THE PENNSYLVANIA STATE UNIVERSITY
SCHREYER HONORS COLLEGE

DEPARTMENT OF CHEMISTRY

THERMOMECHANICAL ANALYSIS OF PHASE TRANSITIONS IN A BINARY MIXTURE OF ETHYLENE GLYCOL AND WATER

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SPRING 2017

A thesis
submitted in partial fulfillment
of the requirements
for a baccalaureate degree
in Chemistry
with honors in Chemistry

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ABSTRACT

Thermomechanical analysis (TMA) was used to study rheological properties in order to better understand the low temperature, solid-liquid phase transition in the phase diagram of the ethylene glycol (EG) and water binary mixture system (EG-water) at mixture compositions of 20, 40, 60, 65, 70, 75, 80, 85, and 88.7 weight percent EG-water. This phase transition was explored using three repeated trials at each composition with the expansion probe and a 10°C/min ramp. In the composition range from 60 to 88.7% wt. EG-water, the experimentally determined solid-liquid transition falls around 50°C below the literature solidus.[1] Another phase diagram from the literature presents a glass transition in the composition range from 55 to 90 % wt. EG-water, and the experimentally determined solid-liquid transition falls about 30°C above the literature's glass phase transition.[4] The sample was also seen to be amorphous in X-ray studies at -150°C by Dr. Milosavljevic.

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ACKNOWLEDGEMENTS

I would like to thank my thesis advisor, Dr. Milosavljevic, as well as my honors advisor, Dr. Maslak. Thanks as well to Dr. Maroncelli. I would also like to thank the Penn State Chemistry Department and the Schreyer Honors College. Thanks also to my family and friends.

Chapter 1

Introduction

Ethylene glycol (EG) is a common and important compound. It has widespread applications, and its use in antifreeze is of particular interest.[1] The binary mixture of ethylene glycol and water (EG-water) is a system that needs to be better understood, and an accurate determination of the mixture's phase diagram would be very useful. The most recently published phase diagram by Cordray et al. addresses the disputed region of 55 to 90 weight percent ethylene glycol and water mixtures, but the results remain uncertain (Figure 1).[1] Disagreement persists over the number of eutectic points in the phase diagram as well as over the exact determination of the mixture's freezing points.[2]

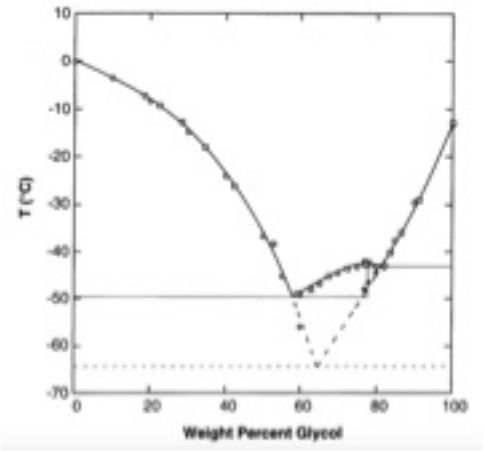

Figure 1. The phase diagram proposed by Cordray et. al.[1]

The solidus in Figure 1 was created through thermal analysis, observation of heating and cooling curves of samples of EG-water in double walled glass vessels. The freezing points were determined by an

observation of a warming up (often less than 1°C) in the temperature vs. time graphs with temperature data collected from platinum thermocouples (Figure 2).[1]

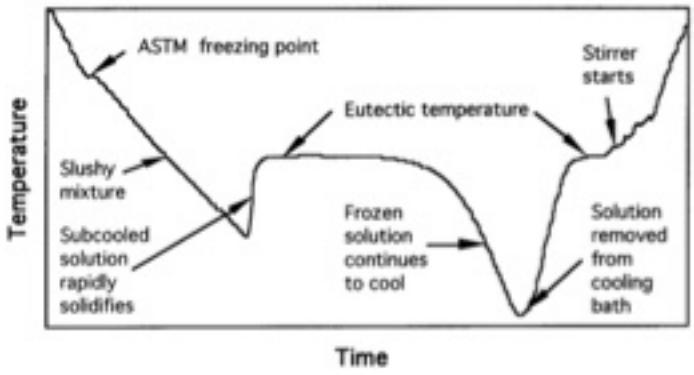

Figure 2. A typical Temperature vs. Time curve used to determine the phase diagram[1]

The flat solidus and phase diagram characteristics between 55 and 85 % wt. EG-water have been called into question by previous experiments, such as measuring viscosities where the phase diagram shows a solid phase (Figure 3).[3] These conflicting results call for further analysis of the EG-water binary mixture. The phase diagram also shows two eutectics at the intersection of the flat solidus and liquidus (Figure 1).[1] There is a metastable single eutectic/supercooling limit/crystallization at -63.5°C at the intersection of the dashed lines (Figure 1).[1] Viscosity data were obtained at -73°C, which falls below the supercooling limit in the phase diagram (Figures 1 & 3).[1,3]

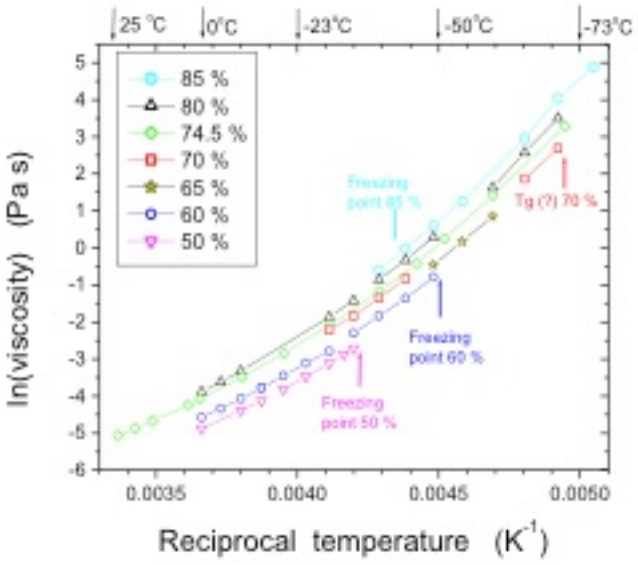

Figure 3. Work done by Ms. So Yeon Kim and Dr. Milosavljevic shows a measured viscosity at a temperature below the solidus proposed by the Cordray phase diagram.[3]

It should not be possible to obtain viscosity data at temperatures below the solidus, since solid materials do not have viscosities that can be measured as easily by these methods as liquids, so the freezing point of the mixture may be lower than previously thought. These viscosity measurements are also not likely explained by supercooling phenomena, since the viscosity measurements require agitation of the sample. Ethylene glycol and water must be mixed completely to ensure a homogenous mixture otherwise pockets of water in the mixture will preferentially freeze earlier. This is clear from the different melting points of each component represented at the zero percent and one hundred percent EG-water compositions. Ethylene glycol has a lower melting point than water such that incompletely mixed samples will always have pockets of water freezing first with interesting behavior only occurring with homogenous mixtures of the two components. The procedure followed by Cordray et. al. also seeded mixtures, which may have disrupted thermal equilibrium in their set up.[1]

