water causes more stickiness
Gellan Gum Xanthan Gum	Amount: yes-interacting with water it increases the viscosity slope Type: no	Amount: no Type: yes-xanthan more sticky than gellan
Total Solids (comprised of sugar, invert syrup, and corn syrup	yes, since the main factor of amount of sugar is significant	yes-since main factor of amount of sugar effects hardness and invert syrup level effects stickiness (see above)

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Appendix A - Raw Viscosity Data

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
1.1	1	0.0026075	1.388	532.298	0.100	707.890	126.313	24.998	89.929	2.029
2.1	1	0.0356225	2.200	61.769	0.158	707.890	206.450	25.002	89.130	1.280
3.1	1	0.1234500	3.481	28.197	0.251	707.890	257.306	25.011	88.060	0.809
4.1	1	0.3301270	5.417	16.409	0.398	707.889	289.754	25.002	86.660	0.519
5.1	1	0.7373360	8.249	11.188	0.631	707.887	310.612	25.007	85.135	0.340
6.1	1	1.4294200	12.173	8.516	1.000	707.882	324.199	24.998	83.713	0.230
7.1	1	2.4732200	17.433	7.049	1.585	707.876	333.107	25.007	82.641	0.160
8.1	1	3.9377400	24.559	6.237	2.512	707.867	338.957	25.002	82.162	0.114
9.1	1	6.1659500	34.386	5.577	3.981	707.856	344.542	25.007	82.107	0.081
10.1	1	9.8761000	48.401	4.901	6.310	707.845	351.530	25.002	82.511	0.058
11.1	1	16.9270000	70.272	4.151	10.000	707.835	358.036	24.998	83.424	0.040
12.1	1	26.9450000	101.450	3.765	15.849	707.822	364.900	25.002	87.305	0.028
13.1	1	38.6584000	146.791	3.797	25.119	707.812	371.576	24.993	96.618	0.019
14.1	1	51.7451000	217.665	4.206	39.811	707.819	378.487	24.993	112.046	0.012
15.1	1	68.7612000	330.991	4.814	63.096	707.850	385.289	24.993	130.495	0.006
16.1	1	91.3104000	510.602	5.592	100.000	707.866	392.137	25.007	147.170	0.003
1.2	1	0.0089962	1.455	161.737	0.100	707.892	126.204	24.998	89.681	1.936
2.2	1	0.0475916	2.307	48.478	0.158	707.893	206.482	25.007	88.873	1.221
3.2	1	0.1465880	3.638	24.819	0.251	707.892	257.338	25.002	87.780	0.774
4.2	1	0.3751110	5.642	15.040	0.398	707.891	289.708	24.998	86.338	0.498
5.2	1	0.8107130	8.554	10.551	0.631	707.889	310.565	25.015	84.819	0.328
6.2	1	1.5458500	12.587	8.143	1.000	707.885	324.059	25.007	83.394	0.222
7.2	1	2.6108300	17.957	6.878	1.585	707.878	332.873	24.998	82.421	0.155
8.2	1	4.1487500	25.189	6.072	2.512	707.869	338.692	24.998	81.884	0.111
9.2	1	6.4468900	35.281	5.473	3.981	707.859	344.479	24.998	81.857	0.079
10.2	1	10.7024000	50.082	4.679	6.310	707.849	351.452	25.002	81.831	0.056
11.2	1	18.1276000	72.447	3.996	10.000	707.839	357.942	25.002	82.683	0.039
12.2	1	28.0004000	104.377	3.728	15.849	707.826	364.775	24.993	86.803	0.027
13.2	1	38.8355000	150.940	3.887	25.119	707.815	371.405	24.998	96.371	0.019
14.2	1	51.2653000	225.219	4.393	39.811	707.822	378.222	24.998	111.480	0.012
15.2	1	67.1491000	344.139	5.125	63.096	707.851	385.008	25.002	129.556	0.006
16.2	1	87.7152000	532.012	6.065	100.000	707.865	391.810	24.998	146.209	0.003
1.3	1	0.0127110	1.533	120.630	0.100	707.895	126.329	24.989	89.558	1.837
2.3	1	0.0592298	2.430	41.023	0.158	707.895	206.466	25.007	88.656	1.159
3.3	1	0.1725630	3.814	22.103	0.251	707.895	257.197	25.002	87.493	0.738
4.3	1	0.4235970	5.897	13.922	0.398	707.894	289.708	25.007	86.027	0.476
5.3	1	0.9008830	8.916	9.897	0.631	707.891	310.518	25.007	84.454	0.314
6.3	1	1.6758700	13.035	7.778	1.000	707.887	324.043	24.993	83.056	0.214
7.3	1	2.8006100	18.487	6.601	1.585	707.880	332.732	25.002	82.058	0.151

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
8.3	1	4.3564600	25.801	5.923	2.512	707.871	338.598	25.002	81.622	0.108
9.3	1	6.7686400	36.126	5.337	3.981	707.861	344.323	25.002	81.545	0.077
10.3	1	11.2293000	51.510	4.587	6.310	707.851	351.047	24.993	81.481	0.054
11.3	1	18.7636000	74.261	3.958	10.000	707.841	357.505	25.002	82.378	0.038
12.3	1	28.5631000	107.057	3.748	15.849	707.829	364.322	24.998	86.582	0.026
13.3	1	39.1033000	156.012	3.990	25.119	707.819	370.999	25.002	96.068	0.018
14.3	1	50.9011000	233.457	4.586	39.811	707.826	377.957	25.002	110.865	0.011
15.3	1	66.3735000	355.454	5.355	63.096	707.852	384.790	24.993	128.725	0.006
16.3	1	87.3502000	550.214	6.299	100.000	707.865	391.560	24.998	145.325	0.003
1.1	2	-0.0001219	0.023	-188.519	0.100	54.961	126.266	24.998	92.503	9.507
2.1	2	-0.0005299	0.035	-66.614	0.158	54.954	206.591	25.007	94.450	6.176
3.1	2	-0.0005900	0.058	-98.750	0.251	54.966	257.478	24.989	96.036	3.733
4.1	2	-0.0004028	0.089	-220.055	0.398	54.959	289.832	24.998	99.224	2.435
5.1	2	-0.0003570	0.135	-378.603	0.631	54.960	310.768	24.998	104.716	1.565
6.1	2	0.0023918	0.217	90.656	1.000	54.983	324.246	24.993	111.608	0.938
7.1	2	0.0094025	0.354	37.627	1.585	55.026	333.091	24.989	120.971	0.531
8.1	2	0.0364510	0.594	16.290	2.512	55.082	339.113	25.002	131.239	0.278
9.1	2	0.1096470	1.009	9.199	3.981	55.138	344.588	25.007	141.964	0.134
10.1	2	0.2929370	1.722	5.878	6.310	55.181	351.593	25.011	151.876	0.060
11.1	2	0.6541040	3.026	4.627	10.000	55.207	358.098	24.998	159.682	0.025
12.1	2	1.6895700	5.629	3.332	15.849	55.219	364.837	24.998	164.634	0.010
13.1	2	5.7206100	9.759	1.706	25.119	55.226	371.436	24.989	168.951	0.004
14.1	2	12.1271000	11.499	0.948	39.811	55.230	378.222	24.989	174.858	0.002
15.1	2	16.0183000	14.493	0.905	63.096	55.231	385.070	24.998	177.526	0.001
16.1	2	22.2009000	19.961	0.899	100.000	55.229	391.903	24.993	178.671	0.000
1.2	2	-0.0001219	0.023	-188.519	0.100	54.961	126.266	24.998	92.503	9.507
2.2	2	-0.0005299	0.035	-66.614	0.158	54.954	206.591	25.007	94.450	6.176
3.2	2	-0.0005900	0.058	-98.750	0.251	54.966	257.478	24.989	96.036	3.733
4.2	2	-0.0004028	0.089	-220.055	0.398	54.959	289.832	24.998	99.224	2.435
5.2	2	-0.0003570	0.135	-378.603	0.631	54.960	310.768	24.998	104.716	1.565
6.2	2	0.0023918	0.217	90.656	1.000	54.983	324.246	24.993	111.608	0.938
7.2	2	0.0094025	0.354	37.627	1.585	55.026	333.091	24.989	120.971	0.531
8.2	2	0.0364510	0.594	16.290	2.512	55.082	339.113	25.002	131.239	0.278
9.2	2	0.1096470	1.009	9.199	3.981	55.138	344.588	25.007	141.964	0.134
10.2	2	0.2929370	1.722	5.878	6.310	55.181	351.593	25.011	151.876	0.060
11.2	2	0.6541040	3.026	4.627	10.000	55.207	358.098	24.998	159.682	0.025
12.2	2	1.6895700	5.629	3.332	15.849	55.219	364.837	24.998	164.634	0.010
13.2	2	5.7206100	9.759	1.706	25.119	55.226	371.436	24.989	168.951	0.004
14.2	2	12.1271000	11.499	0.948	39.811	55.230	378.222	24.989	174.858	0.002
15.2	2	16.0183000	14.493	0.905	63.096	55.231	385.070	24.998	177.526	0.001
16.2	2	22.2009000	19.961	0.899	100.000	55.229	391.903	24.993	178.671	0.000

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
1.3	2	-0.0003406	0.025	-72.480	0.100	54.980	126.266	25.015	92.837	8.850
2.3	2	-0.0002112	0.040	-190.327	0.158	54.987	206.544	25.002	93.458	5.433
3.3	2	0.0000344	0.064	1854.450	0.251	54.988	257.494	25.002	94.965	3.421
4.3	2	0.0000619	0.102	1639.430	0.398	54.992	289.942	25.002	97.812	2.135
5.3	2	0.0021352	0.162	75.939	0.631	54.999	310.892	25.002	101.507	1.322
6.3	2	0.0075963	0.258	33.902	1.000	55.011	324.230	24.989	107.395	0.811
7.3	2	0.0289277	0.431	14.892	1.585	55.040	333.107	25.011	114.112	0.464
8.3	2	0.0748598	0.691	9.231	2.512	55.079	339.035	25.002	124.903	0.260
9.3	2	0.1542590	1.087	7.048	3.981	55.133	344.760	24.989	138.867	0.133
10.3	2	0.3137360	1.743	5.556	6.310	55.180	351.577	25.007	151.427	0.060
11.3	2	0.6210310	2.913	4.691	10.000	55.208	358.051	25.002	160.455	0.025
12.3	2	1.2624700	5.562	4.405	15.849	55.220	364.884	24.998	165.107	0.010
13.3	2	3.6074300	10.582	2.933	25.119	55.226	371.530	24.993	168.516	0.004
14.3	2	9.4130500	16.312	1.733	39.811	55.229	378.487	24.993	172.875	0.002
15.3	2	14.7147000	20.277	1.378	63.096	55.230	385.351	24.998	176.554	0.001
16.3	2	28.5654000	26.155	0.916	100.000	55.229	392.215	24.989	178.246	0.000
1.1	3	0.0578856	2.443	42.202	0.100	1504.730	126.329	25.011	88.663	2.450
2.1	3	0.1344310	3.784	28.149	0.158	1504.730	206.513	25.015	87.999	1.581
3.1	3	0.2937070	5.889	20.051	0.251	1504.730	257.369	25.011	87.199	1.015
4.1	3	0.5841980	9.006	15.416	0.398	1504.730	289.723	25.015	86.377	0.663
5.1	3	1.0407200	13.537	13.008	0.631	1504.720	310.596	24.985	85.751	0.441
6.1	3	1.6211500	19.891	12.270	1.000	1504.720	324.121	25.002	85.593	0.300
7.1	3	2.1821200	28.900	13.244	1.585	1504.710	333.044	25.002	86.119	0.207
8.1	3	2.7416000	42.549	15.520	2.512	1504.700	338.863	25.002	87.060	0.141
9.1	3	3.6194500	64.286	17.761	3.981	1504.700	344.557	24.989	88.020	0.093
10.1	3	5.6270700	99.387	17.662	6.310	1504.690	351.499	24.998	88.780	0.060
11.1	3	9.1919600	155.698	16.938	10.000	1504.690	357.926	25.007	89.862	0.038
12.1	3	14.9093000	244.916	16.427	15.849	1504.690	364.712	25.007	91.694	0.024
13.1	3	22.6469000	380.053	16.782	25.119	1504.690	371.311	24.993	94.962	0.016
14.1	3	35.3776000	594.085	16.793	39.811	1504.690	378.144	25.002	99.966	0.010
15.1	3	53.9149000	932.396	17.294	63.096	1504.680	385.039	25.011	107.679	0.006
16.1	3	79.6553000	1461.820	18.352	100.000	1504.640	391.919	24.993	118.760	0.004
1.2	3	0.0641687	2.529	39.408	0.100	1504.730	126.360	24.989	88.566	2.367
2.2	3	0.1494780	3.988	26.678	0.158	1504.730	206.466	24.993	87.885	1.500
3.2	3	0.3197910	6.230	19.483	0.251	1504.730	257.228	24.998	87.113	0.960
4.2	3	0.6205500	9.525	15.350	0.398	1504.730	289.676	24.998	86.356	0.627
5.2	3	1.0677700	14.229	13.325	0.631	1504.730	310.549	24.998	85.849	0.420
6.2	3	1.6104200	21.001	13.041	1.000	1504.720	324.121	25.002	85.854	0.284
7.2	3	2.1303400	30.582	14.355	1.585	1504.710	333.013	25.007	86.428	0.195
8.2	3	2.6770900	45.511	17.000	2.512	1504.710	338.863	25.007	87.332	0.131
9.2	3	3.7448300	69.536	18.569	3.981	1504.700	344.588	25.007	88.066	0.086

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
10.2	3	5.9442700	107.604	18.102	6.310	1504.700	351.437	25.007	88.704	0.056
11.2	3	9.7734700	169.290	17.321	10.000	1504.700	357.848	25.007	89.676	0.035
12.2	3	15.8440000	264.078	16.667	15.849	1504.700	364.681	24.993	91.367	0.023
13.2	3	24.1709000	408.768	16.912	25.119	1504.700	371.374	25.002	94.401	0.015
14.2	3	38.0094000	637.534	16.773	39.811	1504.690	378.222	25.007	99.067	0.009
15.2	3	58.4591000	999.509	17.098	63.096	1504.680	385.008	25.002	106.316	0.006
16.2	3	86.8902000	1566.340	18.027	100.000	1504.640	391.810	24.993	116.909	0.003
1.3	3	0.0728412	2.658	36.488	0.100	1504.740	126.313	24.993	88.449	2.252
2.3	3	0.1592440	4.143	26.017	0.158	1504.740	206.513	24.993	87.829	1.444
3.3	3	0.3295730	6.433	19.518	0.251	1504.740	257.353	24.993	87.116	0.930
4.3	3	0.6476300	9.856	15.218	0.398	1504.730	289.661	25.002	86.321	0.606
5.3	3	1.0958500	14.716	13.429	0.631	1504.730	310.471	25.007	85.877	0.406
6.3	3	1.6253800	21.525	13.243	1.000	1504.720	323.996	24.998	85.915	0.277
7.3	3	2.1213000	31.477	14.839	1.585	1504.720	332.795	25.007	86.546	0.190
8.3	3	2.6066700	46.906	17.995	2.512	1504.710	338.723	24.985	87.497	0.128
9.3	3	3.6565800	71.856	19.651	3.981	1504.710	344.464	25.011	88.199	0.083
10.3	3	5.8518800	112.061	19.150	6.310	1504.710	351.390	24.989	88.802	0.053
11.3	3	9.5649900	175.919	18.392	10.000	1504.710	357.911	24.998	89.756	0.034
12.3	3	15.5118000	277.228	17.872	15.849	1504.700	364.697	25.015	91.370	0.022
13.3	3	22.8295000	429.833	18.828	25.119	1504.700	371.264	24.989	94.363	0.014
14.3	3	36.5112000	672.845	18.429	39.811	1504.700	378.082	25.002	98.722	0.009
15.3	3	56.3220000	1057.990	18.785	63.096	1504.690	384.977	25.011	105.564	0.005
16.3	3	85.1269000	1660.450	19.506	100.000	1504.640	391.794	24.993	115.629	0.003
1.1	4	0.2846480	2.527	8.878	0.100	718.785	126.329	24.985	83.593	1.125
2.1	4	0.6185810	3.857	6.236	0.158	718.785	206.482	24.993	80.921	0.732
3.1	4	1.1416100	5.561	4.871	0.251	718.782	257.384	25.011	78.455	0.504
4.1	4	1.8128100	7.648	4.219	0.398	718.777	289.676	24.989	76.765	0.364
5.1	4	2.5352600	10.164	4.009	0.631	718.770	310.424	24.989	76.181	0.273
6.1	4	3.2345100	13.320	4.118	1.000	718.759	323.918	24.998	76.710	0.209
7.1	4	3.8075300	17.678	4.643	1.585	718.747	332.748	24.985	78.533	0.159
8.1	4	4.2178600	23.829	5.649	2.512	718.733	338.614	25.007	81.265	0.119
9.1	4	4.6693400	33.656	7.208	3.981	718.722	344.323	25.011	84.449	0.085
10.1	4	5.4956200	49.755	9.054	6.310	718.714	351.250	24.998	87.719	0.057
11.1	4	7.1711100	75.931	10.588	10.000	718.710	357.755	25.011	91.248	0.038
12.1	4	9.8487400	118.588	12.041	15.849	718.709	364.619	24.985	95.931	0.024
13.1	4	13.3475000	183.294	13.733	25.119	718.711	371.140	25.002	103.006	0.015
14.1	4	18.7032000	285.314	15.255	39.811	718.719	377.957	25.015	113.011	0.009
15.1	4	26.8363000	449.364	16.745	63.096	718.733	384.961	24.980	125.836	0.005
16.1	4	36.4063000	710.315	19.511	100.000	718.733	391.747	24.998	139.988	0.003
1.2	4	0.2846480	2.527	8.878	0.100	718.785	126.329	24.985	83.593	1.125
2.2	4	0.6185810	3.857	6.236	0.158	718.785	206.482	24.993	80.921	0.732

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
3.2	4	1.1416100	5.561	4.871	0.251	718.782	257.384	25.011	78.455	0.504
4.2	4	1.8128100	7.648	4.219	0.398	718.777	289.676	24.989	76.765	0.364
5.2	4	2.5352600	10.164	4.009	0.631	718.770	310.424	24.989	76.181	0.273
6.2	4	3.2345100	13.320	4.118	1.000	718.759	323.918	24.998	76.710	0.209
7.2	4	3.8075300	17.678	4.643	1.585	718.747	332.748	24.985	78.533	0.159
8.2	4	4.2178600	23.829	5.649	2.512	718.733	338.614	25.007	81.265	0.119
9.2	4	4.6693400	33.656	7.208	3.981	718.722	344.323	25.011	84.449	0.085
10.2	4	5.4956200	49.755	9.054	6.310	718.714	351.250	24.998	87.719	0.057
11.2	4	7.1711100	75.931	10.588	10.000	718.710	357.755	25.011	91.248	0.038
12.2	4	9.8487400	118.588	12.041	15.849	718.709	364.619	24.985	95.931	0.024
13.2	4	13.3475000	183.294	13.733	25.119	718.711	371.140	25.002	103.006	0.015
14.2	4	18.7032000	285.314	15.255	39.811	718.719	377.957	25.015	113.011	0.009
15.2	4	26.8363000	449.364	16.745	63.096	718.733	384.961	24.980	125.836	0.005
16.2	4	36.4063000	710.315	19.511	100.000	718.733	391.747	24.998	139.988	0.003
1.3	4	0.2846480	2.527	8.878	0.100	718.785	126.329	24.985	83.593	1.125
2.3	4	0.6185810	3.857	6.236	0.158	718.785	206.482	24.993	80.921	0.732
3.3	4	1.1416100	5.561	4.871	0.251	718.782	257.384	25.011	78.455	0.504
4.3	4	1.8128100	7.648	4.219	0.398	718.777	289.676	24.989	76.765	0.364
5.3	4	2.5352600	10.164	4.009	0.631	718.770	310.424	24.989	76.181	0.273
6.3	4	3.2345100	13.320	4.118	1.000	718.759	323.918	24.998	76.710	0.209
7.3	4	3.8075300	17.678	4.643	1.585	718.747	332.748	24.985	78.533	0.159
8.3	4	4.2178600	23.829	5.649	2.512	718.733	338.614	25.007	81.265	0.119
9.3	4	4.6693400	33.656	7.208	3.981	718.722	344.323	25.011	84.449	0.085
10.3	4	5.4956200	49.755	9.054	6.310	718.714	351.250	24.998	87.719	0.057
11.3	4	7.1711100	75.931	10.588	10.000	718.710	357.755	25.011	91.248	0.038
12.3	4	9.8487400	118.588	12.041	15.849	718.709	364.619	24.985	95.931	0.024
13.3	4	13.3475000	183.294	13.733	25.119	718.711	371.140	25.002	103.006	0.015
14.3	4	18.7032000	285.314	15.255	39.811	718.719	377.957	25.015	113.011	0.009
15.3	4	26.8363000	449.364	16.745	63.096	718.733	384.961	24.980	125.836	0.005
16.3	4	36.4063000	710.315	19.511	100.000	718.733	391.747	24.998	139.988	0.003
1.1	5	578970.0000000	616711.000	1.065	0.100	2270.670	126.532	25.011	36.272	0.000
2.1	5	841955.0000000	736892.000	0.875	0.158	2270.670	207.433	24.989	29.546	0.000
3.1	5	1083610.0000000	863755.000	0.797	0.251	2270.670	259.178	24.998	25.799	0.000
4.1	5	1414120.0000000	979427.000	0.693	0.398	2270.670	292.406	25.002	21.407	0.000
5.1	5	1657790.0000000	1181890.000	0.713	0.631	2270.670	313.903	24.976	20.405	0.000
6.1	5	2035890.0000000	1446850.000	0.711	1.000	2270.670	328.224	25.011	18.505	0.000
7.1	5	2587680.0000000	1546230.000	0.598	1.585	2270.670	337.865	25.011	14.639	0.000
8.1	5	3008390.0000000	1865210.000	0.620	2.512	2270.670	344.573	24.989	13.784	0.000
9.1	5	3680330.0000000	2115010.000	0.575	3.981	2270.670	350.891	24.989	11.617	0.000
10.1	5	4301210.0000000	2219390.000	0.516	6.310	2270.670	358.457	25.011	9.767	0.000
11.1	5	4925320.0000000	2244130.000	0.456	10.000	2270.670	365.867	25.020	8.150	0.000