An alternative phase diagram from B. Luyet and D. Rasmussen helps emphasize the non-consensus on the behavior the binary mixture of ethylene glycol and water (Figure 4).[4]

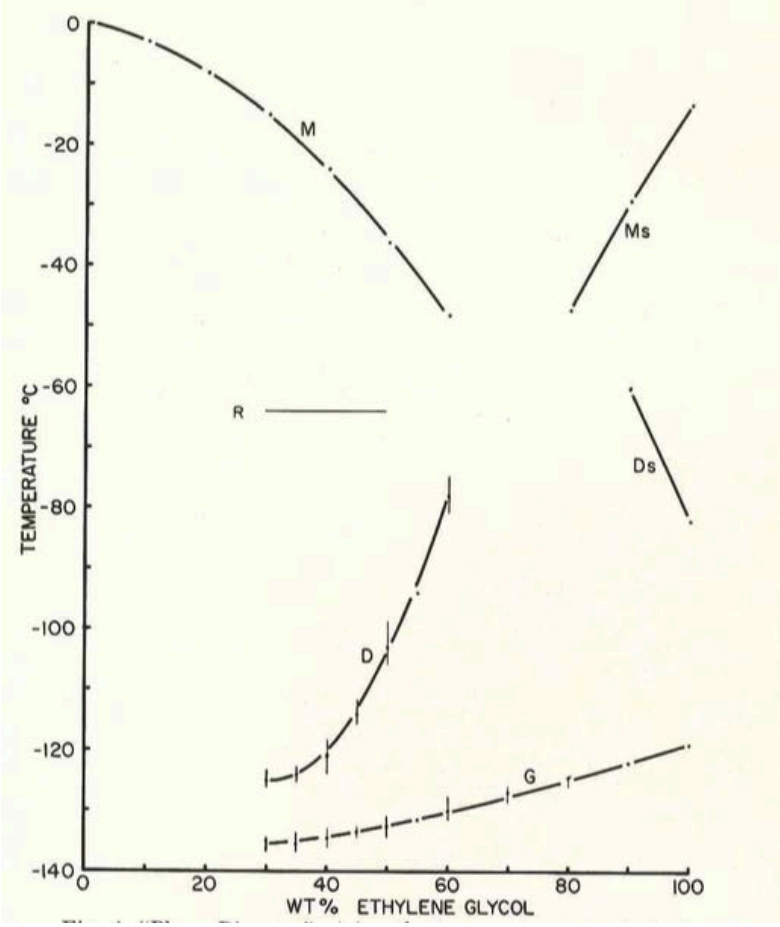

Figure 4. The phase diagram of the binary mixture of EG-water from Luyet and Rasmussen, where M labels melting, D labels devitrification, R labels recrystallization, G labels the glass transformation, and s indicates the solute.[4]

This phase diagram lacks the flat solidus present in Figure 1, and rather shows a glass transition at lower temperatures instead of freezing and melting in the 60% to 80% wt. EG-water region.[4] This phase diagram was constructed through differential thermal analysis.[4] This phase diagram is further supported by neutron powder diffraction studies that only observed a crystalline structure of a 1:1 molar mixture of EG-water (77.276% wt EG-water) at 3.5 K (-269.65 °C).[5] X-ray studies of 70% wt. EG-water performed by Dr. Milosavljevic also indicate an amorphous, glassy sample at -150 °C (Figure 5).

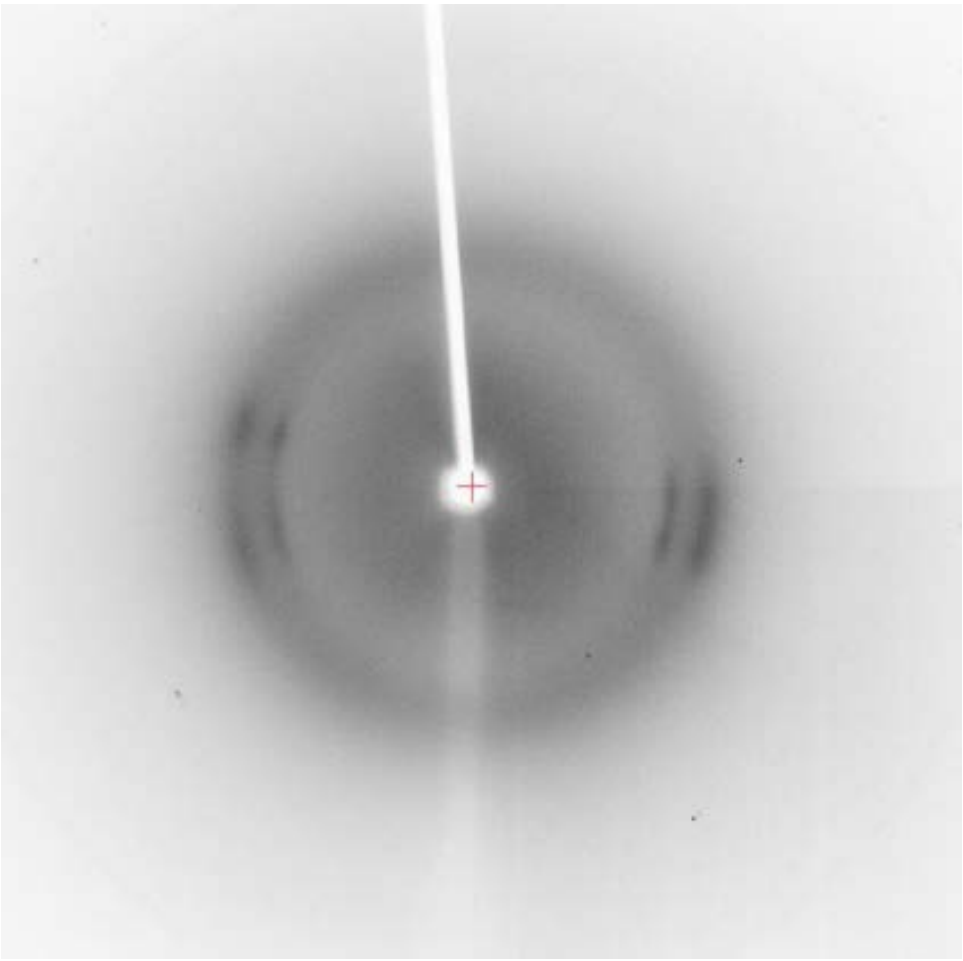

Figure 5. X-ray analysis of 70% wt. EG-water at -150 °C indicates an amorphous sample. Any crystallization in the sample would appear as dark spots like for pure water in Appendix A (Figure 24). Here no evidence of crystal structure is present. The loop containing the sample causes the visible circle in the image.

The behavior of hydrogen bond forming liquids at low temperatures near their glass transition temperature is of great interest. The viscosity characteristics at these low temperatures are key to better understanding the systems as well. Near the glass transition, viscosity can increase tenfold or greater while the temperature varies by only a few degrees.[6,7]

Chapter 2

Materials and Methods

In order to better understand the system, thermomechanical analysis (TMA) provides a useful methodology for finding rheological properties. The TMA instrument utilizes a quartz probe to measure the length of a sample as a function of temperature. Temperatures are monitored by a thermocouple with a purge gas flowing through the furnace covering the sample to keep thermal equilibrium within ± 1°C (Figure 6).[8] The quartz probe and stage are important to reduce error due to its resistance to changing shape and length during temperature changes.

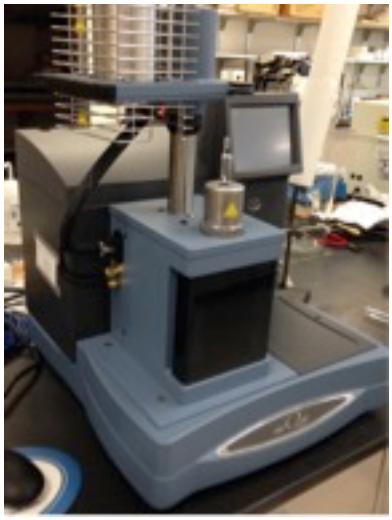

Figure 6. The TMA instrument with the expansion probe equipped and furnace turned away from the sample stage

The TMA instrument most often finds applications in measuring the linear coefficient of thermal expansion with expansion and penetration probe attachements. It also is used to measure materials under tension, compression, and 3 point bending through the use of other probes and set ups. However, it is a dynamic instrument with a great capability for other potential applications (Figure 7).[8]

	Q400EM	Q400
Temperature Range (max)	-150 to 1 000°C	-150 to 1 000°C
Temperature Precision	± 1°C	± 1°C
Furnace Cool Down Time (air cooling)	<10 min from 600°C to 50°C	<10 min from 600°C to 50°C
Maximum Sample Size - solid	26 mm (L) x 10 mm (D)	26 mm (L) x 10 mm (D)
Maximum Sample Size - film/fiber		
Static Operation	26 mm (L) x 1.0 mm (T) x 4.7 mm (W)	26 mm (L) x 1.0 mm (T) x 4.7 mm (W)
Dynamic Operation	26 mm (L) x .35 mm (T) x 4.7 mm (W)	—
Measurement Precision	± 0.1%	± 0.1%
Sensitivity	15 nm	15 nm
Displacement Resolution	<0.5 nm	<0.5 nm
Dynamic Baseline Drift	<1 µm (-100 to 500°C)	<1 µm (-100 to 500°C)
Force Range	0.001 to 2 N	0.001 to 2 N
Force Resolution	0.001 N	0.001 N
Frequency Range	0.01 to 2 Hz	—
Mass Flow Control	Included	Included
Atmosphere (static or controlled flow)	Inert, Oxidizing, or Reactive Gases	Inert, Oxidizing, or Reactive Gases
Operational Modes		
Standard	Included	Included
Stress/Strain	Included	—
Creep	Included	—
Stress Relaxation	Included	—
Dynamic TMA (DTMA)	Included	—
Modulated TMA™ (MTMA™)	Included	—

Figure 7. The capabilities and functional range of the TMA instrument[8]

For all its modes of operation, the TMA relies on removable probes inserted into the machine for analysis of the samples on the quartz sample stage. Inside the instrument, the probe is monitored by the moveable-core linear variable differential transducer (LVDT), which accurately reflects any dimension changes of the sample under the probe. The probe is also under the control of a non-contact, friction-free force motor capable of subjecting the sample to carefully controlled forces applied by the probe. Both the probe and the sample stage are under precise temperature control thanks to the instrument furnace and flowing purge gas.[8]

The TMA's capability lends itself to an application in finding the freezing point of the EG-water system. Though it is less commonly used for measurements like this, it is capable of mechanically

finding the freezing point and even glass transition temperatures (T_g) by identifying solid-liquid phase transitions.[8]

For the EG-water system, the liquid mixture is loaded into a DSC pan and placed onto the stage, then the furnace is cooled using liquid nitrogen and the sample freezes inside the pan. The expansion probe then measures and rests on top of the frozen sample (Figure 8).

All samples were prepared to their compositions gravimetrically (± 0.01%). The ethylene glycol was obtained from Sigma-Aldrich anhydrous, and at 99.8% purity. A new sample of the ethylene glycol was purchased to ensure no polymerization had occurred in storage.

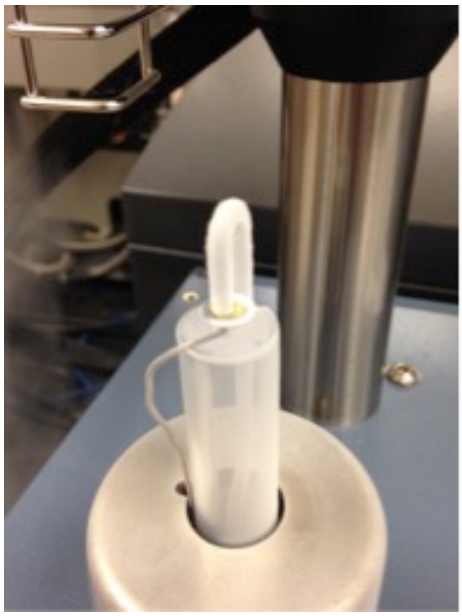

Figure 8. The EG-water mixture is inside the DSC pan on the sample stage with the thermocouple next to the pan. (The mixture is yellow in this picture due to the presence of a small amount of the $[Ru(bpy)_3]^{2=}$ luminescent probe for other applications).

The furnace then ramps the temperature up as the instrument records temperature and length data. The solid-liquid transition is determined from the onset point found at the intersection of tangent lines drawn by the instrument analysis software showing where the probe begins to sink into the melted sample (Figure 9).