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
12.1	5	5546040.0000000	2362660.000	0.426	15.849	2270.670	373.542	24.993	7.134	0.000
13.1	5	6342320.0000000	2026130.000	0.319	25.119	2270.670	380.999	24.980	5.122	0.000
14.1	5	6855260.0000000	2023090.000	0.295	39.811	2270.670	388.658	25.002	4.519	0.000
15.1	5	7454250.0000000	1840790.000	0.247	63.096	2270.650	396.318	25.020	3.622	0.000
16.1	5	7788220.0000000	1718090.000	0.221	100.000	2270.560	403.946	24.993	3.160	0.000
1.2	5	578970.0000000	616711.000	1.065	0.100	2270.670	126.532	25.011	36.272	0.000
2.2	5	841955.0000000	736892.000	0.875	0.158	2270.670	207.433	24.989	29.546	0.000
3.2	5	1083610.0000000	863755.000	0.797	0.251	2270.670	259.178	24.998	25.799	0.000
4.2	5	1414120.0000000	979427.000	0.693	0.398	2270.670	292.406	25.002	21.407	0.000
5.2	5	1657790.0000000	1181890.000	0.713	0.631	2270.670	313.903	24.976	20.405	0.000
6.2	5	2035890.0000000	1446850.000	0.711	1.000	2270.670	328.224	25.011	18.505	0.000
7.2	5	2587680.0000000	1546230.000	0.598	1.585	2270.670	337.865	25.011	14.639	0.000
8.2	5	3008390.0000000	1865210.000	0.620	2.512	2270.670	344.573	24.989	13.784	0.000
9.2	5	3680330.0000000	2115010.000	0.575	3.981	2270.670	350.891	24.989	11.617	0.000
10.2	5	4301210.0000000	2219390.000	0.516	6.310	2270.670	358.457	25.011	9.767	0.000
11.2	5	4925320.0000000	2244130.000	0.456	10.000	2270.670	365.867	25.020	8.150	0.000
12.2	5	5546040.0000000	2362660.000	0.426	15.849	2270.670	373.542	24.993	7.134	0.000
13.2	5	6342320.0000000	2026130.000	0.319	25.119	2270.670	380.999	24.980	5.122	0.000
14.2	5	6855260.0000000	2023090.000	0.295	39.811	2270.670	388.658	25.002	4.519	0.000
15.2	5	7454250.0000000	1840790.000	0.247	63.096	2270.650	396.318	25.020	3.622	0.000
16.2	5	7788220.0000000	1718090.000	0.221	100.000	2270.560	403.946	24.993	3.160	0.000
1.3	5	578970.0000000	616711.000	1.065	0.100	2270.670	126.532	25.011	36.272	0.000
2.3	5	841955.0000000	736892.000	0.875	0.158	2270.670	207.433	24.989	29.546	0.000
3.3	5	1083610.0000000	863755.000	0.797	0.251	2270.670	259.178	24.998	25.799	0.000
4.3	5	1414120.0000000	979427.000	0.693	0.398	2270.670	292.406	25.002	21.407	0.000
5.3	5	1657790.0000000	1181890.000	0.713	0.631	2270.670	313.903	24.976	20.405	0.000
6.3	5	2035890.0000000	1446850.000	0.711	1.000	2270.670	328.224	25.011	18.505	0.000
7.3	5	2587680.0000000	1546230.000	0.598	1.585	2270.670	337.865	25.011	14.639	0.000
8.3	5	3008390.0000000	1865210.000	0.620	2.512	2270.670	344.573	24.989	13.784	0.000
9.3	5	3680330.0000000	2115010.000	0.575	3.981	2270.670	350.891	24.989	11.617	0.000
10.3	5	4301210.0000000	2219390.000	0.516	6.310	2270.670	358.457	25.011	9.767	0.000
11.3	5	4925320.0000000	2244130.000	0.456	10.000	2270.670	365.867	25.020	8.150	0.000
12.3	5	5546040.0000000	2362660.000	0.426	15.849	2270.670	373.542	24.993	7.134	0.000
13.3	5	6342320.0000000	2026130.000	0.319	25.119	2270.670	380.999	24.980	5.122	0.000
14.3	5	6855260.0000000	2023090.000	0.295	39.811	2270.670	388.658	25.002	4.519	0.000
15.3	5	7454250.0000000	1840790.000	0.247	63.096	2270.650	396.318	25.020	3.622	0.000
16.3	5	7788220.0000000	1718090.000	0.221	100.000	2270.560	403.946	24.993	3.160	0.000
1.1	6	0.0468656	2.198	46.909	0.100	1425.220	126.235	25.020	88.802	2.579
2.1	6	0.0945248	3.468	36.688	0.158	1425.220	206.497	25.007	88.475	1.635
3.1	6	0.2096610	5.451	26.001	0.251	1425.220	257.322	24.993	87.856	1.040
4.1	6	0.4141090	8.438	20.377	0.398	1425.220	289.723	25.002	87.285	0.671

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
5.1	6	0.7582020	12.825	16.915	0.631	1425.220	310.518	24.985	86.773	0.441
6.1	6	1.2624100	19.042	15.084	1.000	1425.210	324.059	25.011	86.471	0.297
7.1	6	1.7526100	28.055	16.007	1.585	1425.210	332.826	25.011	86.876	0.202
8.1	6	2.3168500	42.367	18.287	2.512	1425.200	338.832	24.993	87.620	0.134
9.1	6	3.3614700	65.463	19.475	3.981	1425.200	344.526	24.985	88.282	0.087
10.1	6	5.2992700	102.416	19.327	6.310	1425.200	351.437	24.998	88.999	0.055
11.1	6	8.7068200	163.649	18.796	10.000	1425.200	358.176	25.020	90.039	0.035
12.1	6	13.9718000	260.083	18.615	15.849	1425.200	364.993	25.007	91.801	0.022
13.1	6	21.9415000	411.543	18.756	25.119	1425.200	371.701	24.993	94.680	0.014
14.1	6	33.9173000	640.995	18.899	39.811	1425.190	378.628	24.985	99.375	0.009
15.1	6	54.7788000	1017.230	18.570	63.096	1425.190	385.445	25.011	106.237	0.005
16.1	6	87.8863000	1597.240	18.174	100.000	1425.140	392.122	25.015	116.430	0.003
1.2	6	0.0448642	2.344	52.255	0.100	1425.230	126.313	24.989	88.925	2.418
2.2	6	0.0995496	3.713	37.297	0.158	1425.230	206.591	24.998	88.498	1.527
3.2	6	0.1992250	5.828	29.253	0.251	1425.230	257.478	24.989	88.097	0.972
4.2	6	0.4071110	9.044	22.216	0.398	1425.230	289.942	25.007	87.511	0.626
5.2	6	0.7950290	13.636	17.151	0.631	1425.220	310.783	24.998	86.810	0.415
6.2	6	1.3036900	20.228	15.516	1.000	1425.220	324.215	24.989	86.561	0.280
7.2	6	1.9053100	30.281	15.893	1.585	1425.210	333.060	25.007	86.817	0.187
8.2	6	2.4533400	45.387	18.500	2.512	1425.210	338.879	25.015	87.606	0.125
9.2	6	3.8547300	71.089	18.442	3.981	1425.210	344.464	25.011	88.020	0.080
10.2	6	6.1188300	113.108	18.485	6.310	1425.210	351.452	25.007	88.678	0.050
11.2	6	10.1207000	182.196	18.002	10.000	1425.210	357.942	25.002	89.589	0.031
12.2	6	16.1930000	288.763	17.833	15.849	1425.210	364.900	25.002	91.180	0.020
13.2	6	25.1723000	452.129	17.961	25.119	1425.210	371.530	24.998	93.852	0.013
14.2	6	39.5125000	703.429	17.803	39.811	1425.200	378.394	24.993	98.107	0.008
15.2	6	66.2337000	1118.620	16.889	63.096	1425.190	385.211	24.993	104.280	0.005
16.2	6	106.7710000	1757.970	16.465	100.000	1425.150	392.028	24.993	113.786	0.003
1.3	6	0.0438398	2.493	56.867	0.100	1425.230	126.329	25.015	89.013	2.274
2.3	6	0.1130180	3.917	34.656	0.158	1425.230	206.560	24.980	88.380	1.447
3.3	6	0.2467390	6.151	24.930	0.251	1425.230	257.384	24.985	87.755	0.921
4.3	6	0.4536200	9.451	20.834	0.398	1425.230	289.848	24.993	87.337	0.599
5.3	6	0.8014810	14.376	17.937	0.631	1425.230	310.721	25.011	86.948	0.394
6.3	6	1.3816900	21.386	15.478	1.000	1425.220	324.262	24.989	86.539	0.265
7.3	6	1.9938000	31.808	15.954	1.585	1425.220	333.122	25.011	86.811	0.178
8.3	6	2.6956500	47.604	17.660	2.512	1425.210	339.004	25.024	87.427	0.119
9.3	6	3.7899300	73.919	19.504	3.981	1425.210	344.620	25.002	88.146	0.077
10.3	6	6.5102800	120.342	18.485	6.310	1425.210	351.530	24.985	88.571	0.047
11.3	6	10.7107000	193.032	18.022	10.000	1425.210	357.942	24.998	89.437	0.029
12.3	6	17.5648000	308.304	17.552	15.849	1425.210	364.806	25.011	90.850	0.018
13.3	6	27.9719000	485.019	17.340	25.119	1425.210	371.389	25.015	93.261	0.012

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
14.3	6	42.9034000	745.658	17.380	39.811	1425.210	378.269	24.989	97.396	0.008
15.3	6	76.0925000	1199.640	15.766	63.096	1425.200	385.180	24.985	102.901	0.005
16.3	6	134.7310000	1907.920	14.161	100.000	1425.150	391.997	25.011	111.373	0.003
1.1	9	0.0000667	0.031	468.735	0.100	575.584	126.313	25.020	91.496	73.252
2.1	9	-0.0008032	0.044	-54.730	0.158	575.330	206.544	25.002	93.931	51.949
3.1	9	-0.0021097	0.067	-31.605	0.251	575.246	257.462	24.980	96.570	34.102
4.1	9	-0.0058578	0.104	-17.734	0.398	575.259	289.832	24.998	100.817	21.643
5.1	9	-0.0119274	0.165	-13.869	0.631	575.368	310.814	25.007	105.884	13.311
6.1	9	-0.0167133	0.273	-16.361	1.000	575.590	324.152	25.011	111.004	7.819
7.1	9	-0.0337652	0.468	-13.859	1.585	575.967	333.076	24.993	118.636	4.298
8.1	9	-0.0417394	0.835	-20.015	2.512	576.373	339.035	24.985	125.628	2.231
9.1	9	-0.0605761	1.550	-25.592	3.981	576.781	344.776	24.985	133.275	1.078
10.1	9	0.0301186	2.667	88.559	6.310	577.099	351.624	25.011	142.565	0.523
11.1	9	0.3568410	4.910	13.760	10.000	577.336	358.129	25.011	149.901	0.235
12.1	9	1.1220500	10.089	8.991	15.849	577.480	364.962	25.011	154.395	0.098
13.1	9	2.8432800	21.875	7.693	25.119	577.562	371.654	24.993	157.517	0.040
14.1	9	10.3914000	50.820	4.891	39.811	577.602	378.472	24.989	158.575	0.017
15.1	9	53.3771000	109.855	2.058	63.096	577.620	385.367	24.980	159.768	0.007
16.1	9	176.1390000	137.795	0.782	100.000	577.626	392.168	25.011	168.967	0.003
1.2	9	-0.0002316	0.026	-113.494	0.100	575.194	126.313	24.985	92.427	86.981
2.2	9	-0.0007671	0.039	-51.459	0.158	575.068	206.528	25.007	94.324	57.800
3.2	9	-0.0022492	0.062	-27.634	0.251	575.076	257.525	25.002	97.170	36.526
4.2	9	-0.0052655	0.099	-18.725	0.398	575.134	289.864	24.971	101.051	22.779
5.2	9	-0.0098626	0.161	-16.284	0.631	575.294	310.658	25.024	105.655	13.724
6.2	9	-0.0181418	0.265	-14.594	1.000	575.548	324.215	24.993	111.897	8.026
7.2	9	-0.0229745	0.443	-19.273	1.585	575.880	333.247	24.980	118.927	4.529
8.2	9	-0.0332943	0.808	-24.280	2.512	576.346	339.175	24.985	126.136	2.291
9.2	9	-0.0462538	1.408	-30.433	3.981	576.771	344.869	25.015	135.756	1.137
10.2	9	0.0121368	2.523	207.868	6.310	577.112	351.718	25.020	144.232	0.532
11.2	9	0.3342510	4.734	14.164	10.000	577.344	358.270	25.002	150.865	0.236
12.2	9	1.1839300	9.769	8.251	15.849	577.483	365.134	24.976	155.043	0.099
13.2	9	2.8597000	21.010	7.347	25.119	577.565	371.826	24.985	158.316	0.040
14.2	9	9.3835400	48.186	5.135	39.811	577.605	378.862	24.993	159.737	0.017
15.2	9	46.7927000	105.588	2.257	63.096	577.623	385.804	25.002	160.884	0.007
16.2	9	167.8290000	140.402	0.837	100.000	577.626	392.434	25.002	168.891	0.003
1.3	9	-0.0001670	0.025	-152.064	0.100	575.108	126.220	24.989	92.367	90.011
2.3	9	-0.0007984	0.040	-49.819	0.158	575.087	206.575	25.011	94.336	57.360
3.3	9	-0.0021271	0.064	-29.923	0.251	575.134	257.431	25.002	96.895	35.693
4.3	9	-0.0048861	0.102	-20.779	0.398	575.199	289.848	25.007	100.532	22.162
5.3	9	-0.0101865	0.166	-16.290	0.631	575.362	310.705	24.980	105.279	13.309
6.3	9	-0.0218331	0.275	-12.598	1.000	575.625	324.121	25.007	111.816	7.730

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
7.3	9	-0.0272310	0.457	-16.799	1.585	575.925	332.998	25.011	118.558	4.400
8.3	9	-0.0462740	0.770	-16.650	2.512	576.350	338.957	25.007	128.060	2.344
9.3	9	-0.0484123	1.369	-28.284	3.981	576.773	344.588	24.985	136.590	1.152
10.3	9	-0.0303203	2.771	-91.387	6.310	577.106	351.562	24.993	141.985	0.510
11.3	9	0.3085980	5.864	19.002	10.000	577.313	358.176	24.998	145.459	0.222
12.3	9	1.2014900	12.620	10.503	15.849	577.453	364.962	25.007	148.964	0.094
13.3	9	3.2987900	27.082	8.210	25.119	577.544	371.686	25.011	152.668	0.039
14.3	9	12.8936000	61.484	4.769	39.811	577.590	378.581	25.020	154.169	0.016
15.3	9	64.0877000	128.154	2.000	63.096	577.611	385.476	24.993	155.964	0.007
16.3	9	209.0800000	153.220	0.733	100.000	577.622	392.278	24.980	167.189	0.003
1.1	10	0.1117310	0.683	6.115	0.100	156.889	126.329	24.998	80.785	0.902
2.1	10	0.1862550	0.930	4.992	0.158	156.885	206.575	24.989	78.804	0.659
3.1	10	0.3119380	1.289	4.131	0.251	156.881	257.478	24.998	76.627	0.471
4.1	10	0.4674060	1.731	3.704	0.398	156.876	289.942	24.998	75.325	0.349
5.1	10	0.6542770	2.298	3.512	0.631	156.868	310.783	25.020	74.920	0.262
6.1	10	0.8784630	3.049	3.471	1.000	156.859	324.293	25.015	75.471	0.198
7.1	10	1.1567400	4.090	3.536	1.585	156.848	333.216	25.011	77.120	0.149
8.1	10	1.5422000	5.610	3.638	2.512	156.837	339.082	25.007	80.034	0.110
9.1	10	2.0893800	7.846	3.755	3.981	156.824	344.698	25.007	84.962	0.079
10.1	10	2.8556700	11.154	3.906	6.310	156.814	351.624	25.007	93.361	0.056
11.1	10	3.9652200	16.223	4.091	10.000	156.813	358.238	25.007	106.655	0.037
12.1	10	5.5913200	24.077	4.306	15.849	156.834	365.024	24.998	124.520	0.021
13.1	10	7.7947800	36.196	4.644	25.119	156.869	371.717	24.993	142.892	0.010
14.1	10	10.6836000	55.388	5.184	39.811	156.894	378.456	24.993	156.776	0.004
15.1	10	14.7947000	84.996	5.745	63.096	156.904	385.398	24.998	165.815	0.002
16.1	10	19.2044000	130.967	6.820	100.000	156.903	392.200	24.998	171.367	0.001
1.2	10	0.1335030	0.740	5.545	0.100	156.891	126.235	24.998	79.843	0.830
2.2	10	0.2400060	1.048	4.366	0.158	156.888	206.450	25.002	77.214	0.581
3.2	10	0.3793840	1.431	3.771	0.251	156.885	257.353	25.002	75.357	0.422
4.2	10	0.5434750	1.900	3.497	0.398	156.879	289.739	24.998	74.431	0.316
5.2	10	0.7338370	2.501	3.408	0.631	156.872	310.612	24.985	74.388	0.240
6.2	10	0.9601560	3.297	3.434	1.000	156.864	324.074	25.002	75.188	0.183
7.2	10	1.2495300	4.434	3.548	1.585	156.854	332.826	24.993	76.946	0.137
8.2	10	1.6411300	6.070	3.699	2.512	156.843	338.676	25.002	79.869	0.101
9.2	10	2.2170800	8.554	3.858	3.981	156.832	344.417	24.989	84.528	0.073
10.2	10	3.0693900	12.290	4.004	6.310	156.823	351.437	25.007	92.056	0.051
11.2	10	4.2729600	18.028	4.219	10.000	156.821	357.942	24.989	104.152	0.034
12.2	10	5.9904400	26.759	4.467	15.849	156.837	364.744	24.993	121.130	0.020
13.2	10	8.3279200	40.451	4.857	25.119	156.867	371.342	24.998	139.471	0.010
14.2	10	11.3727000	61.948	5.447	39.811	156.892	378.097	24.989	154.243	0.004
15.2	10	15.7730000	95.873	6.078	63.096	156.903	385.008	25.007	164.043	0.002

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
16.2	10	23.3925000	148.454	6.346	100.000	156.903	391.685	25.015	170.189	0.001
1.3	10	0.1843500	0.862	4.677	0.100	156.894	126.173	24.998	77.988	0.708
2.3	10	0.3054990	1.181	3.864	0.158	156.892	206.419	25.002	75.592	0.512
3.3	10	0.4545370	1.577	3.470	0.251	156.888	257.260	25.002	74.113	0.381
4.3	10	0.6280460	2.077	3.307	0.398	156.883	289.708	25.011	73.528	0.288
5.3	10	0.8243560	2.700	3.276	0.631	156.876	310.424	25.002	73.707	0.222
6.3	10	1.0492800	3.532	3.367	1.000	156.867	324.059	25.011	74.779	0.170
7.3	10	1.3426700	4.752	3.539	1.585	156.858	332.904	24.998	76.725	0.128
8.3	10	1.7491500	6.536	3.737	2.512	156.848	338.957	25.011	79.658	0.094
9.3	10	2.3734700	9.227	3.887	3.981	156.838	344.651	25.011	83.963	0.067
10.3	10	3.2604200	13.258	4.066	6.310	156.829	351.608	25.002	91.081	0.047
11.3	10	4.5536400	19.642	4.313	10.000	156.827	358.051	25.015	102.251	0.031
12.3	10	6.3492700	29.131	4.588	15.849	156.839	364.900	24.993	118.478	0.019
13.3	10	8.8060600	44.103	5.008	25.119	156.866	371.530	25.007	136.720	0.010
14.3	10	11.9757000	67.637	5.648	39.811	156.891	378.347	24.998	152.108	0.004
15.3	10	16.8627000	104.390	6.191	63.096	156.902	385.180	24.998	162.654	0.002
16.3	10	23.1764000	161.682	6.976	100.000	156.902	392.059	25.007	169.337	0.001
1.1	11	0.1256070	1.377	10.962	0.100	470.198	126.204	25.002	84.824	1.353
2.1	11	0.2829820	2.113	7.468	0.158	470.198	206.482	25.002	82.432	0.878
3.1	11	0.5757930	3.143	5.459	0.251	470.196	257.322	25.002	79.717	0.586
4.1	11	1.0300600	4.454	4.324	0.398	470.192	289.754	25.007	77.149	0.410
5.1	11	1.6080800	6.037	3.754	0.631	470.185	310.565	25.007	75.397	0.300
6.1	11	2.2397800	7.945	3.547	1.000	470.174	324.012	25.002	74.847	0.227
7.1	11	2.8604600	10.317	3.607	1.585	470.160	332.873	25.002	75.652	0.176
8.1	11	3.4334700	13.451	3.918	2.512	470.142	338.848	25.002	77.927	0.136
9.1	11	3.9968400	17.958	4.493	3.981	470.121	344.448	25.002	81.764	0.103
10.1	11	4.5287400	24.595	5.431	6.310	470.100	351.390	25.002	87.629	0.076
11.1	11	5.5138300	35.555	6.448	10.000	470.088	357.926	24.998	95.310	0.052
12.1	11	7.1691900	53.028	7.397	15.849	470.089	364.868	25.007	105.776	0.034
13.1	11	9.5972900	80.131	8.349	25.119	470.111	371.561	25.002	119.881	0.020
14.1	11	13.5607000	123.574	9.113	39.811	470.151	378.394	25.002	135.602	0.011
15.1	11	19.1389000	191.290	9.995	63.096	470.187	385.164	24.998	150.045	0.005
16.1	11	26.8616000	299.308	11.143	100.000	470.194	391.997	24.993	160.706	0.002
1.2	11	0.1554500	1.482	9.533	0.100	470.201	126.391	25.007	84.045	1.256
2.2	11	0.3335570	2.255	6.759	0.158	470.200	206.560	24.998	81.640	0.821
3.2	11	0.6551090	3.305	5.045	0.251	470.198	257.338	25.002	78.881	0.555
4.2	11	1.1320600	4.621	4.082	0.398	470.193	289.676	24.993	76.399	0.393
5.2	11	1.7147000	6.191	3.611	0.631	470.186	310.346	24.998	74.822	0.292
6.2	11	2.3300100	8.082	3.469	1.000	470.176	323.872	25.007	74.497	0.223
7.2	11	2.9242300	10.456	3.576	1.585	470.161	332.701	24.998	75.507	0.173
8.2	11	3.4576600	13.617	3.938	2.512	470.143	338.582	24.993	77.972	0.134