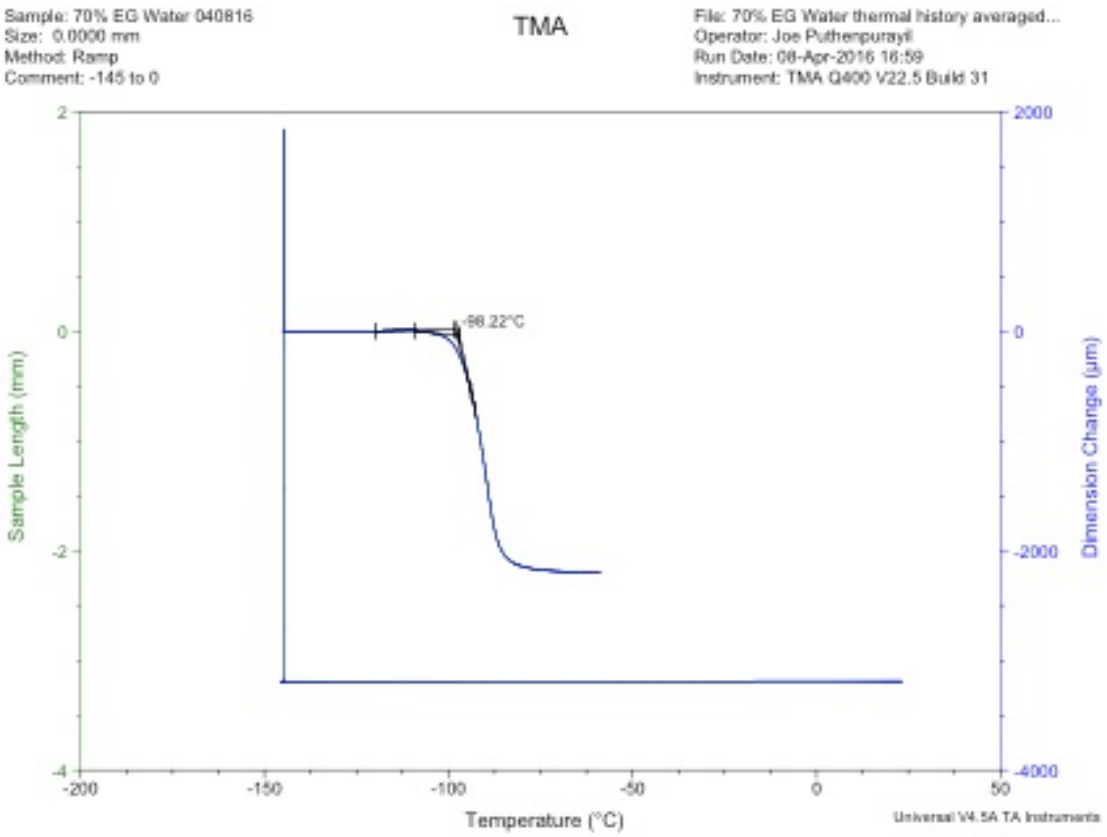

Figure 9. A typical Sample Length vs. Temperature TMA plot used to determine solid-liquid transitions

The TMA instrument also allows control over the temperature ramp rate, the force applied by the probe on the sample, and the purge gas flow rate. For all trials, helium purge gas at 200 ml/min was used as per instrument recommendations for low temperature experiments.[8]

Chapter 3

Results and Discussion

Phase Diagram

The experimentally determined solid-liquid transitions have been found using the averaged results of three trials per sample with 10°C/min ramps and a probe force of 0.05 N (Appendix A, Table 1). The samples observed include 20, 40, 60, 65, 70, 75, 80, 85, and 88.7 % wt. EG-water (± 0.01%). When these points are plotted along with the Cordray phase diagram, the temperature points from the samples with an accuracy of ± 1°C according to the instrument specifications in the region of dispute (55 – 90 % wt. EG-water) fall around 50°C below the diagram's suggested solidus (Figure 7). However, the 20% and 40% wt. EG-water sample experimental results agreed more closely with the liquidus in published results from Cordray et al (Figures 10 & 11).

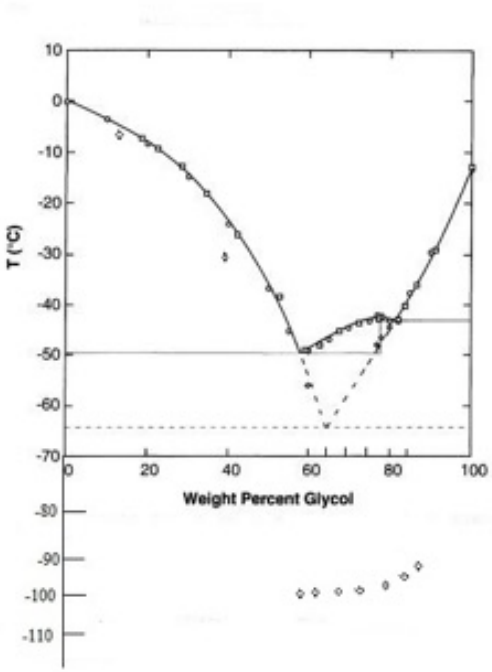

Figure 10. The solid-liquid transition points (◇) fall well below the Cordray diagram's straight line solidus in the 60 to 90 % wt. EG-water region of dispute as well as below the dotted line indicating the supercooling limit.

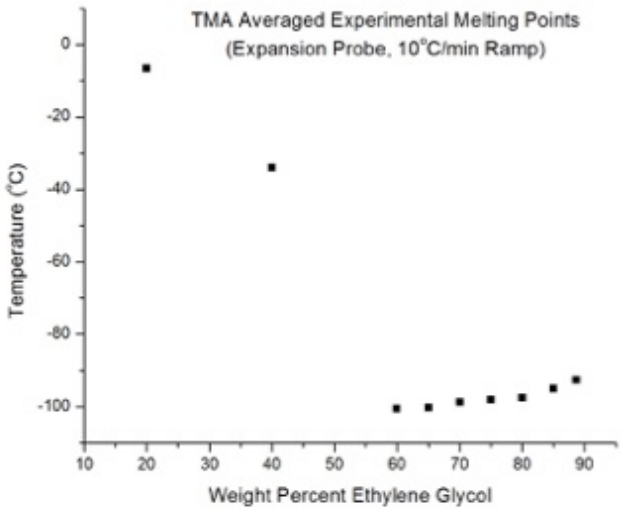

Figure 11. The solid-liquid transition points show a slight upward curve in the 55 to 90 % wt. EG-water region. Figure 25 in Appendix A also shows this trend in the data.

The temperature ramp is important in the determination of the location of the solid-liquid phase transition as a slower temperature ramp allows for more time for thermal equilibration between the sample and the thermocouple monitoring the temperature beside the sample. The difference between a 10°C/min and a 5°C/min ramp can be a few degrees. Analysis of the 60% wt. EG-water sample with a 10°C/min ramp gave a transition temperature of -100.99°C compared to −105.06°C given by analysis of the 60% wt. EG-water sample with a 5°C/min ramp (Figure 12).

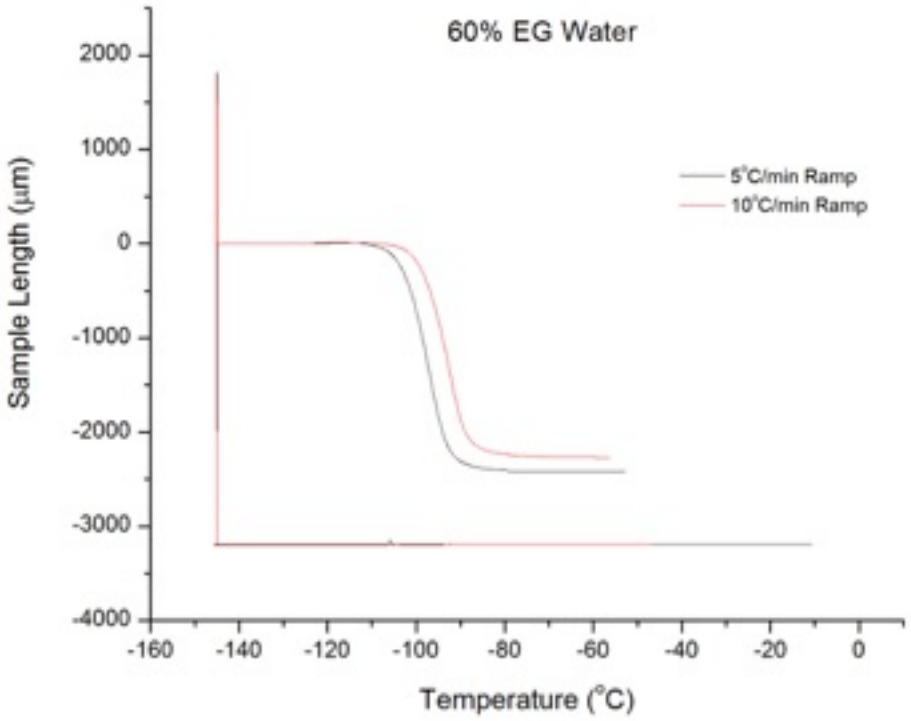

Figure 12. The different onset points due to different equilibration times are illustrated by plotting sample length against temperature.

Analysis of the 20% wt. EG-water sample with a 10°C/min ramp gave an averaged transition temperature of -6.54°C over three trials compared to −8.94°C given by analysis of the 20% wt. EG-water sample with a 5°C/min ramp. The 40% wt. EG-water sample had a transition temperature of −33.9°C with the three trial averaged 10°C/min ramp, while a 5°C/min ramp trial gave a transition

temperature of -36.29°C. However, the experimental transition temperatures remain far below the solidus in the Cordray diagram in the 55 to 90 % wt. EG-water region of dispute. The experimental data in this region falls closer to the glass transition suggested in the phase diagram proposed by Luyet and Rasmussen (Figure 4).[4]

Influence of the Surface

There is a small amount of the mixture in the DSC pan during the experiment, and it is possible to have a slight curvature at the sample surface due to the mixture's meniscus. The TMA instrument is sensitive to this shape, especially since the probe presses down directly on the frozen sample as it melts, and the shape of the surface is reflected in the beginning of the TMA data (Figure 13).

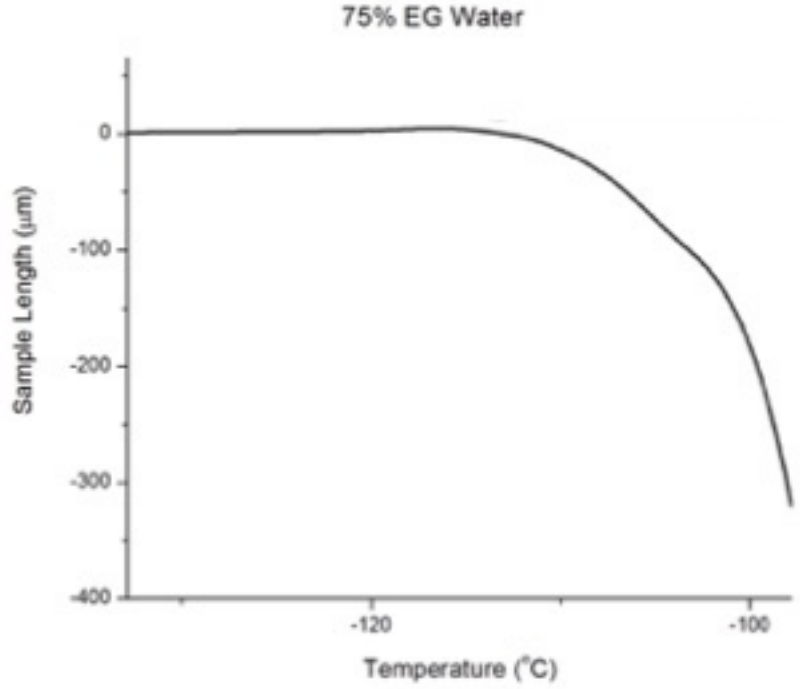

Figure 13. The slight upward trend in the data before penetration is due to the probe compressing the meniscus.

The asymmetric derivative of sample length with respect to time also makes the initial contact between the surface and probe clearer (Figure 14).

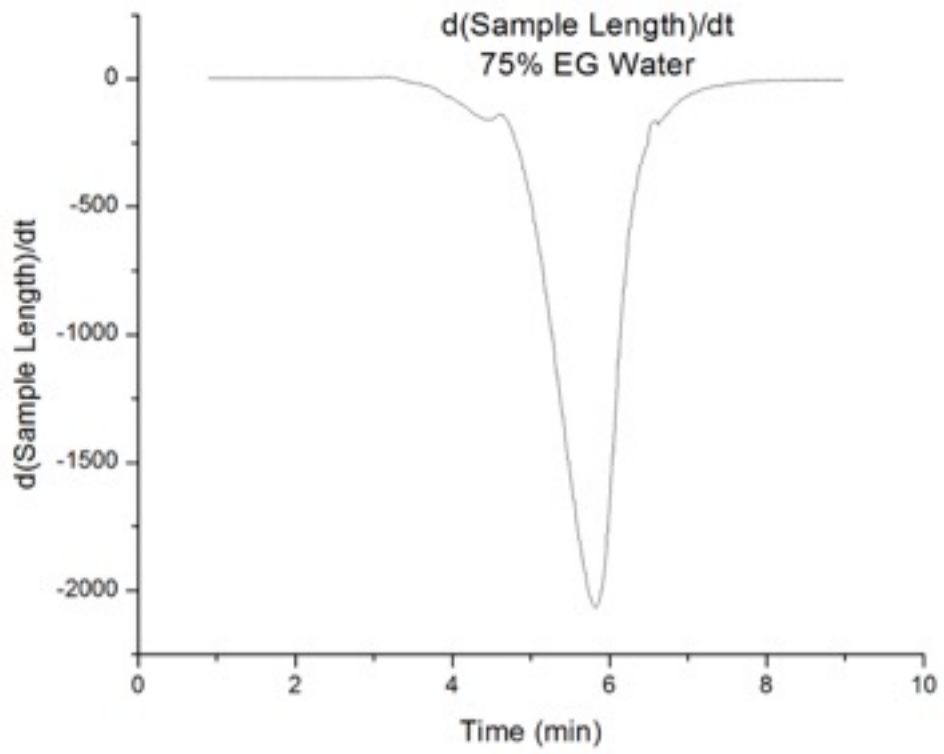

Figure 14. The time derivative of the 75% wt. EG-water sample is asymmetric, showing slightly different behaviors as the probe penetrates the solution then comes to rest at the bottom of the DSC pan. This asymmetry is best seen at around 5 minutes.

This detail due to the surface features is also readily apparent in the 70% wt. EG-water sample length derivatives with respect to both time and temperature (Figures 15 & 16).

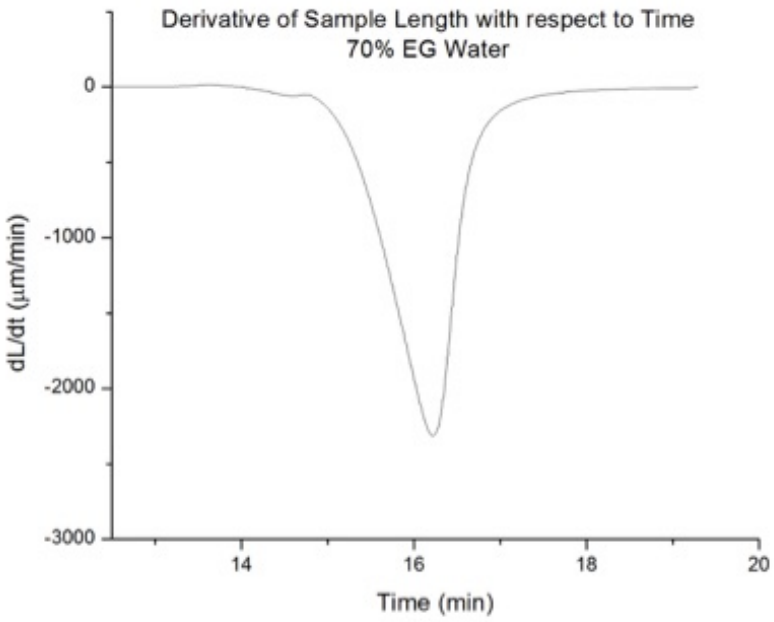

Figure 15. The derivative of sample length with respect to time is asymmetric.