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
9.2	11	3.9419300	18.154	4.605	3.981	470.122	344.417	24.998	82.021	0.102
10.2	11	4.4335600	24.928	5.623	6.310	470.102	351.374	25.007	87.879	0.075
11.2	11	5.3165300	36.067	6.784	10.000	470.090	357.786	25.002	95.545	0.052
12.2	11	6.8734000	53.881	7.839	15.849	470.092	364.697	25.007	105.830	0.033
13.2	11	9.2251800	81.596	8.845	25.119	470.112	371.405	25.002	119.632	0.020
14.2	11	12.9993000	126.054	9.697	39.811	470.152	378.206	24.993	135.160	0.010
15.2	11	18.6945000	195.076	10.435	63.096	470.187	384.992	25.002	149.590	0.005
16.2	11	18.6222000	308.489	16.566	100.000	470.194	391.778	25.007	160.334	0.002
1.3	11	0.1788090	1.569	8.775	0.100	470.203	126.329	25.002	83.531	1.185
2.3	11	0.3749900	2.365	6.307	0.158	470.202	206.497	24.993	81.042	0.781
3.3	11	0.7159560	3.433	4.795	0.251	470.199	257.275	25.002	78.308	0.534
4.3	11	1.2103800	4.756	3.930	0.398	470.195	289.801	24.998	75.881	0.381
5.3	11	1.8002300	6.335	3.519	0.631	470.188	310.549	25.011	74.430	0.284
6.3	11	2.4119000	8.235	3.414	1.000	470.177	324.137	24.998	74.242	0.219
7.3	11	2.9931400	10.616	3.547	1.585	470.162	333.060	24.998	75.367	0.171
8.3	11	3.5059200	13.824	3.943	2.512	470.144	338.848	24.993	77.956	0.132
9.3	11	3.9219800	18.394	4.690	3.981	470.124	344.604	25.002	82.185	0.101
10.3	11	4.3653600	25.311	5.798	6.310	470.104	351.562	24.998	88.066	0.074
11.3	11	5.2261400	36.750	7.032	10.000	470.093	358.020	24.998	95.583	0.051
12.3	11	6.7804500	55.137	8.132	15.849	470.095	364.806	24.998	105.576	0.033
13.3	11	9.1340000	83.581	9.151	25.119	470.114	371.405	24.998	119.092	0.020
14.3	11	12.8440000	129.025	10.046	39.811	470.152	378.253	24.998	134.528	0.010
15.3	11	17.8749000	200.778	11.232	63.096	470.186	385.180	25.011	148.927	0.005
16.3	11	25.2482000	314.178	12.444	100.000	470.193	392.044	24.993	159.858	0.002
1.1	12	699.6620000	1076.340	1.538	0.100	20299.300	126.313	25.002	56.950	0.063
2.1	12	890.0550000	1196.000	1.344	0.158	20299.300	206.482	24.993	53.316	0.054
3.1	12	1092.3500000	1318.140	1.207	0.251	20299.300	257.338	25.011	50.321	0.047
4.1	12	1284.8100000	1437.890	1.119	0.398	20299.300	289.739	24.998	48.186	0.042
5.1	12	1459.6200000	1563.150	1.071	0.631	20299.300	310.440	25.002	46.927	0.038
6.1	12	1608.4100000	1702.260	1.058	1.000	20299.300	324.090	25.015	46.587	0.035
7.1	12	1726.8500000	1865.000	1.080	1.585	20299.300	332.904	25.007	47.164	0.032
8.1	12	1809.0500000	2060.710	1.139	2.512	20299.300	338.738	25.007	48.683	0.029
9.1	12	1866.7600000	2300.410	1.232	3.981	20299.300	344.479	25.007	50.910	0.027
10.1	12	1820.7500000	2565.890	1.409	6.310	20299.300	351.406	24.998	54.634	0.026
11.1	12	1725.8600000	2929.470	1.697	10.000	20299.300	357.802	24.998	59.558	0.024
12.1	12	1533.0100000	3410.170	2.225	15.849	20299.300	364.759	24.998	66.027	0.022
13.1	12	1228.8800000	4098.770	3.335	25.119	20299.200	371.436	25.002	73.933	0.019
14.1	12	861.8630000	5285.730	6.133	39.811	20299.100	378.378	25.002	82.101	0.015
15.1	12	675.0830000	7636.390	11.312	63.096	20298.900	385.164	24.998	87.397	0.011
16.1	12	738.2420000	11760.100	15.930	100.000	20298.100	391.981	24.998	90.434	0.007
1.2	12	668.4500000	1032.390	1.544	0.100	20299.300	126.251	24.998	57.054	0.066

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
2.2	12	865.4240000	1172.160	1.354	0.158	20299.300	206.497	24.998	53.535	0.055
3.2	12	1072.0800000	1301.810	1.214	0.251	20299.300	257.338	24.989	50.498	0.048
4.2	12	1270.6200000	1426.820	1.123	0.398	20299.300	289.723	25.002	48.282	0.042
5.2	12	1446.8000000	1553.830	1.074	0.631	20299.300	310.534	24.998	47.008	0.038
6.2	12	1598.0600000	1693.870	1.060	1.000	20299.300	324.090	24.998	46.630	0.035
7.2	12	1719.3100000	1857.300	1.080	1.585	20299.300	332.920	24.998	47.171	0.032
8.2	12	1820.6200000	2056.610	1.130	2.512	20299.300	338.770	24.989	48.445	0.029
9.2	12	1858.6400000	2291.240	1.233	3.981	20299.300	344.448	24.998	50.920	0.027
10.2	12	1814.0800000	2557.730	1.410	6.310	20299.300	351.437	25.007	54.648	0.026
11.2	12	1719.4800000	2921.520	1.699	10.000	20299.300	357.864	25.007	59.583	0.024
12.2	12	1528.8900000	3404.270	2.227	15.849	20299.300	364.634	25.007	66.048	0.022
13.2	12	1225.0600000	4092.970	3.341	25.119	20299.200	371.155	25.007	73.962	0.019
14.2	12	863.6620000	5276.680	6.110	39.811	20299.100	377.957	24.998	82.069	0.015
15.2	12	674.0270000	7620.400	11.306	63.096	20298.900	384.914	24.998	87.400	0.011
16.2	12	747.0700000	11816.500	15.817	100.000	20298.100	391.607	25.002	90.386	0.007
1.3	12	660.5780000	1026.690	1.554	0.100	20299.300	126.266	25.007	57.219	0.066
2.3	12	860.2780000	1169.220	1.359	0.158	20299.300	206.419	24.989	53.629	0.056
3.3	12	1069.4400000	1300.450	1.216	0.251	20299.300	257.338	24.989	50.538	0.048
4.3	12	1267.5200000	1425.390	1.125	0.398	20299.300	289.692	24.998	48.323	0.042
5.3	12	1447.9000000	1553.520	1.073	0.631	20299.300	310.487	24.993	46.981	0.038
6.3	12	1600.3700000	1693.520	1.058	1.000	20299.300	324.106	25.002	46.583	0.035
7.3	12	1722.0000000	1857.690	1.079	1.585	20299.300	332.904	24.989	47.132	0.032
8.3	12	1806.8800000	2051.980	1.136	2.512	20299.300	338.738	25.011	48.597	0.030
9.3	12	1862.6100000	2290.730	1.230	3.981	20299.300	344.495	24.998	50.854	0.027
10.3	12	1821.2600000	2559.180	1.405	6.310	20299.300	351.484	24.993	54.556	0.026
11.3	12	1723.6600000	2921.550	1.695	10.000	20299.300	358.067	24.989	59.522	0.024
12.3	12	1536.1900000	3404.800	2.216	15.849	20299.300	364.931	25.015	65.949	0.022
13.3	12	1232.7100000	4095.080	3.322	25.119	20299.200	371.498	25.007	73.870	0.019
14.3	12	870.8170000	5278.910	6.062	39.811	20299.100	378.472	25.002	81.996	0.015
15.3	12	679.1590000	7618.440	11.218	63.096	20298.900	385.273	24.998	87.361	0.011
16.3	12	749.2540000	11805.500	15.756	100.000	20298.100	392.122	25.002	90.377	0.007
1.1	13	0.9234220	7.306	7.912	0.100	1922.840	126.251	25.002	82.803	1.039
2.1	13	2.0539900	11.213	5.459	0.158	1922.840	206.482	24.989	79.630	0.671
3.1	13	3.7096900	16.005	4.314	0.251	1922.840	257.431	25.002	76.968	0.466
4.1	13	5.6620000	21.745	3.841	0.398	1922.840	289.754	24.998	75.439	0.341
5.1	13	7.6861700	28.881	3.758	0.631	1922.830	310.502	24.993	75.162	0.256
6.1	13	9.6475900	37.586	3.896	1.000	1922.820	324.090	25.007	75.730	0.197
7.1	13	11.3505000	49.862	4.393	1.585	1922.810	332.873	25.002	77.417	0.150
8.1	13	12.7302000	66.836	5.250	2.512	1922.790	338.723	24.998	79.676	0.113
9.1	13	14.0052000	94.402	6.741	3.981	1922.780	344.464	25.007	82.391	0.080
10.1	13	17.1700000	140.031	8.156	6.310	1922.780	351.421	24.998	84.426	0.054

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
11.1	13	22.8906000	216.921	9.476	10.000	1922.770	357.880	24.998	86.283	0.035
12.1	13	33.1559000	341.241	10.292	15.849	1922.770	364.790	25.007	88.145	0.022
13.1	13	48.2524000	531.254	11.010	25.119	1922.770	371.498	25.002	90.785	0.014
14.1	13	73.1907000	820.982	11.217	39.811	1922.760	378.409	24.998	94.618	0.009
15.1	13	110.7390000	1267.720	11.448	63.096	1922.740	385.336	24.989	100.708	0.006
16.1	13	175.6960000	1987.920	11.315	100.000	1922.680	392.028	24.998	109.519	0.004
1.2	13	1.2599900	8.397	6.665	0.100	1922.850	126.220	24.998	81.472	0.901
2.2	13	2.4265900	12.296	5.067	0.158	1922.850	206.482	25.007	78.846	0.610
3.2	13	4.0554800	17.146	4.228	0.251	1922.840	257.447	24.998	76.710	0.434
4.2	13	5.9464200	22.970	3.863	0.398	1922.840	289.926	24.998	75.518	0.322
5.2	13	7.9612800	30.207	3.794	0.631	1922.830	310.783	24.998	75.296	0.245
6.2	13	9.8565300	39.396	3.997	1.000	1922.820	324.184	25.011	76.073	0.188
7.2	13	11.6457000	51.812	4.449	1.585	1922.810	332.998	25.011	77.565	0.144
8.2	13	13.1987000	70.565	5.346	2.512	1922.800	338.863	24.985	79.841	0.107
9.2	13	15.1754000	101.081	6.661	3.981	1922.790	344.588	25.020	82.236	0.075
10.2	13	19.0795000	152.644	8.000	6.310	1922.780	351.546	24.998	84.173	0.050
11.2	13	26.3468000	239.288	9.082	10.000	1922.780	358.129	24.998	85.805	0.032
12.2	13	37.8249000	379.527	10.034	15.849	1922.780	365.024	25.011	87.627	0.020
13.2	13	57.6447000	593.159	10.290	25.119	1922.780	371.701	24.993	89.793	0.013
14.2	13	100.5550000	951.897	9.466	39.811	1922.780	378.425	24.998	92.338	0.008
15.2	13	168.8390000	1492.200	8.838	63.096	1922.760	385.180	25.002	96.931	0.005
16.2	13	270.8580000	2297.760	8.483	100.000	1922.690	391.981	25.007	104.846	0.003
1.3	13	1.4002300	8.905	6.360	0.100	1922.850	126.220	25.002	81.070	0.849
2.3	13	2.6414200	12.835	4.859	0.158	1922.850	206.497	24.998	78.380	0.584
3.3	13	4.2207200	17.719	4.198	0.251	1922.840	257.338	25.015	76.619	0.420
4.3	13	6.0553000	23.492	3.880	0.398	1922.840	289.692	24.993	75.578	0.315
5.3	13	8.1500600	30.867	3.787	0.631	1922.830	310.502	24.985	75.269	0.240
6.3	13	10.0766000	40.111	3.981	1.000	1922.820	323.965	24.998	76.016	0.185
7.3	13	11.8066000	53.196	4.506	1.585	1922.810	332.764	25.002	77.713	0.141
8.3	13	13.4126000	72.772	5.426	2.512	1922.800	338.723	25.015	79.980	0.104
9.3	13	15.6711000	106.114	6.771	3.981	1922.790	344.417	25.007	82.337	0.071
10.3	13	18.9833000	158.271	8.337	6.310	1922.790	351.515	24.993	84.413	0.048
11.3	13	25.4184000	255.972	10.070	10.000	1922.790	357.958	24.985	86.284	0.030
12.3	13	41.2148000	409.982	9.947	15.849	1922.790	364.790	25.020	87.329	0.019
13.3	13	65.8869000	646.512	9.812	25.119	1922.790	371.436	25.002	89.078	0.012
14.3	13	114.0550000	1025.330	8.990	39.811	1922.780	378.331	24.993	91.413	0.007
15.3	13	181.9810000	1574.620	8.653	63.096	1922.760	385.086	24.989	96.095	0.005
16.3	13	298.1850000	2429.940	8.149	100.000	1922.690	391.841	25.020	103.458	0.003
1.1	14	7.8759900	117.812	14.958	0.100	23339.700	126.204	25.002	86.173	0.787
2.1	14	13.1353000	159.768	12.163	0.158	23339.700	206.560	25.011	85.297	0.579
3.1	14	32.2514000	242.583	7.522	0.251	23339.700	257.462	25.002	82.423	0.379

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
4.1	14	76.0537000	369.431	4.858	0.398	23339.700	289.786	24.976	78.361	0.246
5.1	14	156.4800000	527.012	3.368	0.631	23339.700	310.487	24.993	73.454	0.169
6.1	14	278.4610000	706.104	2.536	1.000	23339.700	323.981	24.993	68.468	0.122
7.1	14	435.4210000	890.159	2.044	1.585	23339.700	332.810	25.002	63.926	0.094
8.1	14	612.4300000	1068.110	1.744	2.512	23339.600	338.692	25.011	60.169	0.075
9.1	14	800.4570000	1243.080	1.553	3.981	23339.600	344.479	25.002	57.239	0.063
10.1	14	960.3170000	1402.770	1.461	6.310	23339.600	351.452	24.998	55.671	0.055
11.1	14	1107.3200000	1574.870	1.422	10.000	23339.600	357.926	25.015	55.068	0.048
12.1	14	1216.3100000	1759.030	1.446	15.849	23339.500	364.712	24.989	55.789	0.044
13.1	14	1284.6200000	1975.250	1.538	25.119	23339.400	371.358	25.002	58.066	0.040
14.1	14	1296.5300000	2240.700	1.728	39.811	23339.200	378.206	25.007	62.644	0.037
15.1	14	1245.6000000	2581.390	2.072	63.096	23338.800	385.008	24.985	70.830	0.034
16.1	14	1157.9200000	3009.650	2.599	100.000	23337.600	391.794	25.015	84.697	0.031
1.2	14	3.5489500	96.556	27.207	0.100	23339.700	126.344	24.993	87.893	0.961
2.2	14	9.1913700	153.523	16.703	0.158	23339.700	206.575	24.998	86.571	0.604
3.2	14	30.8533000	257.073	8.332	0.251	23339.700	257.400	24.998	83.152	0.359
4.2	14	81.6757000	403.069	4.935	0.398	23339.700	289.786	24.985	78.538	0.226
5.2	14	177.0970000	581.700	3.285	0.631	23340.300	310.612	24.998	73.057	0.153
6.2	14	317.4430000	774.579	2.440	1.000	23339.700	324.199	24.993	67.703	0.111
7.2	14	496.3630000	967.904	1.950	1.585	23339.700	333.091	24.989	62.839	0.085
8.2	14	692.3240000	1150.790	1.662	2.512	23339.600	338.941	24.998	58.963	0.069
9.2	14	886.1260000	1322.150	1.492	3.981	23341.500	344.542	24.998	56.181	0.058
10.2	14	1046.9400000	1478.150	1.412	6.310	23339.600	351.577	24.998	54.748	0.051
11.2	14	1196.1300000	1648.720	1.378	10.000	23339.600	358.020	25.002	54.203	0.046
12.2	14	1301.8300000	1833.200	1.408	15.849	23339.500	364.837	24.998	55.041	0.042
13.2	14	1365.4900000	2049.430	1.501	25.119	23339.400	371.452	25.002	57.370	0.038
14.2	14	1370.5400000	2316.820	1.690	39.811	23339.300	378.440	24.998	61.969	0.035
15.2	14	1309.5000000	2657.390	2.029	63.096	23338.800	385.273	25.007	70.108	0.033
16.2	14	1208.1600000	3086.660	2.555	100.000	23337.700	392.059	25.002	83.901	0.030
1.3	14	4.1173400	103.180	25.060	0.100	23339.700	126.204	24.989	87.713	0.899
2.3	14	11.0968000	164.526	14.827	0.158	23339.700	206.466	25.002	86.138	0.563
3.3	14	34.6531000	278.975	8.051	0.251	23339.700	257.306	25.002	82.914	0.330
4.3	14	94.2184000	441.338	4.684	0.398	23339.700	289.708	25.015	77.941	0.206
5.3	14	202.6330000	638.089	3.149	0.631	23339.700	310.362	24.989	72.371	0.139
6.3	14	362.7030000	844.262	2.328	1.000	23339.700	323.872	25.020	66.737	0.101
7.3	14	564.5740000	1047.960	1.856	1.585	23339.700	332.701	24.989	61.673	0.078
8.3	14	780.0970000	1235.100	1.583	2.512	23339.700	338.567	24.993	57.714	0.064
9.3	14	983.3960000	1405.910	1.430	3.981	23339.600	344.245	25.011	55.035	0.054
10.3	14	1149.6700000	1559.870	1.357	6.310	23339.600	351.125	25.015	53.657	0.048
11.3	14	1294.3500000	1728.710	1.336	10.000	23339.600	357.661	24.993	53.325	0.043
12.3	14	1398.1100000	1914.970	1.370	15.849	23339.500	364.603	24.985	54.258	0.039

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
13.3	14	1454.4900000	2132.010	1.466	25.119	23339.500	371.124	25.011	56.682	0.036
14.3	14	1449.2600000	2397.950	1.655	39.811	23339.300	378.035	25.007	61.307	0.034
15.3	14	1376.4600000	2737.280	1.989	63.096	23338.900	385.008	24.985	69.403	0.032
16.3	14	1263.4700000	3168.960	2.508	100.000	23337.700	391.747	25.007	83.067	0.029
1.1	15	0.0022603	0.029	12.839	0.100	11.472	126.329	24.998	87.289	1.571
2.1	15	0.0037748	0.045	12.027	0.158	11.471	206.575	24.993	88.046	1.005
3.1	15	0.0081010	0.071	8.717	0.251	11.470	257.525	24.998	87.965	0.646
4.1	15	0.0170856	0.109	6.369	0.398	11.469	289.879	25.011	88.400	0.419
5.1	15	0.0326647	0.161	4.936	0.631	11.466	310.830	25.002	90.929	0.283
6.1	15	0.0580799	0.235	4.046	1.000	11.462	324.262	24.993	97.405	0.192
7.1	15	0.1004780	0.341	3.394	1.585	11.463	333.091	25.015	109.703	0.126
8.1	15	0.1685570	0.490	2.905	2.512	11.476	339.128	25.007	128.576	0.073
9.1	15	0.2672670	0.700	2.620	3.981	11.496	344.776	25.011	148.382	0.034
10.1	15	0.4181410	1.001	2.395	6.310	11.508	351.546	25.002	162.151	0.014
11.1	15	0.6035370	1.448	2.399	10.000	11.513	358.004	25.011	170.054	0.005
12.1	15	0.8642430	2.140	2.477	15.849	11.515	364.775	24.985	174.288	0.002
13.1	15	1.6254400	3.140	1.932	25.119	11.515	371.405	24.976	176.690	0.001
14.1	15	2.3455700	4.727	2.015	39.811	11.516	378.300	24.985	178.040	0.000
15.1	15	3.3458200	7.567	2.262	63.096	11.516	385.133	24.998	178.760	0.000
16.1	15	8.7907400	11.994	1.364	100.000	11.515	391.966	25.007	179.217	0.000
1.2	15	0.0022937	0.031	13.652	0.100	11.475	126.251	24.985	87.427	1.457
2.2	15	0.0044383	0.051	11.384	0.158	11.476	206.372	24.989	87.493	0.903
3.2	15	0.0092973	0.078	8.367	0.251	11.475	257.322	24.998	87.273	0.586
4.2	15	0.0190346	0.118	6.177	0.398	11.473	289.676	25.007	87.570	0.388
5.2	15	0.0362117	0.177	4.890	0.631	11.470	310.424	25.015	89.699	0.258
6.2	15	0.0640486	0.257	4.017	1.000	11.467	323.872	25.002	95.455	0.177
7.2	15	0.1084350	0.370	3.415	1.585	11.466	332.732	25.007	107.137	0.118
8.2	15	0.1782620	0.533	2.991	2.512	11.476	338.738	25.011	125.540	0.070
9.2	15	0.2811990	0.767	2.726	3.981	11.495	344.495	25.002	145.691	0.034
10.2	15	0.4267390	1.112	2.605	6.310	11.508	351.437	25.002	160.280	0.014
11.2	15	0.6196400	1.622	2.618	10.000	11.513	357.895	25.002	168.867	0.005
12.2	15	0.9177240	2.398	2.613	15.849	11.515	364.712	24.989	173.589	0.002
13.2	15	1.3477600	3.791	2.813	25.119	11.515	371.311	25.015	176.026	0.001
14.2	15	2.7087700	5.616	2.073	39.811	11.516	378.253	24.989	177.665	0.000
15.2	15	3.5657800	8.789	2.465	63.096	11.516	385.102	25.015	178.558	0.000
16.2	15	8.2532300	14.375	1.742	100.000	11.515	391.950	24.985	179.062	0.000
1.3	15	0.0024546	0.035	14.388	0.100	11.480	126.173	25.002	87.457	1.292
2.3	15	0.0051266	0.056	10.918	0.158	11.480	206.450	25.002	87.034	0.815
3.3	15	0.0107396	0.086	7.972	0.251	11.478	257.260	25.002	86.559	0.532
4.3	15	0.0217533	0.130	5.996	0.398	11.477	289.739	25.011	86.618	0.349
5.3	15	0.0414660	0.198	4.783	0.631	11.475	310.534	24.998	88.214	0.230

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
6.3	15	0.0716788	0.287	4.006	1.000	11.471	324.043	25.002	93.377	0.159
7.3	15	0.1185210	0.415	3.502	1.585	11.471	332.779	24.993	104.078	0.107
8.3	15	0.1896800	0.595	3.136	2.512	11.477	338.614	25.007	121.848	0.065
9.3	15	0.2909670	0.858	2.947	3.981	11.494	344.354	24.985	142.398	0.033
10.3	15	0.4390910	1.242	2.829	6.310	11.507	351.234	25.002	158.093	0.014
11.3	15	0.6350930	1.824	2.872	10.000	11.513	357.692	25.002	167.498	0.005
12.3	15	0.9652530	2.665	2.761	15.849	11.515	364.650	24.998	172.867	0.002
13.3	15	1.5023400	4.056	2.700	25.119	11.515	371.155	25.015	175.737	0.001
14.3	15	3.0984700	5.960	1.923	39.811	11.516	377.957	24.989	177.516	0.000
15.3	15	3.6160700	9.737	2.693	63.096	11.516	384.883	25.015	178.403	0.000
16.3	15	5.7208800	17.176	3.002	100.000	11.515	391.560	24.993	178.882	0.000
1.1	17	0.0014694	0.017	11.783	0.100	8.093	126.251	25.007	88.071	1.859
2.1	17	0.0025317	0.027	10.711	0.158	8.092	206.497	24.998	89.354	1.187
3.1	17	0.0053070	0.042	7.944	0.251	8.091	257.353	24.998	90.387	0.764
4.1	17	0.0106691	0.065	6.053	0.398	8.089	289.801	25.002	92.993	0.498
5.1	17	0.0195630	0.098	4.998	0.631	8.088	310.612	24.998	99.132	0.325
6.1	17	0.0336935	0.146	4.344	1.000	8.091	324.262	25.002	110.572	0.206
7.1	17	0.0555794	0.217	3.911	1.585	8.103	333.076	24.998	127.537	0.118
8.1	17	0.0864211	0.320	3.706	2.512	8.122	338.972	24.998	145.885	0.057
9.1	17	0.1207090	0.473	3.919	3.981	8.136	344.666	24.993	159.774	0.024
10.1	17	0.1980140	0.684	3.453	6.310	8.141	351.593	24.998	168.397	0.010
11.1	17	0.2775810	1.007	3.628	10.000	8.144	358.114	25.002	173.309	0.004
12.1	17	0.4806950	1.506	3.134	15.849	8.144	364.900	24.998	176.043	0.001
13.1	17	0.9294010	2.339	2.517	25.119	8.145	371.530	25.002	177.564	0.001
14.1	17	2.2749600	3.699	1.626	39.811	8.145	378.472	24.993	178.467	0.000
15.1	17	5.4539900	5.784	1.060	63.096	8.145	385.304	25.007	179.046	0.000
16.1	17	11.1251000	11.206	1.007	100.000	8.144	392.059	25.002	179.266	0.000
1.2	17	0.0015110	0.018	12.007	0.100	8.095	126.220	25.007	88.028	1.774
2.2	17	0.0026936	0.029	10.660	0.158	8.095	206.575	25.007	89.067	1.122
3.2	17	0.0058009	0.045	7.725	0.251	8.094	257.494	25.011	89.732	0.719
4.2	17	0.0112797	0.069	6.119	0.398	8.093	289.895	24.993	92.295	0.466
5.2	17	0.0207868	0.105	5.033	0.631	8.091	310.768	25.007	97.887	0.305
6.2	17	0.0354393	0.157	4.423	1.000	8.093	324.308	25.002	108.742	0.195
7.2	17	0.0585587	0.233	3.976	1.585	8.104	333.310	24.998	125.165	0.113
8.2	17	0.0899982	0.344	3.819	2.512	8.122	339.269	24.989	143.774	0.056
9.2	17	0.1253490	0.506	4.038	3.981	8.135	344.900	25.002	158.411	0.024
10.2	17	0.2110080	0.737	3.491	6.310	8.141	351.749	25.007	167.481	0.010
11.2	17	0.3092590	1.091	3.527	10.000	8.144	358.379	24.998	172.732	0.004
12.2	17	0.5198420	1.647	3.168	15.849	8.144	365.149	24.993	175.668	0.001
13.2	17	0.9250080	2.495	2.698	25.119	8.145	371.842	25.002	177.402	0.001
14.2	17	1.8654300	3.877	2.078	39.811	8.145	378.877	24.998	178.398	0.000

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
15.2	17	2.7078100	6.297	2.325	63.096	8.145	385.819	25.002	178.970	0.000
16.2	17	7.4788700	10.937	1.462	100.000	8.144	392.543	25.002	179.287	0.000
1.3	17	0.0016947	0.020	11.513	0.100	8.099	126.298	25.002	87.627	1.650
2.3	17	0.0029625	0.031	10.453	0.158	8.099	206.450	24.993	88.638	1.040
3.3	17	0.0063365	0.048	7.633	0.251	8.098	257.260	25.007	89.118	0.666
4.3	17	0.0121003	0.074	6.139	0.398	8.097	289.661	25.007	91.500	0.434
5.3	17	0.0223057	0.112	5.023	0.631	8.095	310.580	24.998	96.606	0.286
6.3	17	0.0379301	0.168	4.429	1.000	8.096	324.230	25.002	106.792	0.184
7.3	17	0.0621269	0.251	4.040	1.585	8.104	333.169	25.007	122.592	0.108
8.3	17	0.0960688	0.371	3.857	2.512	8.121	338.972	25.007	141.332	0.054
9.3	17	0.1353440	0.548	4.050	3.981	8.134	344.713	25.002	156.639	0.023
10.3	17	0.2207010	0.802	3.636	6.310	8.141	351.562	25.002	166.364	0.010
11.3	17	0.3135350	1.199	3.826	10.000	8.143	358.145	24.989	172.013	0.004
12.3	17	0.5364480	1.806	3.366	15.849	8.144	365.024	24.993	175.249	0.001
13.3	17	1.0620900	2.812	2.648	25.119	8.145	371.857	25.002	177.065	0.001
14.3	17	1.6332700	4.393	2.689	39.811	8.145	378.830	24.998	178.188	0.000
15.3	17	5.6322800	7.302	1.296	63.096	8.145	385.819	25.007	178.795	0.000
16.3	17	5.6137100	11.705	2.085	100.000	8.144	392.434	24.998	179.238	0.000
1.1	20	125.5850000	802.461	6.38978	0.1	81463.7	126.266	25.011	81.0872	0.399087
2.1	20	282.4170000	1198.61	4.2441	0.15849	81463.7	206.388	24.998	76.7145	0.263247
3.1	20	557.9100000	1700.85	3.04861	0.251189	81463.7	257.182	25.011	71.801	0.181119
4.1	20	899.2150000	2228.97	2.47879	0.398107	81463.7	289.583	24.993	67.9794	0.134907
5.1	20	1256.8000000	2777.81	2.21022	0.630957	81463.7	310.268	25.007	65.5934	0.106365
6.1	20	1601.3400000	3349.44	2.09166	1	81463.6	323.825	24.998	64.3731	0.0873638
7.1	20	1941.5100000	3985.77	2.05292	1.5849	81463.6	332.654	25.007	63.9408	0.0731682
8.1	20	2203.6000000	4678.37	2.12306	2.51189	81463.6	338.551	24.998	64.6779	0.0627363
9.1	20	2346.4900000	5458	2.32603	3.98105	81463.6	344.261	24.993	66.6246	0.0546145
10.1	20	2259.7600000	6294.2	2.78534	6.30957	81463.6	351.031	24.989	70.136	0.0485204
11.1	20	2002.3900000	7437.35	3.71423	10.0001	81463.5	357.677	24.993	74.8256	0.0421295
12.1	20	1456.0100000	9040.82	6.20931	15.849	81463.5	364.51	24.989	80.7826	0.0354306
13.1	20	834.6890000	11847.2	14.1936	25.1188	81463.4	371.077	24.993	85.9686	0.0273104
14.1	20	665.5850000	17714.6	26.615	39.8105	81463.2	377.832	24.989	87.8973	0.0182957
15.1	20	831.9660000	27199.1	32.6925	63.0957	81462.4	384.712	24.993	88.3693	0.0119213
16.1	20	1261.8700000	42850.7	33.9581	100	81459.3	391.482	24.993	88.5191	0.00757108
1.1	21	131.5940000	1146.79	8.71467	0.1	162469	126.204	25.007	83.4279	0.56005
2.1	21	156.6940000	1293.14	8.25266	0.15849	162469	206.497	25.007	83.0617	0.496302
3.1	21	257.6080000	1677.02	6.50998	0.251189	162469	257.384	25.015	81.2291	0.381041
4.1	21	409.6580000	2165.97	5.28726	0.398107	162469	289.739	24.993	79.241	0.293304
5.1	21	642.6010000	2791.88	4.34465	0.630957	162469	310.502	25.007	76.9753	0.225703
6.1	21	987.9830000	3595.85	3.63959	1	162469	323.996	24.989	74.5563	0.17342
7.1	21	1428.9600000	4522.93	3.16519	1.5849	162469	332.717	25.002	72.3665	0.136365

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
8.1	21	1921.2300000	5553.45	2.89057	2.51189	162469	338.645	25.011	70.7957	0.110095
9.1	21	2418.1500000	6757.67	2.79456	3.98105	162469	344.354	24.993	70.1678	0.0901605
10.1	21	2723.9900000	7991.23	2.93365	6.30957	162469	351.047	24.989	71.0181	0.0766615
11.1	21	2924.0300000	9588.48	3.2792	10.0001	162469	357.63	25.002	72.8712	0.064579
12.1	21	2802.0900000	11561.2	4.12593	15.849	162469	364.588	25.024	76.217	0.0544268
13.1	21	2272.6200000	14413.2	6.34213	25.1188	162469	371.155	25.002	80.9281	0.044371
14.1	21	1400.5300000	19909.4	14.2156	39.8105	162469	377.972	24.989	85.9243	0.0324252
15.1	21	1174.4000000	33918.5	28.8814	63.0957	162467	384.914	24.976	87.8388	0.0190662
16.1	21	1753.6300000	61326.2	34.9711	100	162461	391.622	25.007	87.7926	0.0105536
1.1	18	1.9289500	27.5163	14.2649	0.1	11026.1	126.298	24.989	85.9912	1.59048
2.1	18	5.7271500	43.4578	7.58804	0.15849	11026.1	206.575	24.993	82.4943	1.00087
3.1	18	12.5722000	64.0723	5.09633	0.251189	11026.1	257.384	24.993	78.9018	0.671919
4.1	18	23.5395000	89.6821	3.80985	0.398107	11026.1	289.754	24.998	75.2992	0.473183
5.1	18	39.2816000	120.474	3.06693	0.630957	11026.1	310.596	25.002	71.9534	0.346253
6.1	18	57.6211000	154.952	2.68915	1	11026.1	324.137	25.011	69.6268	0.26543
7.1	18	78.6016000	194.407	2.47332	1.5849	11026.1	333.044	24.993	68.0376	0.209304
8.1	18	101.5760000	241.936	2.38181	2.51189	11026.1	338.832	24.998	67.3319	0.167341
9.1	18	121.4040000	294.057	2.42214	3.98105	11026	344.542	25.02	67.7933	0.138139
10.1	18	132.7950000	350.588	2.64008	6.30957	11026	351.499	24.998	69.7512	0.117414
11.1	18	136.5450000	421.998	3.09054	10.0001	11025.9	357.895	24.993	73.1537	0.0995088
12.1	18	131.0470000	517.819	3.95141	15.849	11025.8	364.744	25.015	78.1182	0.0829146
13.1	18	123.0900000	671.484	5.45524	25.1188	11025.7	371.342	24.998	84.2608	0.0650115
14.1	18	125.7760000	941.878	7.48851	39.8105	11025.6	378.331	24.985	90.8513	0.046576
15.1	18	147.1820000	1406.18	9.55406	63.0957	11025.5	385.164	25.007	98.2556	0.0308776
16.1	18	195.0790000	2177.29	11.161	100	11025.1	391.934	25.015	107.518	0.0192163

Appendix B - Scrubbed Viscosity Data (Negative Values Set to 0.0000001 to Remove Zeroing Error

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
1.1	1	0.0026075	1.388	532.299	0.100	707.890	126.313	24.998	89.929	2.029
2.1	1	0.0356225	2.200	61.769	0.158	707.890	206.450	25.002	89.130	1.280
3.1	1	0.1234500	3.481	28.197	0.251	707.890	257.306	25.011	88.060	0.809
4.1	1	0.3301270	5.417	16.409	0.398	707.889	289.754	25.002	86.660	0.519
5.1	1	0.7373360	8.249	11.188	0.631	707.887	310.612	25.007	85.135	0.340
6.1	1	1.4294200	12.173	8.516	1.000	707.882	324.199	24.998	83.713	0.230
7.1	1	2.4732200	17.433	7.049	1.585	707.876	333.107	25.007	82.641	0.160
8.1	1	3.9377400	24.559	6.237	2.512	707.867	338.957	25.002	82.162	0.114
9.1	1	6.1659500	34.386	5.577	3.981	707.856	344.542	25.007	82.107	0.081
10.1	1	9.8761000	48.401	4.901	6.310	707.845	351.530	25.002	82.511	0.058
11.1	1	16.9270000	70.272	4.151	10.000	707.835	358.036	24.998	83.424	0.040
12.1	1	26.9450000	101.450	3.765	15.849	707.822	364.900	25.002	87.305	0.028
13.1	1	38.6584000	146.791	3.797	25.119	707.812	371.576	24.993	96.618	0.019
14.1	1	51.7451000	217.665	4.206	39.811	707.819	378.487	24.993	112.046	0.012
15.1	1	68.7612000	330.991	4.814	63.096	707.850	385.289	24.993	130.495	0.006
16.1	1	91.3104000	510.602	5.592	100.000	707.866	392.137	25.007	147.170	0.003
1.1	2	0.0000001	0.023	229803.000	0.100	54.961	126.266	24.998	92.503	9.507
2.1	2	0.0000001	0.035	352992.000	0.158	54.954	206.591	25.007	94.450	6.176
3.1	2	0.0000001	0.058	582582.000	0.251	54.966	257.478	24.989	96.036	3.733
4.1	2	0.0000001	0.089	886352.000	0.398	54.959	289.832	24.998	99.224	2.435
5.1	2	0.0000001	0.135	1351640.000	0.631	54.960	310.768	24.998	104.716	1.565
6.1	2	0.0023918	0.217	90.656	1.000	54.983	324.246	24.993	111.608	0.938
7.1	2	0.0094025	0.354	37.627	1.585	55.026	333.091	24.989	120.971	0.531
8.1	2	0.0364510	0.594	16.290	2.512	55.082	339.113	25.002	131.239	0.278
9.1	2	0.1096470	1.009	9.199	3.981	55.138	344.588	25.007	141.964	0.134
10.1	2	0.2929370	1.722	5.878	6.310	55.181	351.593	25.011	151.876	0.060
11.1	2	0.6541040	3.026	4.627	10.000	55.207	358.098	24.998	159.682	0.025
12.1	2	1.6895700	5.629	3.332	15.849	55.219	364.837	24.998	164.634	0.010
13.1	2	5.7206100	9.759	1.706	25.119	55.226	371.436	24.989	168.951	0.004
14.1	2	12.1271000	11.499	0.948	39.811	55.230	378.222	24.989	174.858	0.002
15.1	2	16.0183000	14.493	0.905	63.096	55.231	385.070	24.998	177.526	0.001
16.1	2	22.2009000	19.961	0.899	100.000	55.229	391.903	24.993	178.671	0.000
1.1	3	0.0578856	2.443	42.202	0.100	1504.730	126.329	25.011	88.663	2.450
2.1	3	0.1344310	3.784	28.149	0.158	1504.730	206.513	25.015	87.999	1.581
3.1	3	0.2937070	5.889	20.051	0.251	1504.730	257.369	25.011	87.199	1.015
4.1	3	0.5841980	9.006	15.416	0.398	1504.730	289.723	25.015	86.377	0.663

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
5.1	3	1.0407200	13.537	13.008	0.631	1504.720	310.596	24.985	85.751	0.441
6.1	3	1.6211500	19.891	12.270	1.000	1504.720	324.121	25.002	85.593	0.300
7.1	3	2.1821200	28.900	13.244	1.585	1504.710	333.044	25.002	86.119	0.207
8.1	3	2.7416000	42.549	15.520	2.512	1504.700	338.863	25.002	87.060	0.141
9.1	3	3.6194500	64.286	17.761	3.981	1504.700	344.557	24.989	88.020	0.093
10.1	3	5.6270700	99.387	17.662	6.310	1504.690	351.499	24.998	88.780	0.060
11.1	3	9.1919600	155.698	16.938	10.000	1504.690	357.926	25.007	89.862	0.038
12.1	3	14.9093000	244.916	16.427	15.849	1504.690	364.712	25.007	91.694	0.024
13.1	3	22.6469000	380.053	16.782	25.119	1504.690	371.311	24.993	94.962	0.016
14.1	3	35.3776000	594.085	16.793	39.811	1504.690	378.144	25.002	99.966	0.010
15.1	3	53.9149000	932.396	17.294	63.096	1504.680	385.039	25.011	107.679	0.006
16.1	3	79.6553000	1461.820	18.352	100.000	1504.640	391.919	24.993	118.760	0.004
1.1	4	0.2846480	2.527	8.878	0.100	718.785	126.329	24.985	83.593	1.125
2.1	4	0.6185810	3.857	6.236	0.158	718.785	206.482	24.993	80.921	0.732
3.1	4	1.1416100	5.561	4.871	0.251	718.782	257.384	25.011	78.455	0.504
4.1	4	1.8128100	7.648	4.219	0.398	718.777	289.676	24.989	76.765	0.364
5.1	4	2.5352600	10.164	4.009	0.631	718.770	310.424	24.989	76.181	0.273
6.1	4	3.2345100	13.320	4.118	1.000	718.759	323.918	24.998	76.710	0.209
7.1	4	3.8075300	17.678	4.643	1.585	718.747	332.748	24.985	78.533	0.159
8.1	4	4.2178600	23.829	5.649	2.512	718.733	338.614	25.007	81.265	0.119
9.1	4	4.6693400	33.656	7.208	3.981	718.722	344.323	25.011	84.449	0.085
10.1	4	5.4956200	49.755	9.054	6.310	718.714	351.250	24.998	87.719	0.057
11.1	4	7.1711100	75.931	10.588	10.000	718.710	357.755	25.011	91.248	0.038
12.1	4	9.8487400	118.588	12.041	15.849	718.709	364.619	24.985	95.931	0.024
13.1	4	13.3475000	183.294	13.733	25.119	718.711	371.140	25.002	103.006	0.015
14.1	4	18.7032000	285.314	15.255	39.811	718.719	377.957	25.015	113.011	0.009
15.1	4	26.8363000	449.364	16.745	63.096	718.733	384.961	24.980	125.836	0.005
16.1	4	36.4063000	710.315	19.511	100.000	718.733	391.747	24.998	139.988	0.003
1.1	5	578970.0000000	616711.000	1.065	0.100	2270.670	126.532	25.011	36.272	0.000
2.1	5	841955.0000000	736892.000	0.875	0.158	2270.670	207.433	24.989	29.546	0.000
3.1	5	1083610.0000000	863755.000	0.797	0.251	2270.670	259.178	24.998	25.799	0.000
4.1	5	1414120.0000000	979427.000	0.693	0.398	2270.670	292.406	25.002	21.407	0.000
5.1	5	1657790.0000000	1181890.000	0.713	0.631	2270.670	313.903	24.976	20.405	0.000
6.1	5	2035890.0000000	1446850.000	0.711	1.000	2270.670	328.224	25.011	18.505	0.000
7.1	5	2587680.0000000	1546230.000	0.598	1.585	2270.670	337.865	25.011	14.639	0.000
8.1	5	3008390.0000000	1865210.000	0.620	2.512	2270.670	344.573	24.989	13.784	0.000
9.1	5	3680330.0000000	2115010.000	0.575	3.981	2270.670	350.891	24.989	11.617	0.000
10.1	5	4301210.0000000	2219390.000	0.516	6.310	2270.670	358.457	25.011	9.767	0.000
11.1	5	4925320.0000000	2244130.000	0.456	10.000	2270.670	365.867	25.020	8.150	0.000
12.1	5	5546040.0000000	2362660.000	0.426	15.849	2270.670	373.542	24.993	7.134	0.000
13.1	5	6342320.0000000	2026130.000	0.319	25.119	2270.670	380.999	24.980	5.122	0.000

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
14.1	5	6855260.0000000	2023090.000	0.295	39.811	2270.670	388.658	25.002	4.519	0.000
15.1	5	7454250.0000000	1840790.000	0.247	63.096	2270.650	396.318	25.020	3.622	0.000
16.1	5	7788220.0000000	1718090.000	0.221	100.000	2270.560	403.946	24.993	3.160	0.000
1.1	6	0.0468656	2.198	46.909	0.100	1425.220	126.235	25.020	88.802	2.579
2.1	6	0.0945248	3.468	36.688	0.158	1425.220	206.497	25.007	88.475	1.635
3.1	6	0.2096610	5.451	26.001	0.251	1425.220	257.322	24.993	87.856	1.040
4.1	6	0.4141090	8.438	20.377	0.398	1425.220	289.723	25.002	87.285	0.671
5.1	6	0.7582020	12.825	16.915	0.631	1425.220	310.518	24.985	86.773	0.441
6.1	6	1.2624100	19.042	15.084	1.000	1425.210	324.059	25.011	86.471	0.297
7.1	6	1.7526100	28.055	16.007	1.585	1425.210	332.826	25.011	86.876	0.202
8.1	6	2.3168500	42.367	18.287	2.512	1425.200	338.832	24.993	87.620	0.134
9.1	6	3.3614700	65.463	19.475	3.981	1425.200	344.526	24.985	88.282	0.087
10.1	6	5.2992700	102.416	19.327	6.310	1425.200	351.437	24.998	88.999	0.055
11.1	6	8.7068200	163.649	18.796	10.000	1425.200	358.176	25.020	90.039	0.035
12.1	6	13.9718000	260.083	18.615	15.849	1425.200	364.993	25.007	91.801	0.022
13.1	6	21.9415000	411.543	18.756	25.119	1425.200	371.701	24.993	94.680	0.014
14.1	6	33.9173000	640.995	18.899	39.811	1425.190	378.628	24.985	99.375	0.009
15.1	6	54.7788000	1017.230	18.570	63.096	1425.190	385.445	25.011	106.237	0.005
16.1	6	87.8863000	1597.240	18.174	100.000	1425.140	392.122	25.015	116.430	0.003
1.1	9	0.0000667	0.031	468.735	0.100	575.584	126.313	25.020	91.496	73.252
2.1	9	0.0000001	0.044	439618.000	0.158	575.330	206.544	25.002	93.931	51.949
3.1	9	0.0000010	0.067	66676.000	0.251	575.246	257.462	24.980	96.570	34.102
4.1	9	0.0000010	0.104	103880.000	0.398	575.259	289.832	24.998	100.817	21.643
5.1	9	0.0000010	0.165	165420.000	0.631	575.368	310.814	25.007	105.884	13.311
6.1	9	0.0000010	0.273	273446.000	1.000	575.590	324.152	25.011	111.004	7.819
7.1	9	0.0000010	0.468	467935.000	1.585	575.967	333.076	24.993	118.636	4.298
8.1	9	0.0000010	0.835	835413.000	2.512	576.373	339.035	24.985	125.628	2.231
9.1	9	0.0000010	1.550	1550250.000	3.981	576.781	344.776	24.985	133.275	1.078
10.1	9	0.0301186	2.667	88.559	6.310	577.099	351.624	25.011	142.565	0.523
11.1	9	0.3568410	4.910	13.760	10.000	577.336	358.129	25.011	149.901	0.235
12.1	9	1.1220500	10.089	8.991	15.849	577.480	364.962	25.011	154.395	0.098
13.1	9	2.8432800	21.875	7.693	25.119	577.562	371.654	24.993	157.517	0.040
14.1	9	10.3914000	50.820	4.891	39.811	577.602	378.472	24.989	158.575	0.017
15.1	9	53.3771000	109.855	2.058	63.096	577.620	385.367	24.980	159.768	0.007
16.1	9	176.1390000	137.795	0.782	100.000	577.626	392.168	25.011	168.967	0.003
1.1	10	0.1117310	0.683	6.115	0.100	156.889	126.329	24.998	80.785	0.902
2.1	10	0.1862550	0.930	4.992	0.158	156.885	206.575	24.989	78.804	0.659
3.1	10	0.3119380	1.289	4.131	0.251	156.881	257.478	24.998	76.627	0.471
4.1	10	0.4674060	1.731	3.704	0.398	156.876	289.942	24.998	75.325	0.349
5.1	10	0.6542770	2.298	3.512	0.631	156.868	310.783	25.020	74.920	0.262
6.1	10	0.8784630	3.049	3.471	1.000	156.859	324.293	25.015	75.471	0.198