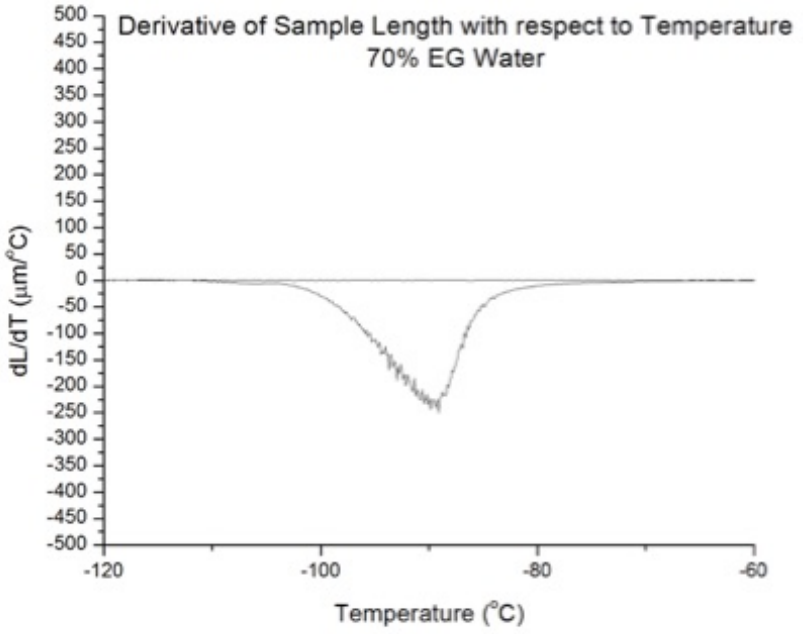

Figure 16. The derivative of sample length with respect to temperature is also asymmetric.

The thermal history of the sample is also important and shows information about the heating and cooling the sample undergoes within the TMA furnace (Figures 17 & 18). However, unlike the Cordray solidus, the experimental points are not constructed solely based on thermal analysis but rather by the thermomechanical analysis allowed by the physical interaction of the probe with the sample.

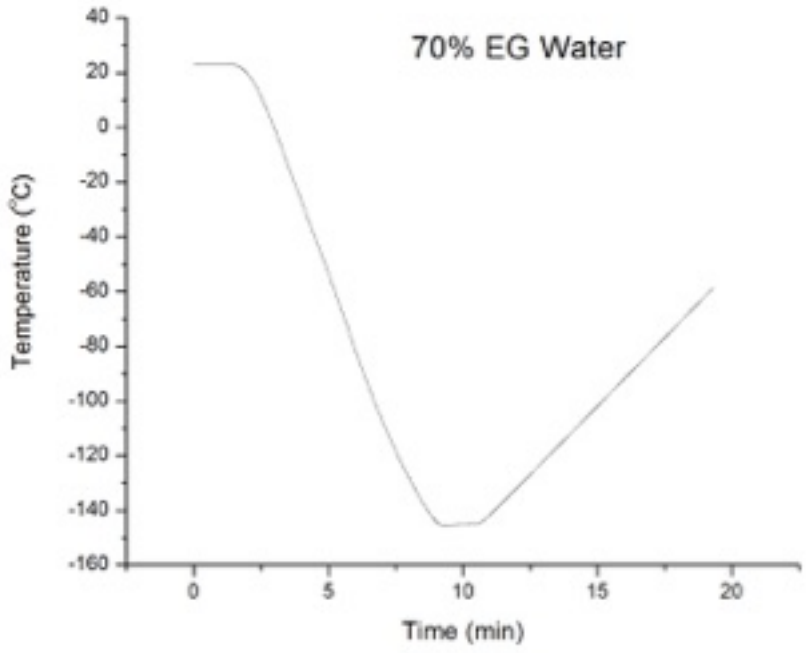

Figure 17. The thermal history of the sample shows how it was cooled then warmed in the TMA instrument.

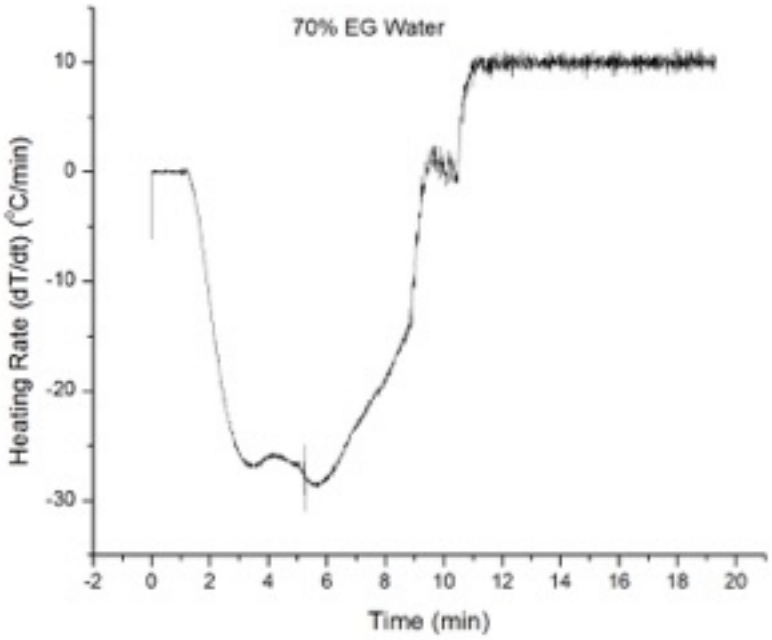

Figure 18. The derivative of the temperature vs time plot shows the heating and cooling rates of the sample in the TMA furnace.

Penetration Trials

The TMA instrument also shows promise for further rheological studies. When working with liquid conditions, the penetration rate of the probe into the solution is dependent upon the solution viscosity (Figures 19 & 20).

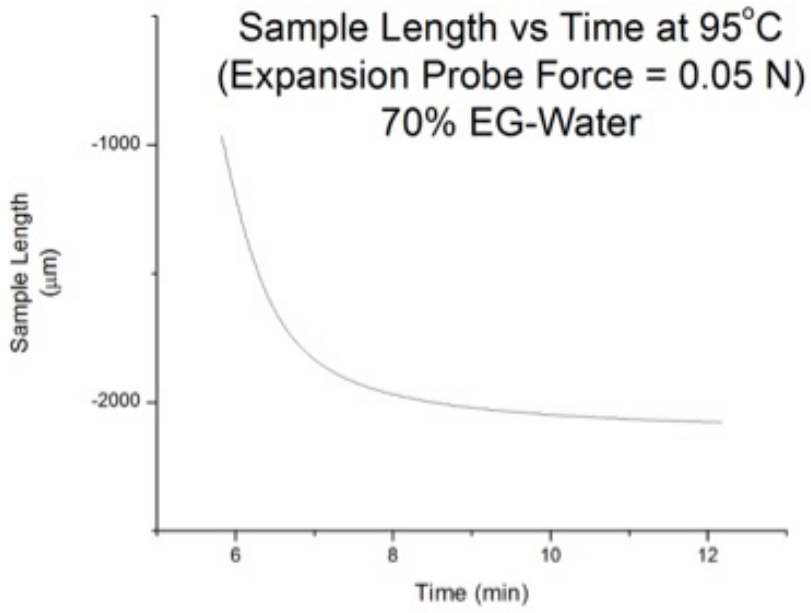

Figure 19. The 70% wt. EG-water sample penetration at 95°C

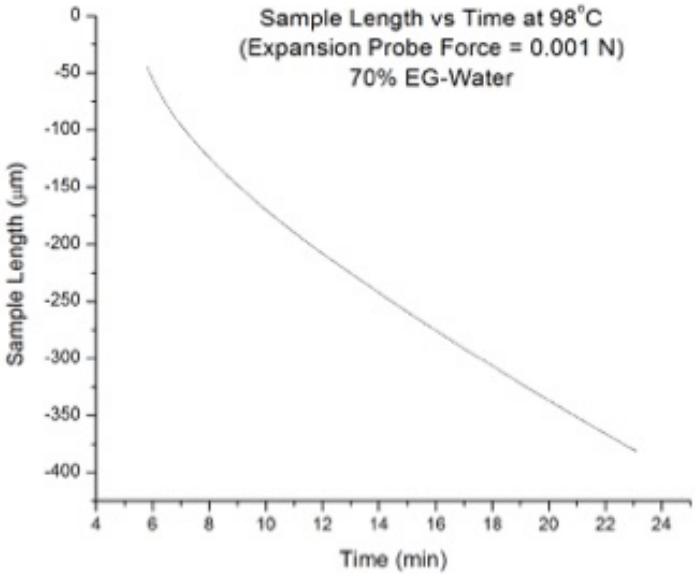

Figure 20. The 70% wt. EG-water sample penetration at 98°C

Penetrometry is a technique that uses an incompressible probe under a constant driving force, like the TMA's quartz probe, to penetrate into a viscous liquid, while its movement is monitored.[9] The geometry of the probe affects the calculations of viscosity. Hemispherical probes penetrate differently than flat-bottomed cylindrical probes (Equations 1 & 2).[9]

$$\eta = \frac{mt}{kr^{1/2}l^{3/2}}$$

Equation 1. This equation is used to calculate viscosity from data from a hemisphere tipped probe where m is the mass equivalence of the force on the probe, *t* is the penetration time, *k* is a constant that depends on the viscous liquid, *r* is the probe radius, and *l* is the penetration depth.[9]

$$\eta = k\frac{F}{dv}$$

Equation 2. This equation is used to calculate viscosity from data from a cylindrical probe with a flat tip penetrating into solution. *k* is a proportionality constant related to the viscous liquid, *F* is the driving force pushing the probe into the liquid, *d* is the probe diameter, and *v* is the penetration rate of the probe into the solution, which can be monitored experimentally.[10]

The analysis of the liquid system requires adjusting the force of the probe as well as the isotherm temperature in order to view the probe's progress through the solution. A lesser probe force leads to slower penetration.[3] The different mixture compositions also have different melting points. However, the TMA does collect useful data while probing the system this way. The derivative of the sample length with respect to time gives the penetration rate, with a flat line corresponding to a constant penetration rate (Figure 21).

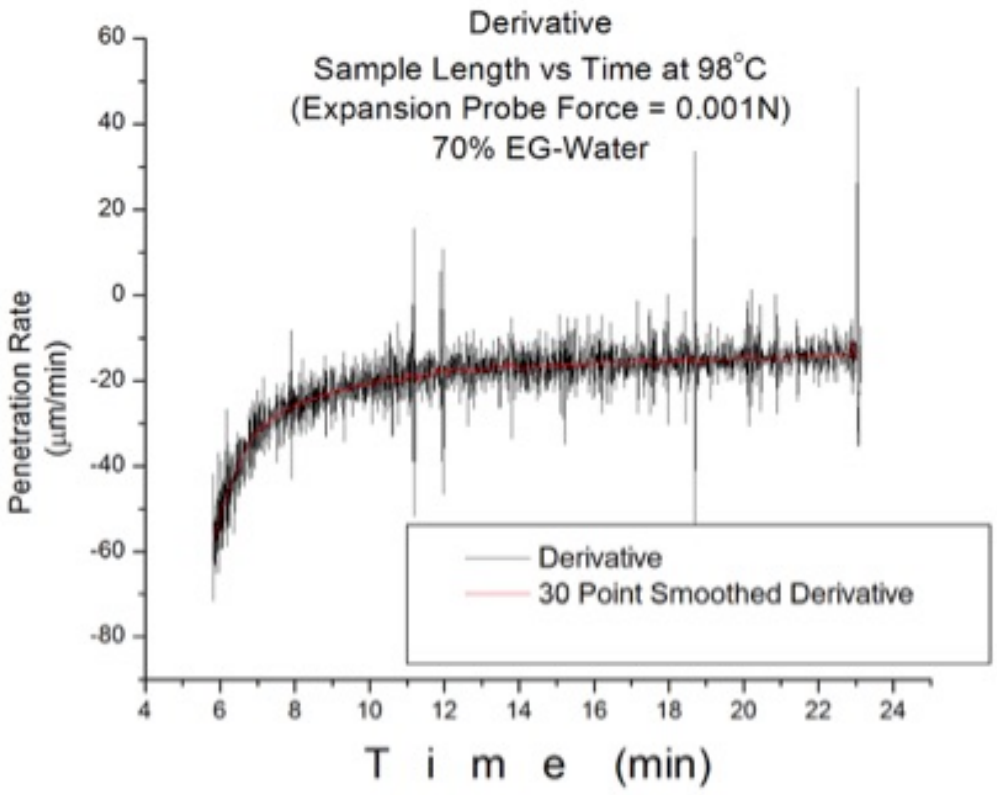

Figure 21. The derivative plot of Figure 20 shows a fairly constant penetration rate at the end of the trial.

The TMA instrument has some advantages over typical methods when it comes to measuring viscosities, especially at low temperatures. It is more suited to measuring highly viscous samples at very low temperatures, and can be applied to find viscosities at a sample's glass transition temperature (T_g).[9,10]

Some effort has been made to improving the accuracy and repeatability of TMA trials used to determine low temperature viscosity information. Trials with viscosity standards as well as a modification to lengthen the probe tip of the penetration probe to ensure constant and full penetration through the sample have helped improve data collection (Figure 22).