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
7.1	10	1.1567400	4.090	3.536	1.585	156.848	333.216	25.011	77.120	0.149
8.1	10	1.5422000	5.610	3.638	2.512	156.837	339.082	25.007	80.034	0.110
9.1	10	2.0893800	7.846	3.755	3.981	156.824	344.698	25.007	84.962	0.079
10.1	10	2.8556700	11.154	3.906	6.310	156.814	351.624	25.007	93.361	0.056
11.1	10	3.9652200	16.223	4.091	10.000	156.813	358.238	25.007	106.655	0.037
12.1	10	5.5913200	24.077	4.306	15.849	156.834	365.024	24.998	124.520	0.021
13.1	10	7.7947800	36.196	4.644	25.119	156.869	371.717	24.993	142.892	0.010
14.1	10	10.6836000	55.388	5.184	39.811	156.894	378.456	24.993	156.776	0.004
15.1	10	14.7947000	84.996	5.745	63.096	156.904	385.398	24.998	165.815	0.002
16.1	10	19.2044000	130.967	6.820	100.000	156.903	392.200	24.998	171.367	0.001
1.1	11	0.1256070	1.377	10.962	0.100	470.198	126.204	25.002	84.824	1.353
2.1	11	0.2829820	2.113	7.468	0.158	470.198	206.482	25.002	82.432	0.878
3.1	11	0.5757930	3.143	5.459	0.251	470.196	257.322	25.002	79.717	0.586
4.1	11	1.0300600	4.454	4.324	0.398	470.192	289.754	25.007	77.149	0.410
5.1	11	1.6080800	6.037	3.754	0.631	470.185	310.565	25.007	75.397	0.300
6.1	11	2.2397800	7.945	3.547	1.000	470.174	324.012	25.002	74.847	0.227
7.1	11	2.8604600	10.317	3.607	1.585	470.160	332.873	25.002	75.652	0.176
8.1	11	3.4334700	13.451	3.918	2.512	470.142	338.848	25.002	77.927	0.136
9.1	11	3.9968400	17.958	4.493	3.981	470.121	344.448	25.002	81.764	0.103
10.1	11	4.5287400	24.595	5.431	6.310	470.100	351.390	25.002	87.629	0.076
11.1	11	5.5138300	35.555	6.448	10.000	470.088	357.926	24.998	95.310	0.052
12.1	11	7.1691900	53.028	7.397	15.849	470.089	364.868	25.007	105.776	0.034
13.1	11	9.5972900	80.131	8.349	25.119	470.111	371.561	25.002	119.881	0.020
14.1	11	13.5607000	123.574	9.113	39.811	470.151	378.394	25.002	135.602	0.011
15.1	11	19.1389000	191.290	9.995	63.096	470.187	385.164	24.998	150.045	0.005
16.1	11	26.8616000	299.308	11.143	100.000	470.194	391.997	24.993	160.706	0.002
1.1	12	699.6620000	1076.340	1.538	0.100	20299.300	126.313	25.002	56.950	0.063
2.1	12	890.0550000	1196.000	1.344	0.158	20299.300	206.482	24.993	53.316	0.054
3.1	12	1092.3500000	1318.140	1.207	0.251	20299.300	257.338	25.011	50.321	0.047
4.1	12	1284.8100000	1437.890	1.119	0.398	20299.300	289.739	24.998	48.186	0.042
5.1	12	1459.6200000	1563.150	1.071	0.631	20299.300	310.440	25.002	46.927	0.038
6.1	12	1608.4100000	1702.260	1.058	1.000	20299.300	324.090	25.015	46.587	0.035
7.1	12	1726.8500000	1865.000	1.080	1.585	20299.300	332.904	25.007	47.164	0.032
8.1	12	1809.0500000	2060.710	1.139	2.512	20299.300	338.738	25.007	48.683	0.029
9.1	12	1866.7600000	2300.410	1.232	3.981	20299.300	344.479	25.007	50.910	0.027
10.1	12	1820.7500000	2565.890	1.409	6.310	20299.300	351.406	24.998	54.634	0.026
11.1	12	1725.8600000	2929.470	1.697	10.000	20299.300	357.802	24.998	59.558	0.024
12.1	12	1533.0100000	3410.170	2.225	15.849	20299.300	364.759	24.998	66.027	0.022
13.1	12	1228.8800000	4098.770	3.335	25.119	20299.200	371.436	25.002	73.933	0.019
14.1	12	861.8630000	5285.730	6.133	39.811	20299.100	378.378	25.002	82.101	0.015
15.1	12	675.0830000	7636.390	11.312	63.096	20298.900	385.164	24.998	87.397	0.011

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
16.1	12	738.2420000	11760.100	15.930	100.000	20298.100	391.981	24.998	90.434	0.007
1.1	13	0.9234220	7.306	7.912	0.100	1922.840	126.251	25.002	82.803	1.039
2.1	13	2.0539900	11.213	5.459	0.158	1922.840	206.482	24.989	79.630	0.671
3.1	13	3.7096900	16.005	4.314	0.251	1922.840	257.431	25.002	76.968	0.466
4.1	13	5.6620000	21.745	3.841	0.398	1922.840	289.754	24.998	75.439	0.341
5.1	13	7.6861700	28.881	3.758	0.631	1922.830	310.502	24.993	75.162	0.256
6.1	13	9.6475900	37.586	3.896	1.000	1922.820	324.090	25.007	75.730	0.197
7.1	13	11.3505000	49.862	4.393	1.585	1922.810	332.873	25.002	77.417	0.150
8.1	13	12.7302000	66.836	5.250	2.512	1922.790	338.723	24.998	79.676	0.113
9.1	13	14.0052000	94.402	6.741	3.981	1922.780	344.464	25.007	82.391	0.080
10.1	13	17.1700000	140.031	8.156	6.310	1922.780	351.421	24.998	84.426	0.054
11.1	13	22.8906000	216.921	9.476	10.000	1922.770	357.880	24.998	86.283	0.035
12.1	13	33.1559000	341.241	10.292	15.849	1922.770	364.790	25.007	88.145	0.022
13.1	13	48.2524000	531.254	11.010	25.119	1922.770	371.498	25.002	90.785	0.014
14.1	13	73.1907000	820.982	11.217	39.811	1922.760	378.409	24.998	94.618	0.009
15.1	13	110.7390000	1267.720	11.448	63.096	1922.740	385.336	24.989	100.708	0.006
16.1	13	175.6960000	1987.920	11.315	100.000	1922.680	392.028	24.998	109.519	0.004
1.1	14	7.8759900	117.812	14.958	0.100	23339.700	126.204	25.002	86.173	0.787
2.1	14	13.1353000	159.768	12.163	0.158	23339.700	206.560	25.011	85.297	0.579
3.1	14	32.2514000	242.583	7.522	0.251	23339.700	257.462	25.002	82.423	0.379
4.1	14	76.0537000	369.431	4.858	0.398	23339.700	289.786	24.976	78.361	0.246
5.1	14	156.4800000	527.012	3.368	0.631	23339.700	310.487	24.993	73.454	0.169
6.1	14	278.4610000	706.104	2.536	1.000	23339.700	323.981	24.993	68.468	0.122
7.1	14	435.4210000	890.159	2.044	1.585	23339.700	332.810	25.002	63.926	0.094
8.1	14	612.4300000	1068.110	1.744	2.512	23339.600	338.692	25.011	60.169	0.075
9.1	14	800.4570000	1243.080	1.553	3.981	23339.600	344.479	25.002	57.239	0.063
10.1	14	960.3170000	1402.770	1.461	6.310	23339.600	351.452	24.998	55.671	0.055
11.1	14	1107.3200000	1574.870	1.422	10.000	23339.600	357.926	25.015	55.068	0.048
12.1	14	1216.3100000	1759.030	1.446	15.849	23339.500	364.712	24.989	55.789	0.044
13.1	14	1284.6200000	1975.250	1.538	25.119	23339.400	371.358	25.002	58.066	0.040
14.1	14	1296.5300000	2240.700	1.728	39.811	23339.200	378.206	25.007	62.644	0.037
15.1	14	1245.6000000	2581.390	2.072	63.096	23338.800	385.008	24.985	70.830	0.034
16.1	14	1157.9200000	3009.650	2.599	100.000	23337.600	391.794	25.015	84.697	0.031
1.1	15	0.0022603	0.029	12.839	0.100	11.472	126.329	24.998	87.289	1.571
2.1	15	0.0037748	0.045	12.027	0.158	11.471	206.575	24.993	88.046	1.005
3.1	15	0.0081010	0.071	8.717	0.251	11.470	257.525	24.998	87.965	0.646
4.1	15	0.0170856	0.109	6.369	0.398	11.469	289.879	25.011	88.400	0.419
5.1	15	0.0326647	0.161	4.936	0.631	11.466	310.830	25.002	90.929	0.283
6.1	15	0.0580799	0.235	4.046	1.000	11.462	324.262	24.993	97.405	0.192
7.1	15	0.1004780	0.341	3.394	1.585	11.463	333.091	25.015	109.703	0.126
8.1	15	0.1685570	0.490	2.905	2.512	11.476	339.128	25.007	128.576	0.073

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
9.1	15	0.2672670	0.700	2.620	3.981	11.496	344.776	25.011	148.382	0.034
10.1	15	0.4181410	1.001	2.395	6.310	11.508	351.546	25.002	162.151	0.014
11.1	15	0.6035370	1.448	2.399	10.000	11.513	358.004	25.011	170.054	0.005
12.1	15	0.8642430	2.140	2.477	15.849	11.515	364.775	24.985	174.288	0.002
13.1	15	1.6254400	3.140	1.932	25.119	11.515	371.405	24.976	176.690	0.001
14.1	15	2.3455700	4.727	2.015	39.811	11.516	378.300	24.985	178.040	0.000
15.1	15	3.3458200	7.567	2.262	63.096	11.516	385.133	24.998	178.760	0.000
16.1	15	8.7907400	11.994	1.364	100.000	11.515	391.966	25.007	179.217	0.000
1.1	17	0.0014694	0.017	11.783	0.100	8.093	126.251	25.007	88.071	1.859
2.1	17	0.0025317	0.027	10.711	0.158	8.092	206.497	24.998	89.354	1.187
3.1	17	0.0053070	0.042	7.944	0.251	8.091	257.353	24.998	90.387	0.764
4.1	17	0.0106691	0.065	6.053	0.398	8.089	289.801	25.002	92.993	0.498
5.1	17	0.0195630	0.098	4.998	0.631	8.088	310.612	24.998	99.132	0.325
6.1	17	0.0336935	0.146	4.344	1.000	8.091	324.262	25.002	110.572	0.206
7.1	17	0.0555794	0.217	3.911	1.585	8.103	333.076	24.998	127.537	0.118
8.1	17	0.0864211	0.320	3.706	2.512	8.122	338.972	24.998	145.885	0.057
9.1	17	0.1207090	0.473	3.919	3.981	8.136	344.666	24.993	159.774	0.024
10.1	17	0.1980140	0.684	3.453	6.310	8.141	351.593	24.998	168.397	0.010
11.1	17	0.2775810	1.007	3.628	10.000	8.144	358.114	25.002	173.309	0.004
12.1	17	0.4806950	1.506	3.134	15.849	8.144	364.900	24.998	176.043	0.001
13.1	17	0.9294010	2.339	2.517	25.119	8.145	371.530	25.002	177.564	0.001
14.1	17	2.2749600	3.699	1.626	39.811	8.145	378.472	24.993	178.467	0.000
15.1	17	5.4539900	5.784	1.060	63.096	8.145	385.304	25.007	179.046	0.000
16.1	17	11.1251000	11.206	1.007	100.000	8.144	392.059	25.002	179.266	0.000
1.1	20	125.5850000	802.461	6.38978	0.1	81463.7	126.266	25.011	81.0872	0.399087
2.1	20	282.4170000	1198.61	4.2441	0.15849	81463.7	206.388	24.998	76.7145	0.263247
3.1	20	557.9100000	1700.85	3.04861	0.251189	81463.7	257.182	25.011	71.801	0.181119
4.1	20	899.2150000	2228.97	2.47879	0.398107	81463.7	289.583	24.993	67.9794	0.134907
5.1	20	1256.8000000	2777.81	2.21022	0.630957	81463.7	310.268	25.007	65.5934	0.106365
6.1	20	1601.3400000	3349.44	2.09166	1	81463.6	323.825	24.998	64.3731	0.0873638
7.1	20	1941.5100000	3985.77	2.05292	1.5849	81463.6	332.654	25.007	63.9408	0.0731682
8.1	20	2203.6000000	4678.37	2.12306	2.51189	81463.6	338.551	24.998	64.6779	0.0627363
9.1	20	2346.4900000	5458	2.32603	3.98105	81463.6	344.261	24.993	66.6246	0.0546145
10.1	20	2259.7600000	6294.2	2.78534	6.30957	81463.6	351.031	24.989	70.136	0.0485204
11.1	20	2002.3900000	7437.35	3.71423	10.0001	81463.5	357.677	24.993	74.8256	0.0421295
12.1	20	1456.0100000	9040.82	6.20931	15.849	81463.5	364.51	24.989	80.7826	0.0354306
13.1	20	834.6890000	11847.2	14.1936	25.1188	81463.4	371.077	24.993	85.9686	0.0273104
14.1	20	665.5850000	17714.6	26.615	39.8105	81463.2	377.832	24.989	87.8973	0.0182957
15.1	20	831.9660000	27199.1	32.6925	63.0957	81462.4	384.712	24.993	88.3693	0.0119213
16.1	20	1261.8700000	42850.7	33.9581	100	81459.3	391.482	24.993	88.5191	0.00757108
1.1	21	131.5940000	1146.79	8.71467	0.1	162469	126.204	25.007	83.4279	0.56005

Point	sample	Storage modulus	Loss modulus	Tan(delta)	Angular frequency	Oscillation torque	Step time	Temperature	Raw phase	Oscillation displacement
2.1	21	156.6940000	1293.14	8.25266	0.15849	162469	206.497	25.007	83.0617	0.496302
3.1	21	257.6080000	1677.02	6.50998	0.251189	162469	257.384	25.015	81.2291	0.381041
4.1	21	409.6580000	2165.97	5.28726	0.398107	162469	289.739	24.993	79.241	0.293304
5.1	21	642.6010000	2791.88	4.34465	0.630957	162469	310.502	25.007	76.9753	0.225703
6.1	21	987.9830000	3595.85	3.63959	1	162469	323.996	24.989	74.5563	0.17342
7.1	21	1428.9600000	4522.93	3.16519	1.5849	162469	332.717	25.002	72.3665	0.136365
8.1	21	1921.2300000	5553.45	2.89057	2.51189	162469	338.645	25.011	70.7957	0.110095
9.1	21	2418.1500000	6757.67	2.79456	3.98105	162469	344.354	24.993	70.1678	0.0901605
10.1	21	2723.9900000	7991.23	2.93365	6.30957	162469	351.047	24.989	71.0181	0.0766615
11.1	21	2924.0300000	9588.48	3.2792	10.0001	162469	357.63	25.002	72.8712	0.064579
12.1	21	2802.0900000	11561.2	4.12593	15.849	162469	364.588	25.024	76.217	0.0544268
13.1	21	2272.6200000	14413.2	6.34213	25.1188	162469	371.155	25.002	80.9281	0.044371
14.1	21	1400.5300000	19909.4	14.2156	39.8105	162469	377.972	24.989	85.9243	0.0324252
15.1	21	1174.4000000	33918.5	28.8814	63.0957	162467	384.914	24.976	87.8388	0.0190662
16.1	21	1753.6300000	61326.2	34.9711	100	162461	391.622	25.007	87.7926	0.0105536
1.1	18	1.9289500	27.5163	14.2649	0.1	11026.1	126.298	24.989	85.9912	1.59048
2.1	18	5.7271500	43.4578	7.58804	0.15849	11026.1	206.575	24.993	82.4943	1.00087
3.1	18	12.5722000	64.0723	5.09633	0.251189	11026.1	257.384	24.993	78.9018	0.671919
4.1	18	23.5395000	89.6821	3.80985	0.398107	11026.1	289.754	24.998	75.2992	0.473183
5.1	18	39.2816000	120.474	3.06693	0.630957	11026.1	310.596	25.002	71.9534	0.346253
6.1	18	57.6211000	154.952	2.68915	1	11026.1	324.137	25.011	69.6268	0.26543
7.1	18	78.6016000	194.407	2.47332	1.5849	11026.1	333.044	24.993	68.0376	0.209304
8.1	18	101.5760000	241.936	2.38181	2.51189	11026.1	338.832	24.998	67.3319	0.167341
9.1	18	121.4040000	294.057	2.42214	3.98105	11026	344.542	25.02	67.7933	0.138139
10.1	18	132.7950000	350.588	2.64008	6.30957	11026	351.499	24.998	69.7512	0.117414
11.1	18	136.5450000	421.998	3.09054	10.0001	11025.9	357.895	24.993	73.1537	0.0995088
12.1	18	131.0470000	517.819	3.95141	15.849	11025.8	364.744	25.015	78.1182	0.0829146
13.1	18	123.0900000	671.484	5.45524	25.1188	11025.7	371.342	24.998	84.2608	0.0650115
14.1	18	125.7760000	941.878	7.48851	39.8105	11025.6	378.331	24.985	90.8513	0.046576
15.1	18	147.1820000	1406.18	9.55406	63.0957	11025.5	385.164	25.007	98.2556	0.0308776
16.1	18	195.0790000	2177.29	11.161	100	11025.1	391.934	25.015	107.518	0.0192163

Appendix C - Frequency Sweep Graphs from TRIOS

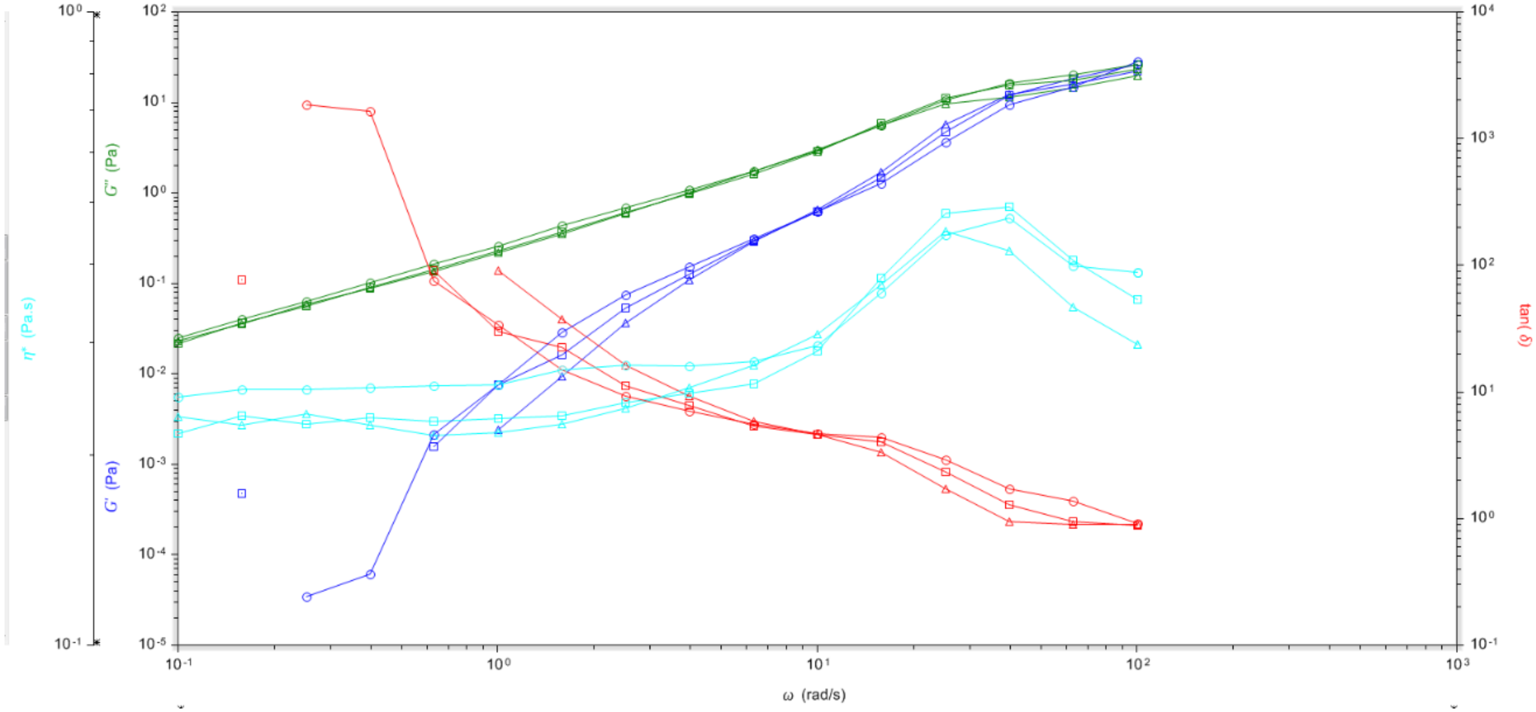

Figure C.1 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 1

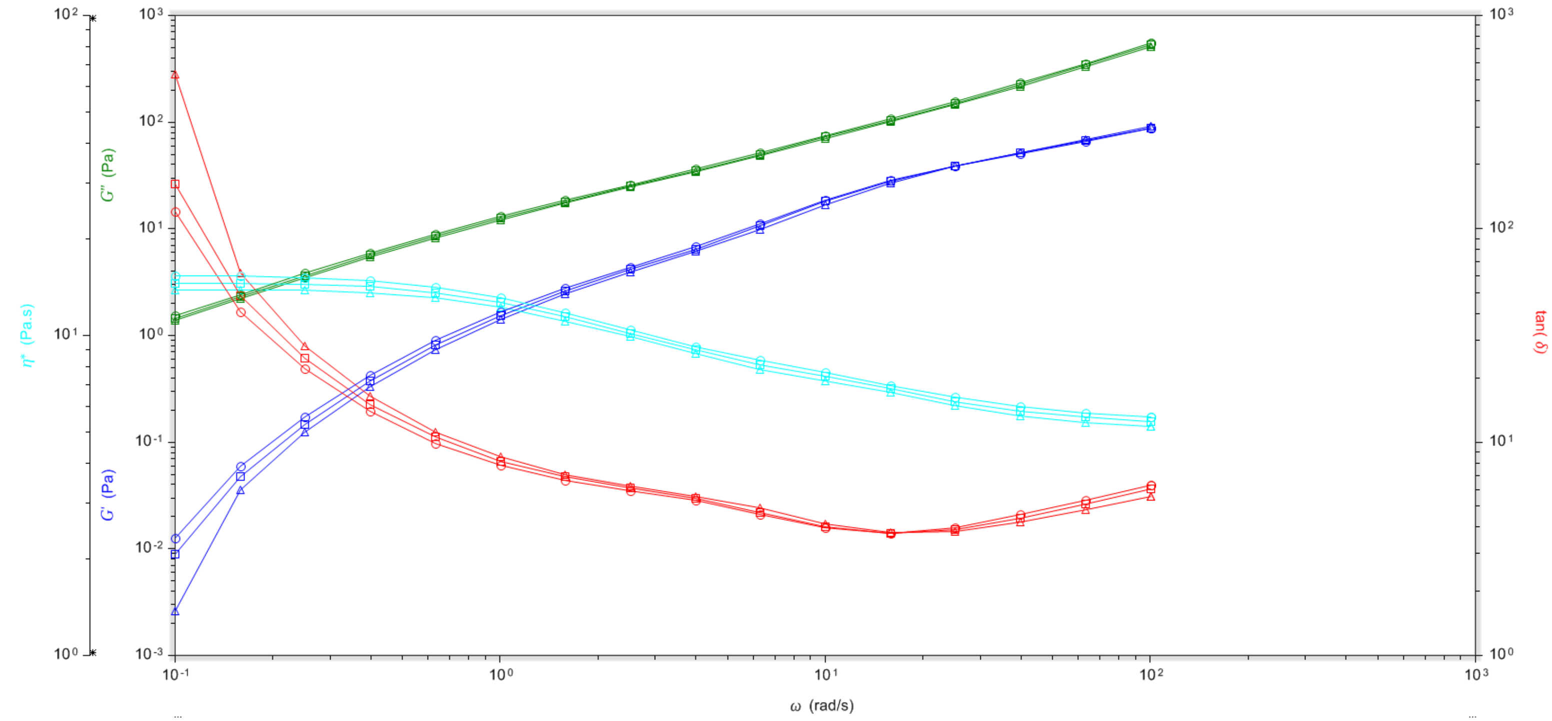

Figure C.2 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 2

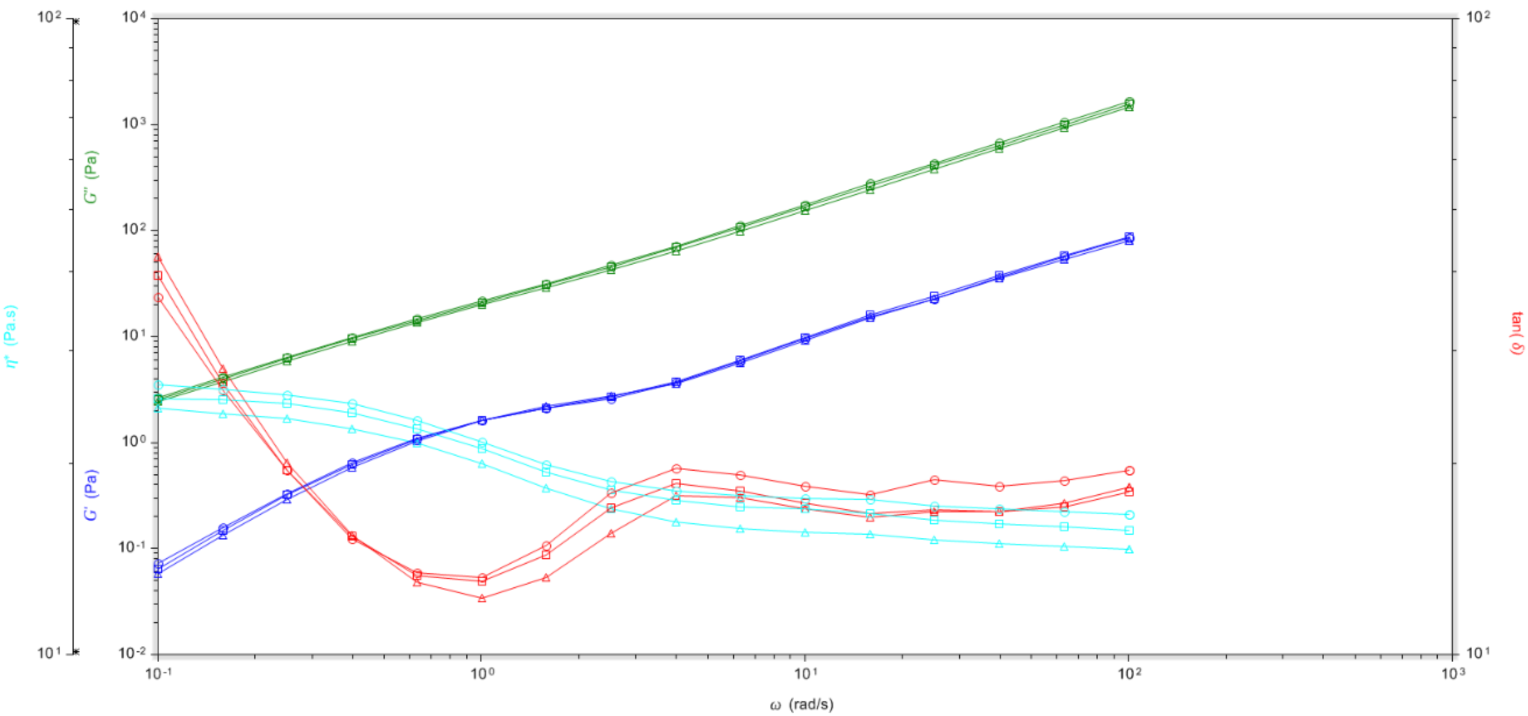

Figure C.3 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 3

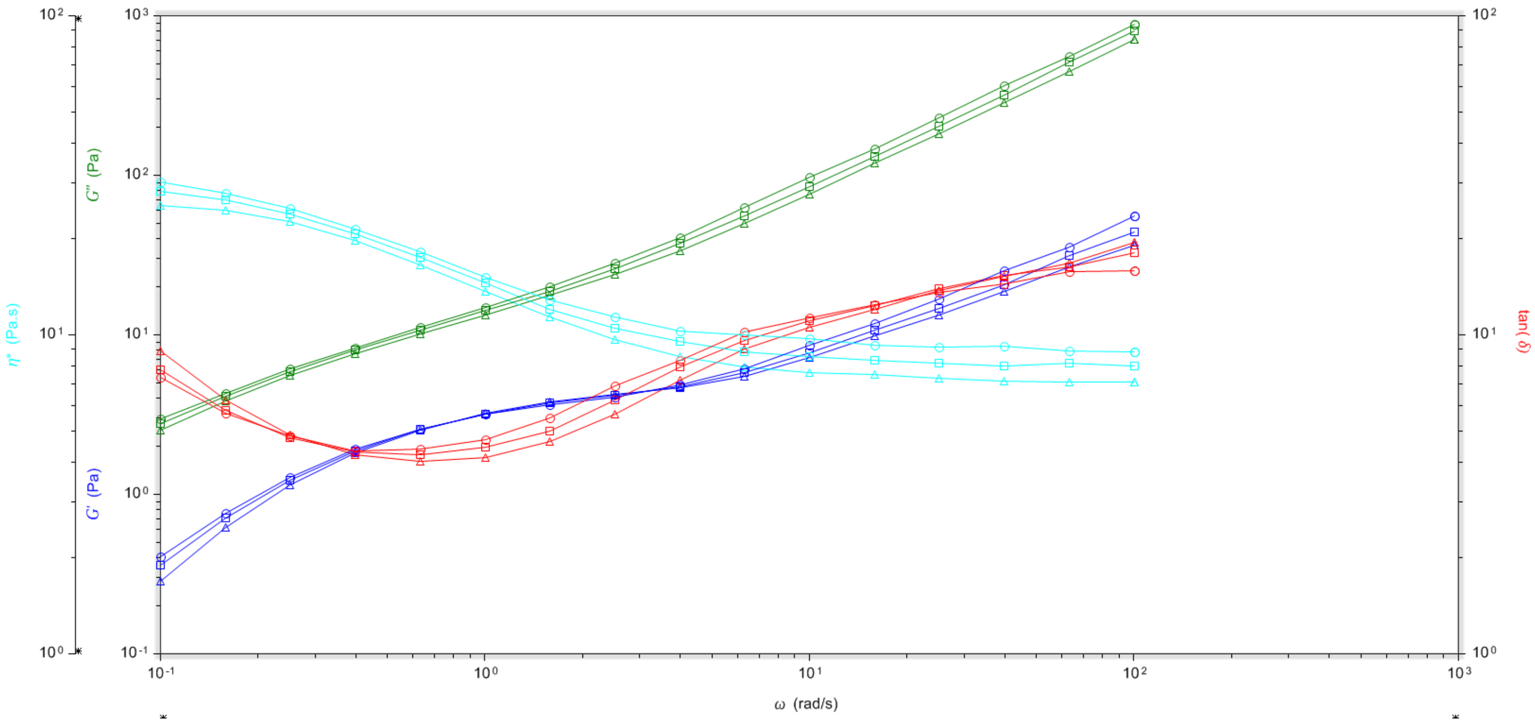

Figure C.4 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 4

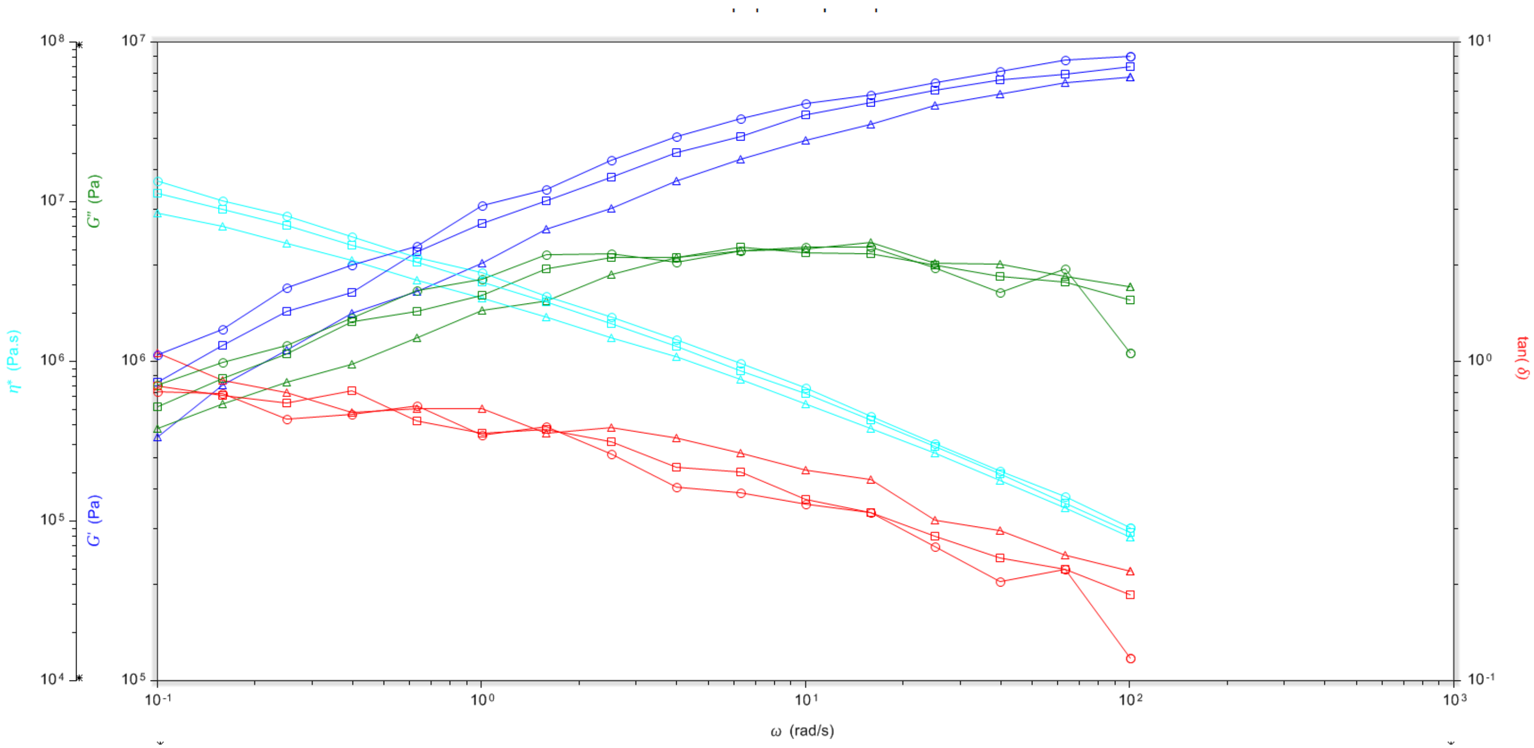

Figure C.5 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 5

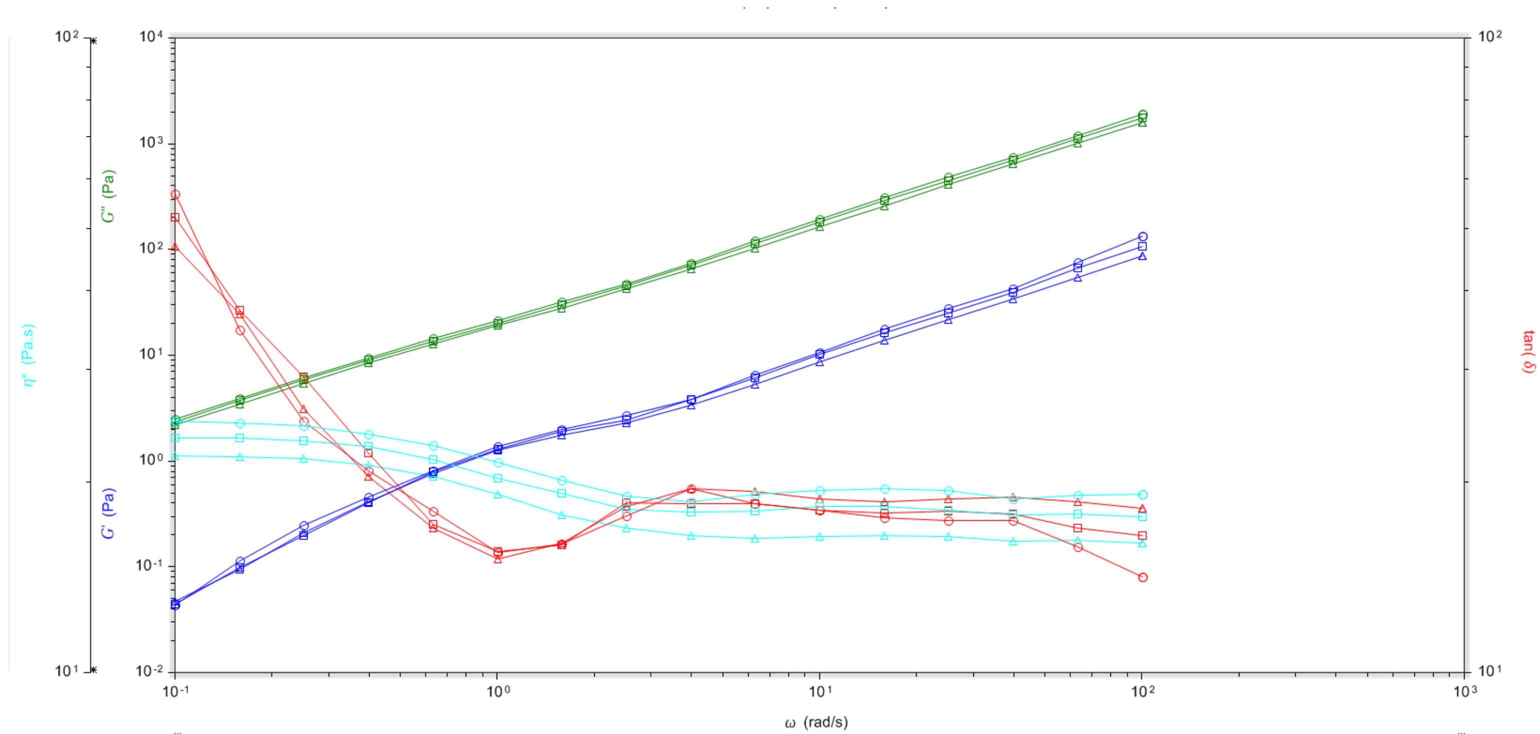

Figure C.6 G’, G”, Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 6

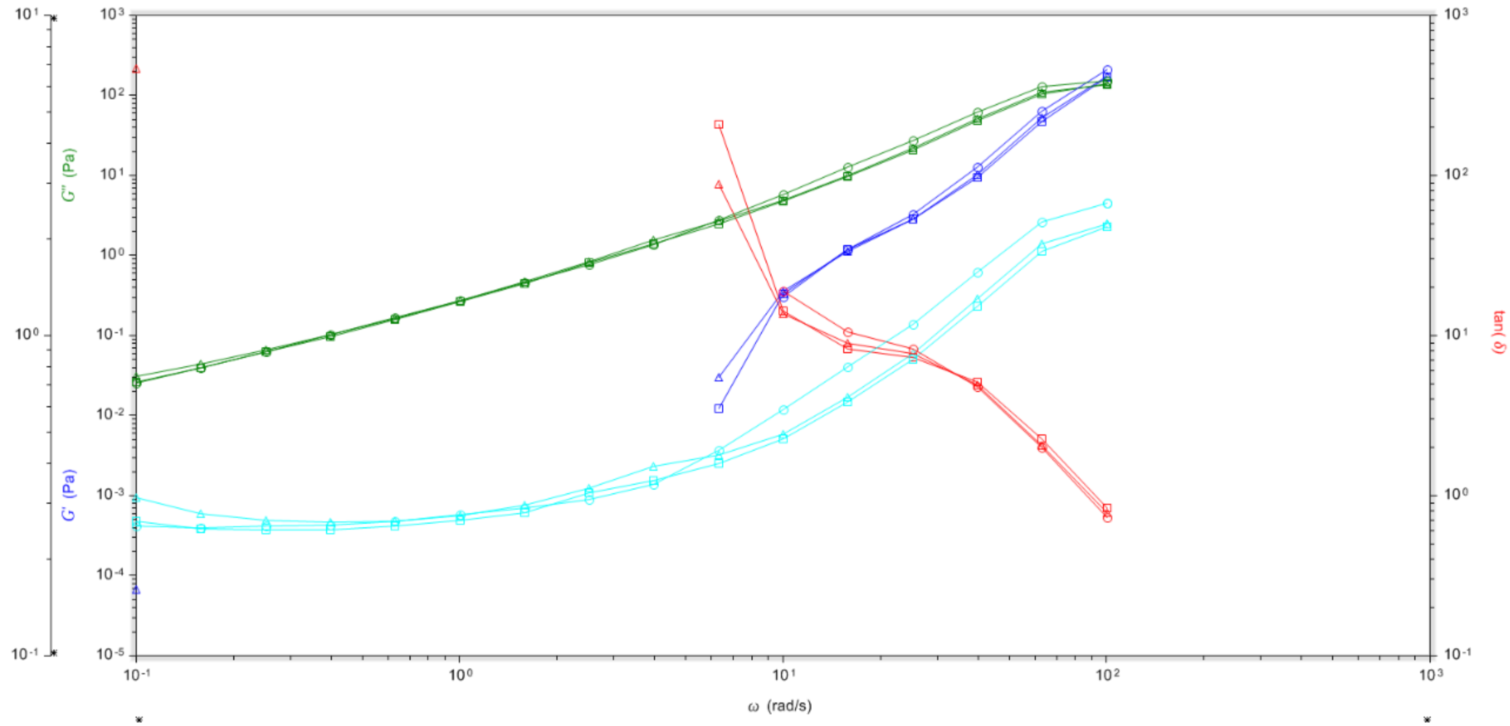

Figure C.7 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 9

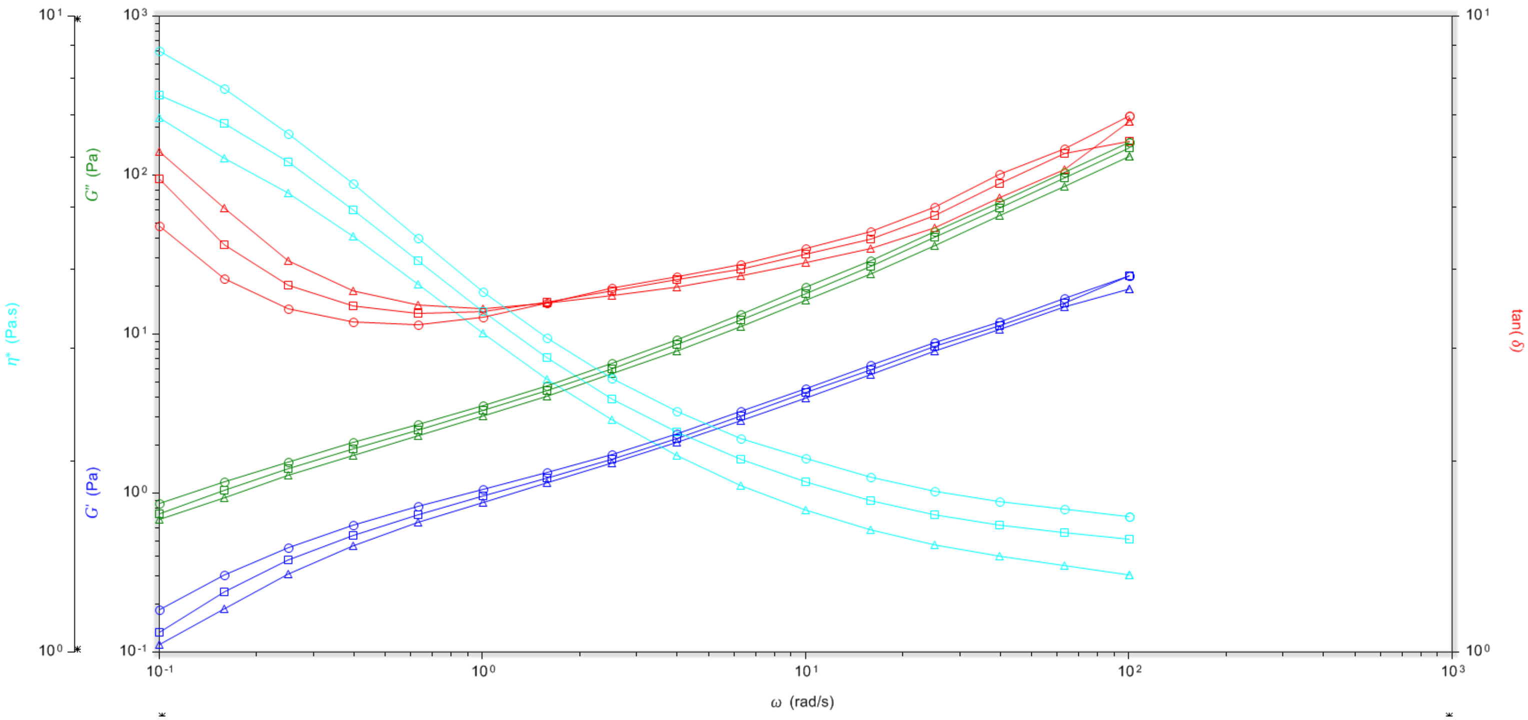

Figure C.8 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 10

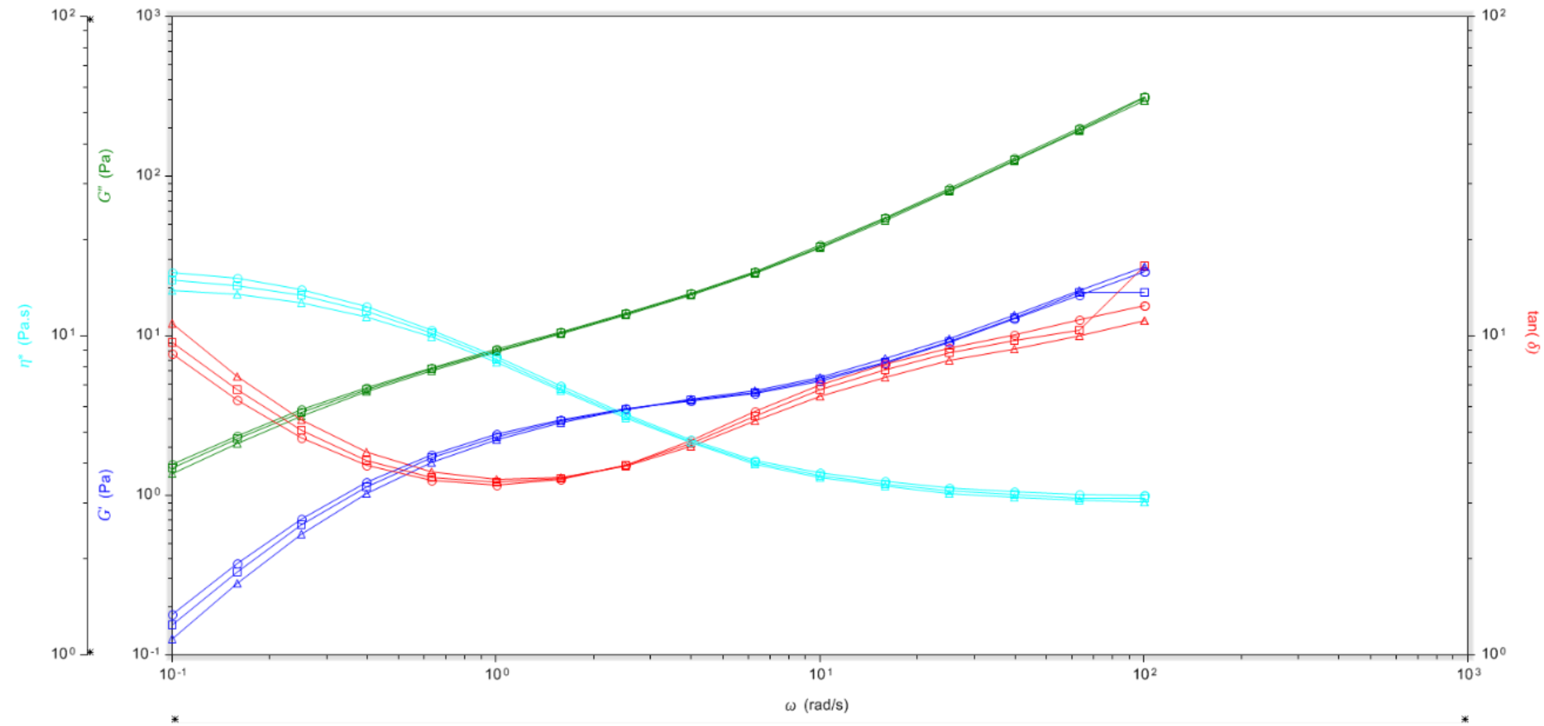

Figure C.9 G’, G”, Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 11

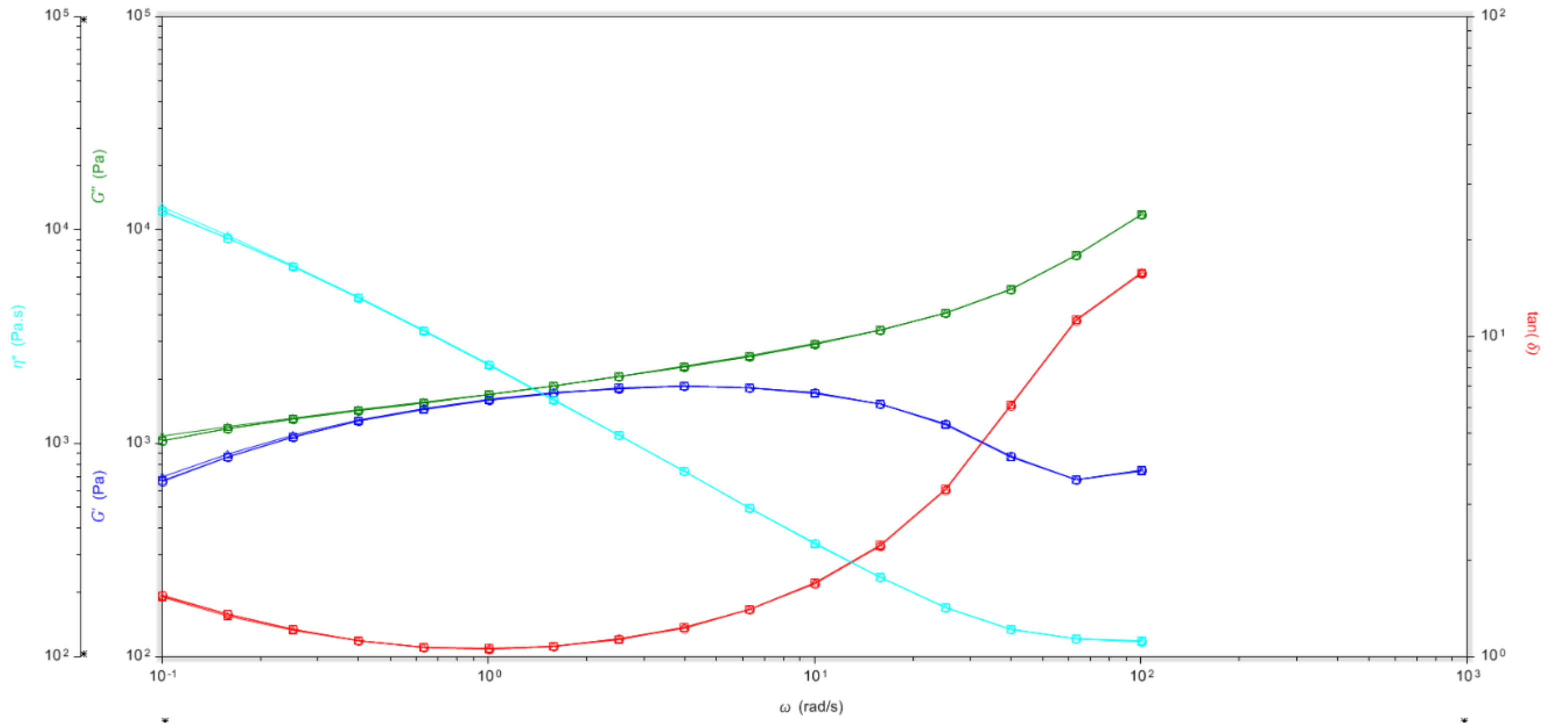

Figure C.10 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 12

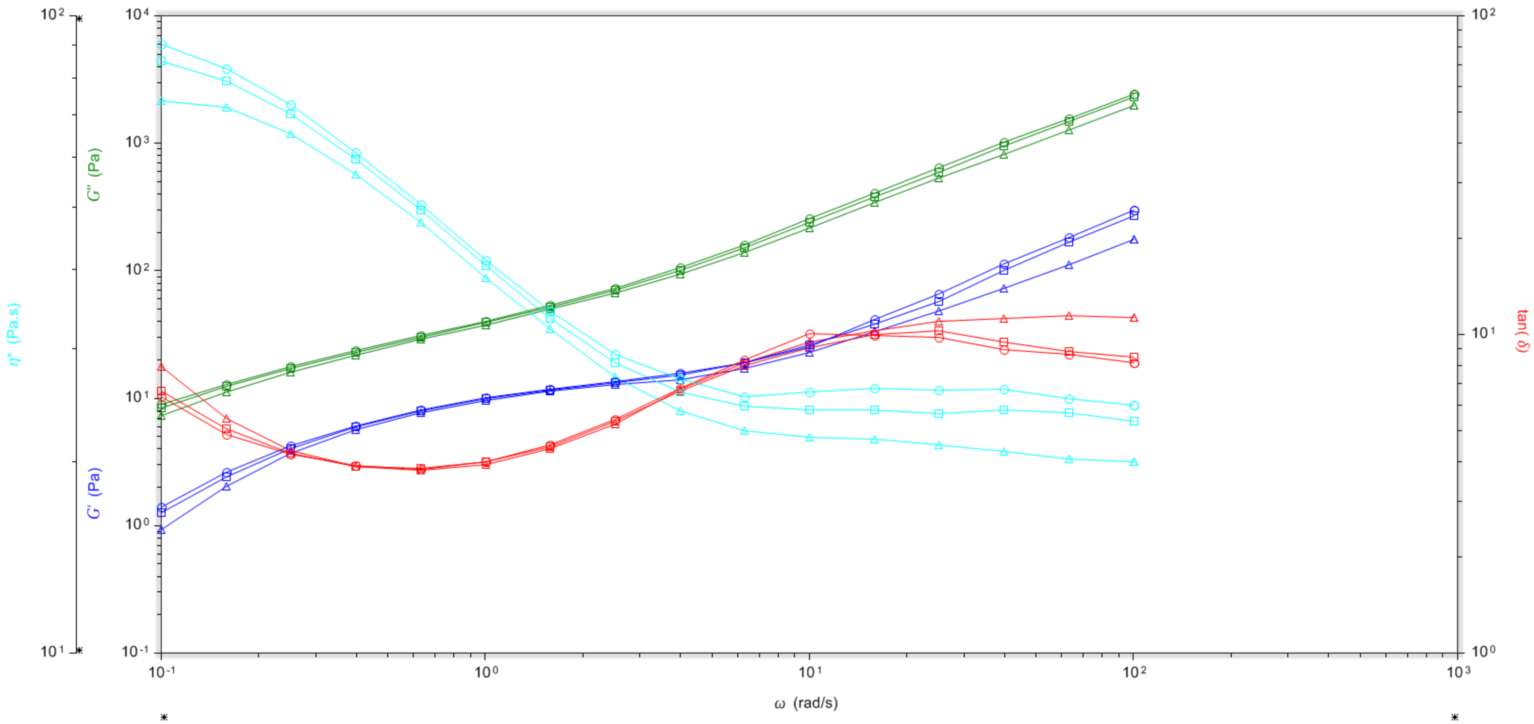

Figure C.11 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 13

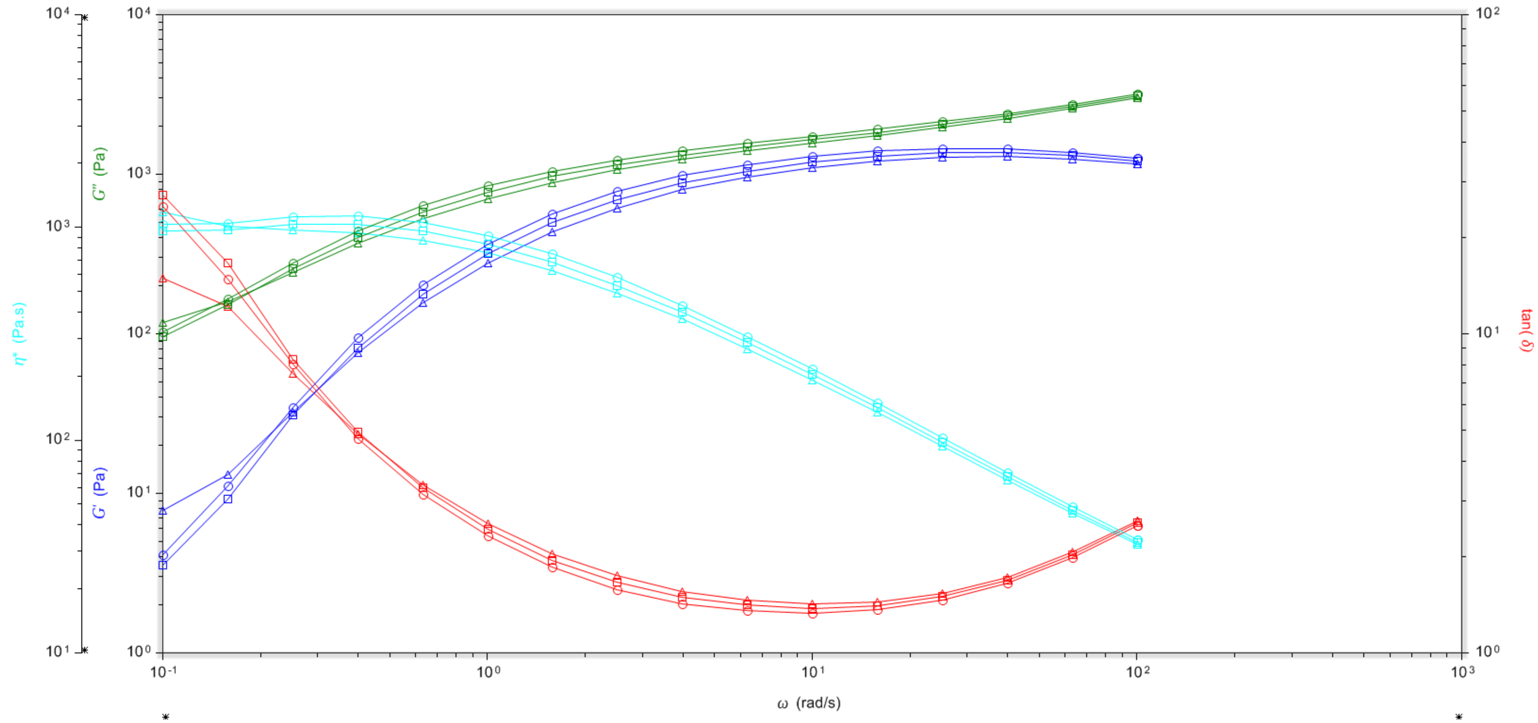

Figure C.12 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 14

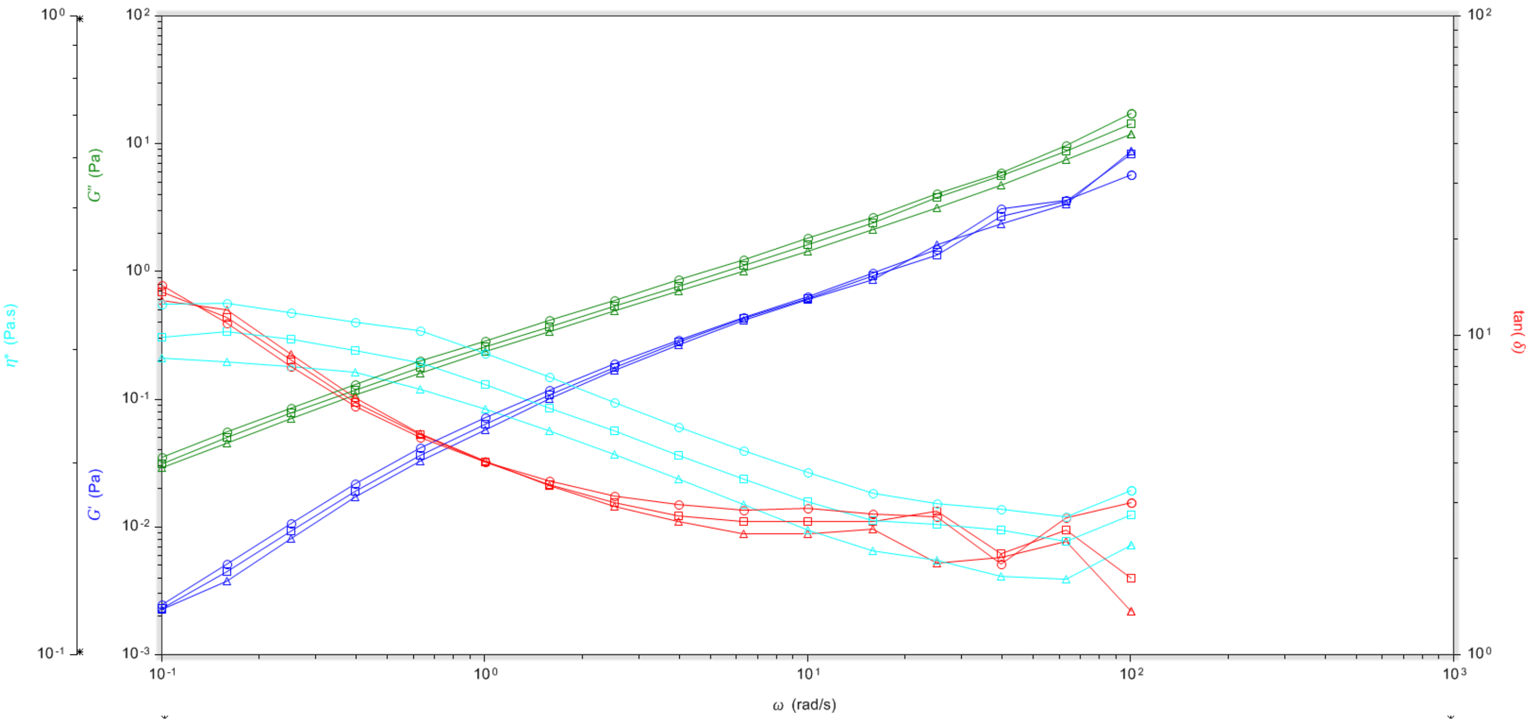

Figure C.13 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 15

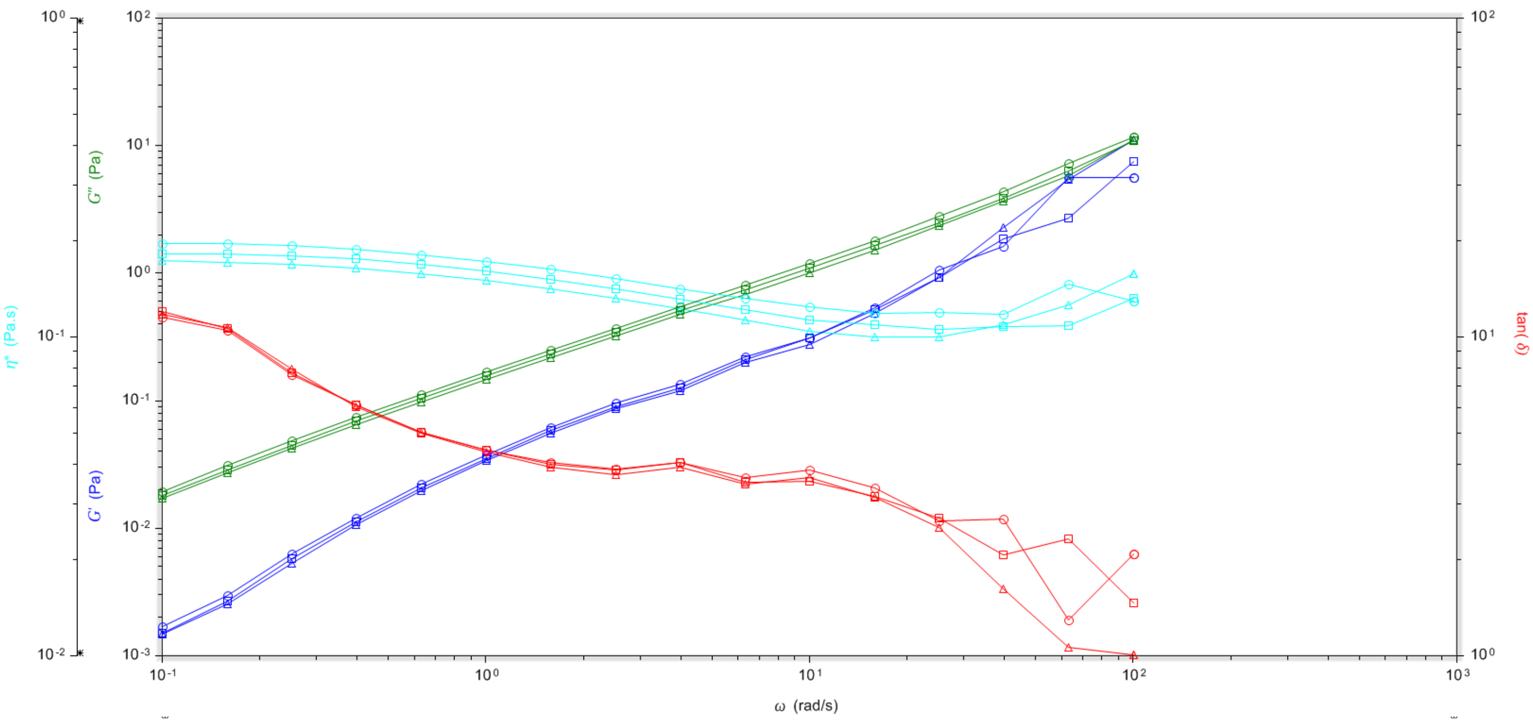

Figure C.14 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 17

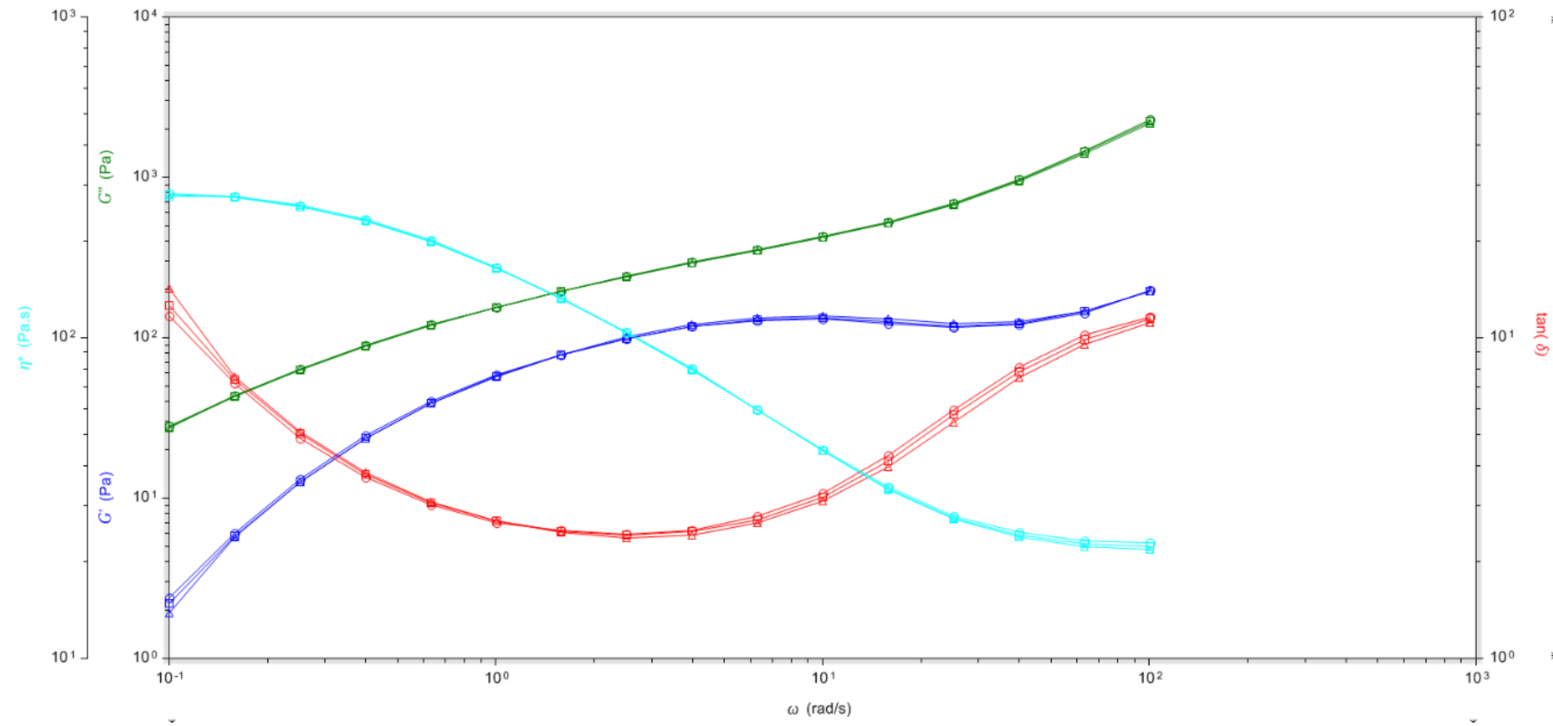

Figure C.15 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 18

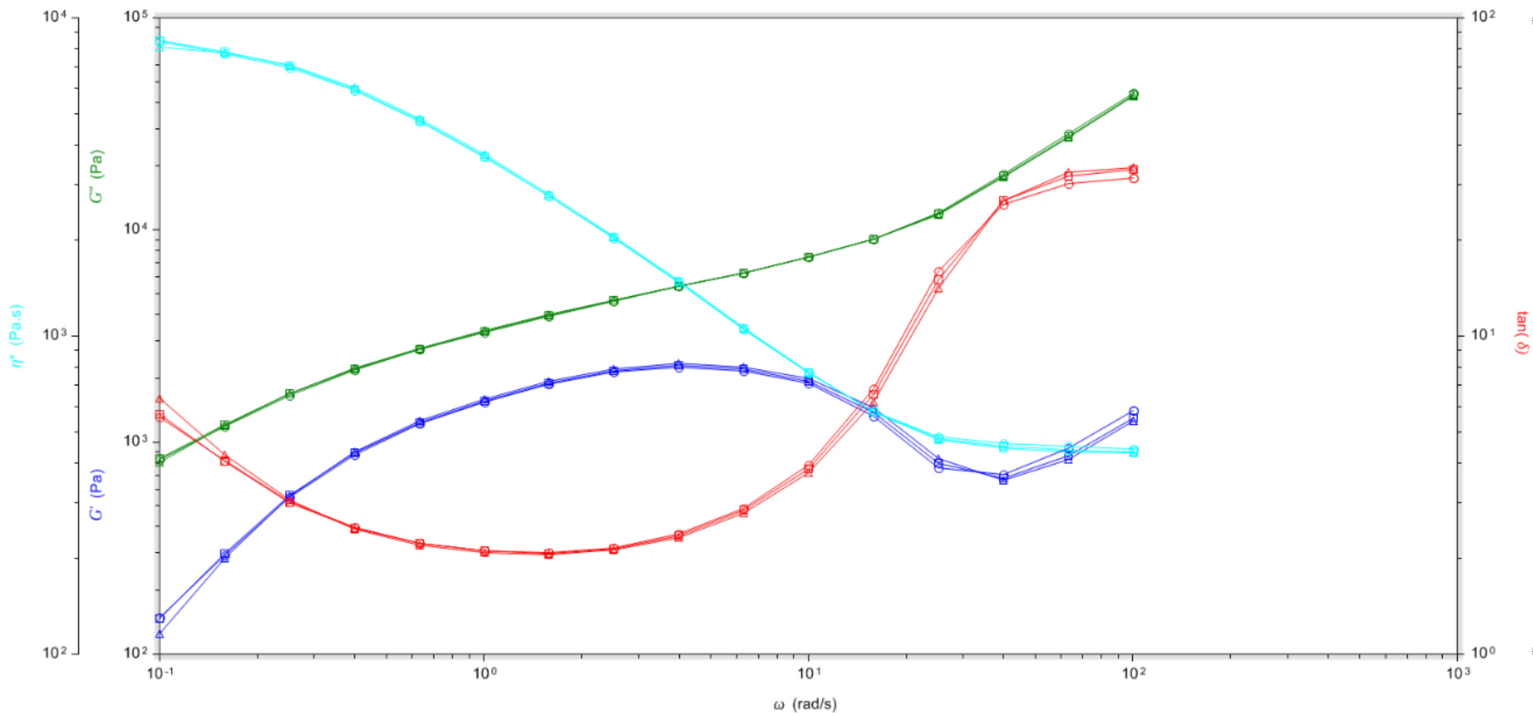

Figure C.16 G', G", Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 20

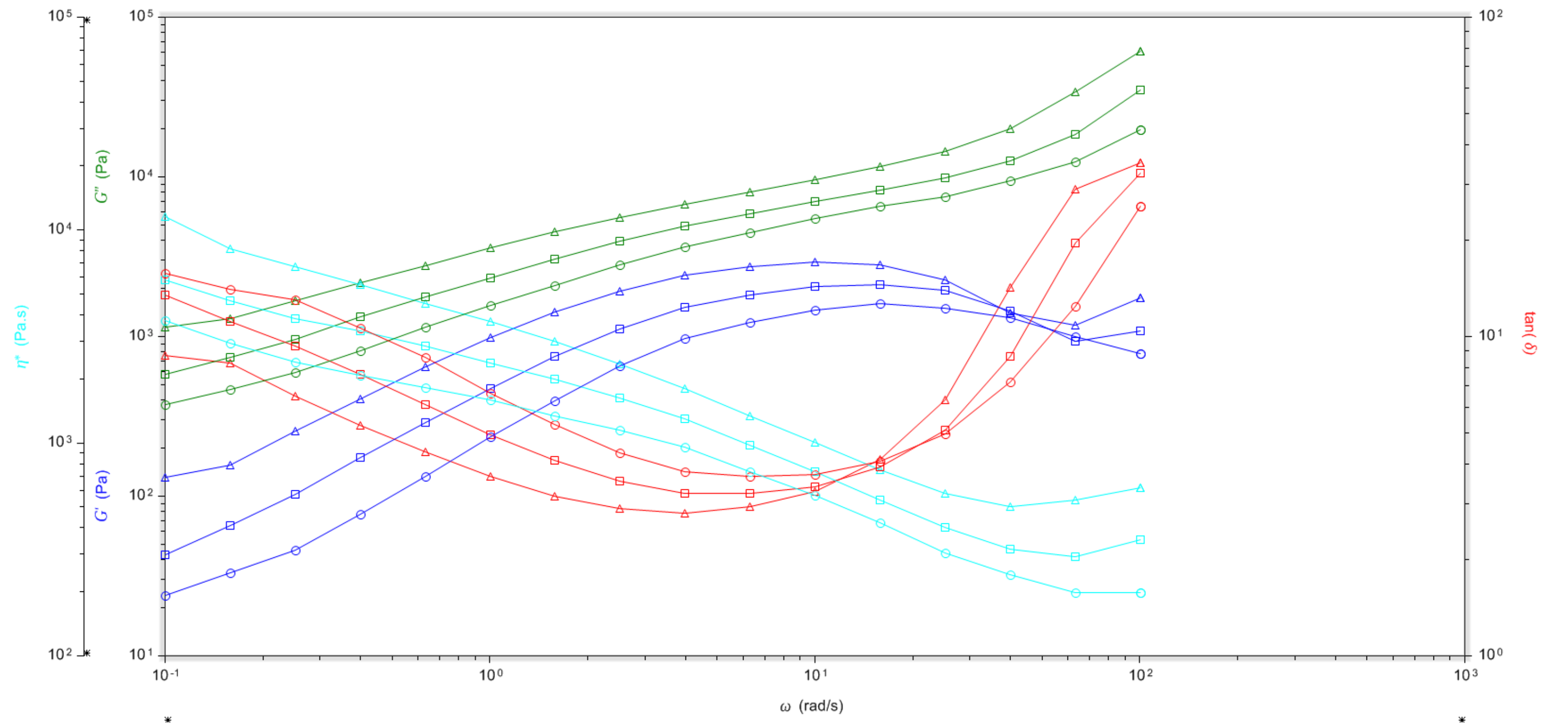

Figure C.17 G’, G”, Complex Viscosity, and tan (δ) vs. Frequency (rad/s) for Sample 21

Appendix D - Raw Texture Data from Exponent Stable Micro Systems Software Macro

Test ID	1.0 mm Force	Peak Force	dpkmx	F2mm	t-2mm	d-2mm	work of compression	Loss in pk force at 2-mm	negative peak force	negative area
	g	g	mm	g	sec	mm	g.mm	g	g	g.mm
	1.0 mm Force	Hardness	dpkmx	F2mm	t-2mm	d-2mm	Area F-D 1:2	ratio	negative peak force	Area F-D 3:4
2_12_16SAMPLE 09-1	6.296	12.243	0.022	2.309	0.418	2.000	9.773	0.189	-15.881	-8.380
2_12_16SAMPLE 09-2	4.547	10.144	0.022	3.218	0.418	2.000	8.894	0.317	-15.881	-4.002
2_12_16SAMPLE 09-3	3.988	11.263	0.029	2.588	0.418	2.000	7.928	0.230	-16.091	-5.853
2_12_16SAMPLE 09-4	4.407	11.194	0.022	2.379	0.418	2.000	8.651	0.213	-16.021	-5.620
2_12_16SAMPLE 09-5	4.198	13.782	0.022	1.259	0.418	2.000	8.393	0.091	-17.000	-5.113
2_12_16SAMPLE 20-1	305.583	619.421	1.960	530.852	0.418	2.000	622.728	0.857	-18.889	-4.292
2_12_16SAMPLE 20-2	132.853	383.378	1.951	318.385	0.418	2.000	311.701	0.830	-15.531	-4.904
2_12_16SAMPLE 20-3	421.436	888.765	1.960	780.537	0.418	2.000	857.247	0.878	-7.765	-2.898
2_12_16SAMPLE 20-4	305.723	658.948	1.951	556.457	0.418	2.000	628.316	0.844	-23.856	-5.471
2_12_16SAMPLE 20-5	154.470	414.789	1.940	347.349	0.418	2.000	352.451	0.837	-10.844	-3.559
2_12_16SAMPLE 22-1	11787.604	23517.772	1.984	23397.791	0.418	2.000	22966.899	0.995	-87.939	-21.719
2_12_16SAMPLE 22-2	4035.400	14993.005	2.000	14993.005	0.418	2.000	10759.626	1.000	-154.960	-4.663
2_12_16SAMPLE 22-3	2677.347	17372.326	2.000	17309.502	0.418	2.000	9778.088	0.996	-118.511	-23.530
2_12_16SAMPLE 22-4	7999.511	17519.030	1.984	17434.380	0.418	2.000	16188.762	0.995	-56.457	-12.975
2_12_16SAMPLE 22-5	16836.856	21947.882	1.642	18771.234	0.418	2.000	27728.918	0.855	-280.747	-55.075
2_14_16SAMPLE 01-1	3.218	10.494	0.022	0.280	0.418	2.000	8.243	0.027	-15.881	-25.128
2_14_16SAMPLE 01-2	3.498	10.634	0.022	1.469	0.418	2.000	7.159	0.138	-18.189	-22.998
2_14_16SAMPLE 01-3	4.337	13.992	0.056	0.280	0.418	2.000	7.212	0.020	-18.399	-21.324
2_14_16SAMPLE 01-4	4.058	10.494	0.022	2.379	0.418	2.000	6.311	0.227	-17.420	-22.374
2_14_16SAMPLE 01-5	4.198	11.333	0.022	2.239	0.418	2.000	9.014	0.198	-15.811	-30.767
2_14_16SAMPLE 02-1	3.918	12.383	0.022	1.469	0.418	2.000	7.619	0.119	-16.440	-6.492
2_14_16SAMPLE 02-2	4.058	12.873	0.022	0.840	0.418	2.000	8.275	0.065	-14.342	-5.385
2_14_16SAMPLE 02-3	2.798	10.424	0.022	0.210	0.418	2.000	7.081	0.020	-16.440	-1.112
2_14_16SAMPLE 02-4	4.897	11.473	0.022	2.099	0.418	2.000	7.587	0.183	-15.391	-2.385
2_14_16SAMPLE 02-5	1.539	9.724	0.022	-0.490	0.418	2.000	2.279	-0.050	-22.037	-8.942
2_14_16SAMPLE 03-1	4.198	10.914	0.022	2.029	0.418	2.000	8.089	0.186	-17.280	-75.324
2_14_16SAMPLE 03-2	4.757	13.292	0.022	1.609	0.418	2.000	8.781	0.121	-15.741	-80.989

Test ID	1.0 mm Force	Peak Force	dpkmx	F2mm	t-2mm	d-2mm	work of compression	Loss in pk force at 2-mm	negative peak force	negative area
	g	g	mm	g	sec	mm	g.mm	g	g	g.mm
	1.0 mm Force	Hardness	dpkmx	F2mm	t-2mm	d-2mm	Area F-D 1:2	ratio	negative peak force	Area F-D 3:4
2_14_16SAMPLE 03-3	4.827	10.634	0.022	1.189	0.418	2.000	8.498	0.112	-15.601	-90.047
2_14_16SAMPLE 03-5	6.156	10.914	0.022	0.700	0.418	2.000	7.776	0.064	-17.420	-77.709
2_14_16SAMPLE 03-6	6.366	12.873	0.022	1.959	0.418	2.000	8.100	0.152	-17.700	-83.244
2_14_16SAMPLE 04-1	4.687	10.284	0.022	3.778	0.418	2.000	9.164	0.367	-17.770	-104.714
2_14_16SAMPLE 04-2	5.247	9.864	0.022	2.239	0.418	2.000	9.143	0.227	-16.790	-108.486
2_14_16SAMPLE 04-3	3.568	9.654	0.022	2.588	0.418	2.000	9.162	0.268	-17.350	-112.705
2_14_16SAMPLE 04-4	3.498	10.914	0.022	1.539	0.418	2.000	9.259	0.141	-16.650	-135.814
2_14_16SAMPLE 04-5	3.358	13.712	0.056	3.218	0.418	2.000	8.698	0.235	-15.041	-106.301
2_14_16SAMPLE 05-1	4407.724	10275.781	1.984	10220.513	0.418	2.000	9048.295	0.995	-274.241	-49.189
2_14_16SAMPLE 05-2	11887.786	23448.652	1.990	23422.627	0.418	2.000	22939.685	0.999	-434.728	-248.010
2_14_16SAMPLE 05-3	1472.226	7780.468	1.990	7766.756	0.418	2.000	4842.202	0.998	-386.106	-136.731
2_14_16SAMPLE 05-4	1956.345	6839.304	1.984	6801.876	0.418	2.000	4867.027	0.995	-185.462	-67.736
2_14_16SAMPLE 05-5	5814.748	13892.544	1.984	13790.263	0.418	2.000	12066.613	0.993	-346.579	-283.324
2_14_16SAMPLE 06-1	4.337	9.864	0.037	2.938	0.418	2.000	8.674	0.298	-16.930	-79.625
2_14_16SAMPLE 06-2	2.938	10.564	0.022	2.449	0.418	2.000	7.944	0.232	-17.000	-76.498
2_14_16SAMPLE 06-3	2.519	9.584	0.022	0.700	0.418	2.000	6.429	0.073	-17.070	-68.058
2_14_16SAMPLE 06-4	5.667	13.222	0.056	0.350	0.418	2.000	9.110	0.026	-17.700	-82.052
2_14_16SAMPLE 06-5	2.868	9.095	0.022	1.889	0.418	2.000	7.087	0.208	-17.490	-71.937
2_14_16SAMPLE 07-1	2776.340	6511.684	1.969	6268.995	0.418	2.000	5725.483	0.963	-118.721	-9.878
2_14_16SAMPLE 07-2	6720.023	9602.631	1.960	9226.529	0.418	2.000	11371.704	0.961	-5.877	-5.278
2_14_16SAMPLE 07-4	974.605	4125.927	1.977	4007.136	0.418	2.000	2981.471	0.971	-124.318	-12.086
2_14_16SAMPLE 07-5	2860.571	8327.901	1.977	8168.533	0.418	2.000	6624.377	0.981	-5.807	-6.119
2_14_16SAMPLE 07-6	4264.867	11016.022	1.977	10755.982	0.418	2.000	9839.719	0.976	-5.457	-7.389
2_14_16SAMPLE 08-1	2149.364	6354.065	1.969	6171.681	0.418	2.000	5064.283	0.971	-7.556	-3.059
2_14_16SAMPLE 08-2	2437.666	6693.019	1.969	6501.400	0.418	2.000	5387.820	0.971	-5.807	-5.225
2_14_16SAMPLE 08-3	5241.430	7406.605	1.960	7053.520	0.418	2.000	9526.847	0.952	-5.177	-5.674
2_14_16SAMPLE 08-4	3344.830	6122.499	1.960	5852.876	0.418	2.000	6504.064	0.956	-4.897	-4.475
2_14_16SAMPLE 08-5	2699.874	5051.211	1.969	4863.929	0.418	2.000	5296.578	0.963	-4.407	-1.724
2_14_16SAMPLE 10-1	2.938	11.124	0.037	1.889	0.418	2.000	7.316	0.170	-17.560	-34.944
2_14_16SAMPLE 10-2	5.457	11.263	0.022	1.469	0.418	2.000	7.617	0.130	-16.021	-27.694

Test ID	1.0 mm Force	Peak Force	dpkmx	F2mm	t-2mm	d-2mm	work of compression	Loss in pk force at 2-mm	negative peak force	negative area
	g	g	mm	g	sec	mm	g.mm	g	g	g.mm
	1.0 mm Force	Hardness	dpkmx	F2mm	t-2mm	d-2mm	Area F-D 1:2	ratio	negative peak force	Area F-D 3:4
2_14_16SAMPLE 10-3	1.469	10.144	-52.838	0.630	0.418	2.000	5.347	0.062	-18.189	-18.683
2_14_16SAMPLE 10-4	5.387	10.914	0.037	1.119	0.418	2.000	7.808	0.103	-17.490	-29.355
2_14_16SAMPLE 10-5	4.687	13.292	0.022	2.658	0.418	2.000	6.540	0.200	-17.350	-22.333
2_14_16SAMPLE 10-6	3.778	11.333	0.022	2.728	0.418	2.000	8.646	0.241	-15.811	-30.719
2_14_16SAMPLE 11-1	5.527	10.914	0.029	2.868	0.418	2.000	8.827	0.263	-16.021	-51.444
2_14_16SAMPLE 11-3	5.387	14.062	0.022	1.119	0.418	2.000	9.154	0.080	-16.231	-49.029
2_14_16SAMPLE 11-4	4.687	10.774	0.022	3.218	0.418	2.000	8.474	0.299	-15.741	-50.841
2_14_16SAMPLE 11-5	1.889	12.173	0.022	0.420	0.418	2.000	6.300	0.034	-18.259	-47.501
2_14_16SAMPLE 11-6	3.498	11.473	0.022	0.770	0.418	2.000	7.796	0.067	-17.000	-51.678
2_14_16SAMPLE 12-2	470.127	1588.499	1.977	1511.753	0.418	2.000	1157.304	0.952	-138.660	-194.422
2_14_16SAMPLE 12-3	1069.050	2559.676	1.969	2459.983	0.418	2.000	2265.714	0.961	-392.682	-586.838
2_14_16SAMPLE 12-4	392.612	1541.906	1.969	1465.160	0.418	2.000	1056.796	0.950	-206.660	-289.779
2_14_16SAMPLE 12-5	131.384	811.110	1.969	756.961	0.418	2.000	481.822	0.933	-124.738	-228.313
2_14_16SAMPLE 12-6	267.525	1391.563	1.969	1309.710	0.418	2.000	870.399	0.941	-291.031	-504.009
2_14_16SAMPLE 12-7	329.439	1422.135	1.969	1329.229	0.418	2.000	957.654	0.935	-356.093	-813.909
2_14_16SAMPLE 13-1	4.687	11.473	0.029	2.728	0.418	2.000	9.977	0.238	-15.461	-230.873
2_14_16SAMPLE 13-2	7.066	11.683	0.022	1.819	0.418	2.000	9.284	0.156	-17.350	-273.952
2_14_16SAMPLE 13-3	4.407	11.823	0.022	3.358	0.418	2.000	9.533	0.284	-16.580	-291.976
2_14_16SAMPLE 13-4	4.337	12.803	0.022	1.049	0.418	2.000	9.947	0.082	-17.630	-320.522
2_14_16SAMPLE 13-5	3.358	10.564	0.022	2.868	0.418	2.000	7.774	0.272	-17.140	-267.589
2_14_16SAMPLE 14-1	201.623	360.011	1.951	317.966	0.418	2.000	385.346	0.883	-148.734	-354.881
2_14_16SAMPLE 14-2	140.269	316.636	1.951	277.039	0.418	2.000	297.771	0.875	-182.454	-508.363
2_14_16SAMPLE 14-3	149.993	300.756	1.951	258.500	0.418	2.000	300.214	0.860	-75.276	-166.747
2_14_16SAMPLE 14-4	132.223	229.257	1.940	197.426	0.418	2.000	251.494	0.861	-17.280	-18.439
2_14_16SAMPLE 14-5	58.486	159.997	1.940	142.367	0.418	2.000	134.566	0.890	-50.791	-115.753
2_14_16SAMPLE 14-6	77.025	192.388	1.951	168.322	0.418	2.000	170.389	0.875	-63.453	-138.494
2_14_16SAMPLE 15-1	3.428	13.292	0.022	1.749	0.418	2.000	8.003	0.132	-17.560	11.117
2_14_16SAMPLE 15-2	4.547	10.354	0.022	0.700	0.418	2.000	7.059	0.068	-16.860	6.790
2_14_16SAMPLE 15-3	4.337	10.774	0.029	0.350	0.418	2.000	7.744	0.032	-16.860	7.588
2_14_16SAMPLE 15-4	2.519	14.622	0.022	0.210	0.418	2.000	7.663	0.014	-16.231	10.484

Test ID	1.0 mm Force	Peak Force	dpkmx	F2mm	t-2mm	d-2mm	work of compression	Loss in pk force at 2-mm	negative peak force	negative area
	g	g	mm	g	sec	mm	g.mm	g	g	g.mm
	1.0 mm Force	Hardness	dpkmx	F2mm	t-2mm	d-2mm	Area F-D 1:2	ratio	negative peak force	Area F-D 3:4
2_14_16SAMPLE 15-5	3.848	10.354	0.022	0.280	0.418	2.000	8.009	0.027	-18.819	11.860
2_14_16SAMPLE 17-1	3.918	15.251	0.022	0.560	0.418	2.000	8.507	0.037	-16.440	18.399
2_14_16SAMPLE 17-2	5.807	13.572	0.056	0.770	0.418	2.000	7.524	0.057	-15.741	11.830
2_14_16SAMPLE 17-3	3.988	13.292	0.022	0.840	0.418	2.000	6.482	0.063	-16.371	6.216
2_14_16SAMPLE 17-4	3.428	10.284	0.022	1.259	0.418	2.000	7.191	0.122	-16.231	8.660
2_14_16SAMPLE 17-5	4.268	13.222	0.022	-0.210	0.418	2.000	7.321	-0.016	-16.860	7.205
2_14_16SAMPLE 19-1	76.466	114.454	1.492	87.799	0.418	2.000	140.607	0.767	-15.041	-0.789
2_14_16SAMPLE 19-2	437.946	1820.414	1.984	1796.908	0.418	2.000	1195.344	0.987	-13.432	-0.445
2_14_16SAMPLE 19-3	2361.061	3387.995	1.969	3273.612	0.418	2.000	3672.289	0.966	-12.103	-2.801
2_14_16SAMPLE 19-4	85.351	841.822	1.951	725.899	0.418	2.000	545.949	0.862	-12.103	-4.779
2_14_16SAMPLE 19-5	42.885	46.453	0.979	12.733	0.418	2.000	43.003	0.274	-10.144	-4.326
2_14_16SAMPLE 21-1	1712.257	2811.809	1.960	2592.696	0.418	2.000	3197.639	0.922	-393.102	-2411.658
2_14_16SAMPLE 21-2	970.127	1927.172	1.969	1712.677	0.418	2.000	1921.117	0.889	-302.644	-650.800
2_14_16SAMPLE 21-3	1067.721	2037.149	1.960	1840.213	0.418	2.000	2073.549	0.903	-302.225	-716.623
2_14_16SAMPLE 21-4	1240.800	2051.490	1.960	1844.550	0.418	2.000	2305.571	0.899	-163.355	-117.644
2_14_16SAMPLE 21-5	787.743	1683.993	1.960	1523.017	0.418	2.000	1588.727	0.904	-71.778	-23.942
2_14_16SAMPLE 21-6	492.444	1648.524	1.960	1472.576	0.418	2.000	1266.873	0.893	-196.796	-138.964
2_14_16SAMPLE 18-1	5.387	12.733	0.056	3.568	0.418	2.000	13.063	0.280	-17.700	-314.670
2_14_16SAMPLE 18-2	6.926	10.354	1.791	3.918	0.418	2.000	12.324	0.378	-20.078	-321.988
2_14_16SAMPLE 18-3	6.017	11.893	1.841	5.107	0.418	2.000	13.066	0.429	-20.358	-343.666
2_14_16SAMPLE 18-4	6.996	10.214	0.022	3.638	0.418	2.000	11.596	0.356	-21.757	-399.271
2_14_16SAMPLE 18-5	7.276	12.453	1.729	5.597	0.418	2.000	15.037	0.449	-25.605	-430.940
Average:	1061.903									
S.D.	2556.402									
Coef. of Variation	240.738									
End of Test Data										