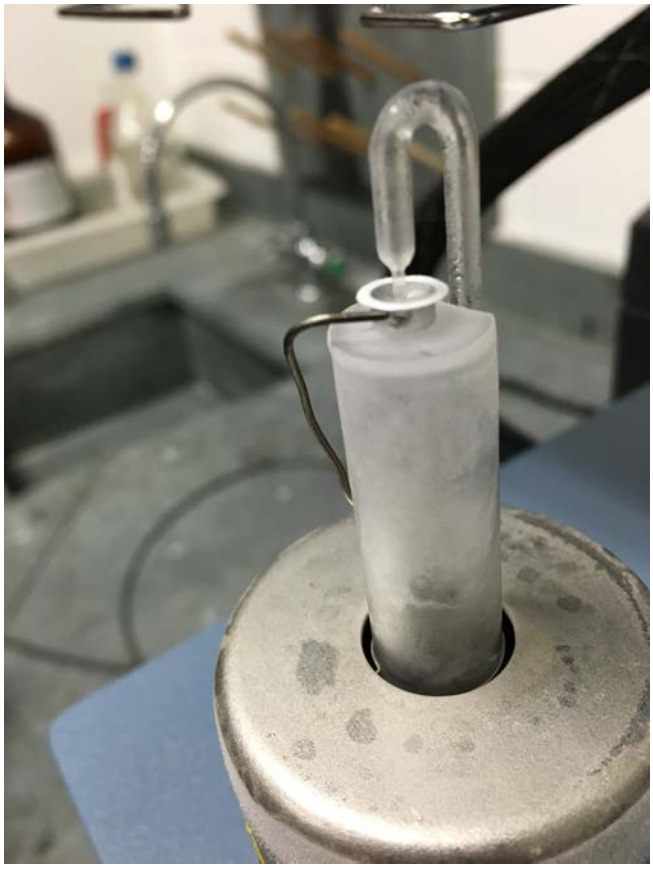

Figure 22. Lengthening the tip of a penetration probe ensures its consistent and full penetration through the sample so the viscosity is more accurately reflected in the penetration rate.

A linear penetration rate is the most useful for viscosity determination. Applying the second equation to the data from the -98°C isotherm of the 70% EG-water binary mixture with a 0.001 N driving force gives a penetration rate, v, of 17.04 μm/min.[10] This gives a viscosity of about 3.122 x 10^6 Pa•s with a k of 2.5.[10]

Chapter 4

Conclusion

The experimental TMA trials show a much lower solid-liquid transition than the most recently published phase diagram, around 50°C below.[1] It falls more in line with a glass transition, though around 30°C above, as proposed by another published phase diagram (Figure 23).[4] TMA methodology is much less disputable than thermal analysis for solid-liquid phase transitions and the region of dispute for binary mixtures of ethylene glycol and water (55 to 90% wt. EG water) deserves further analysis to better understand the properties of the binary mixture. TMA analysis and penetrometry methods also prove useful in collecting high viscosity data at very low temperatures through analysis of penetration rate.

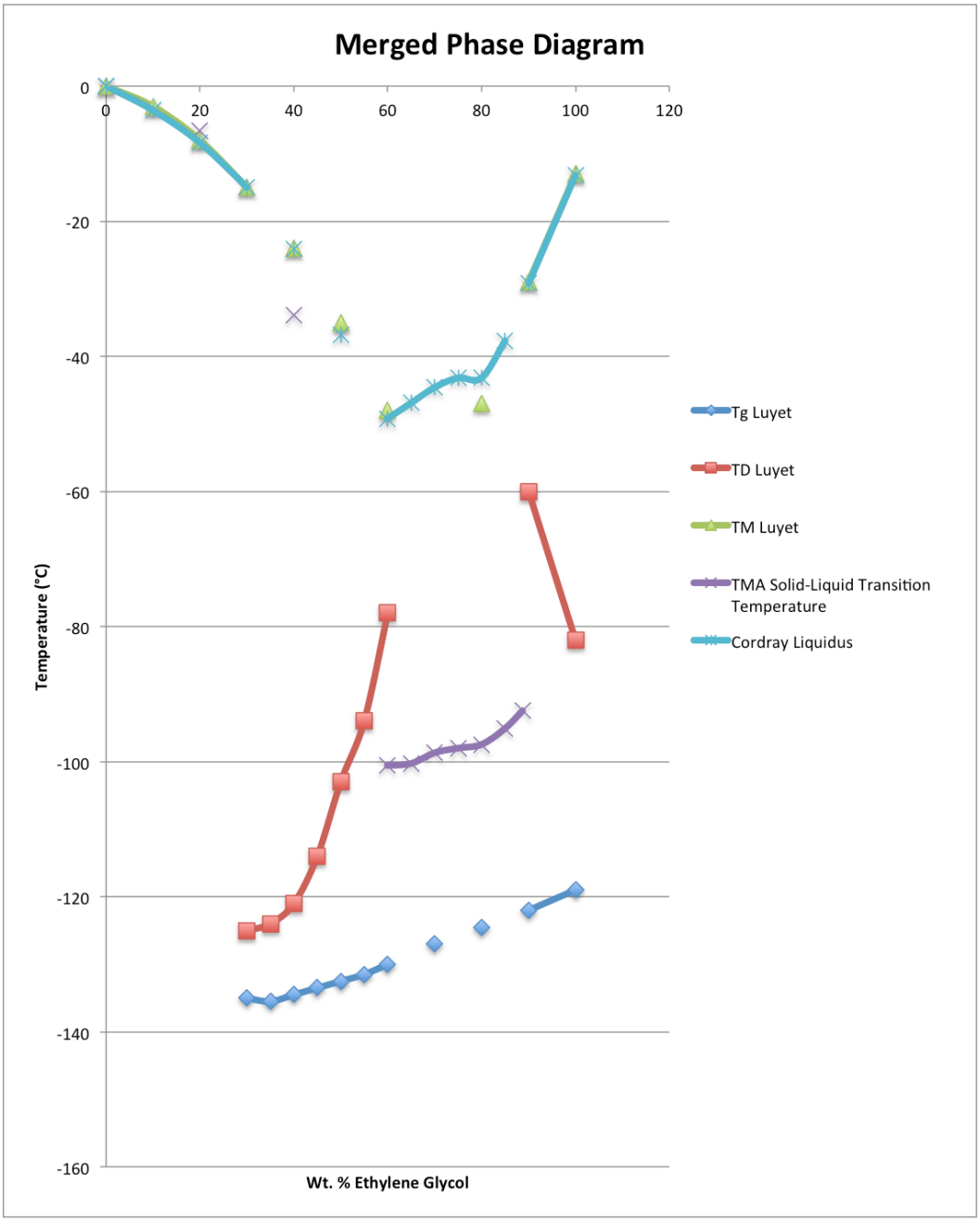

Figure 23. The phase diagram for the binary mixture of ethylene glycol and water displaying all literature and experimental data with Tg being the glass transition, T_D for devitrification, and T_M for melting

Appendix A

Supplementary Data

Sample (%wt EG water)	Trial 1 (Solid-Liquid Transition Temperature °C)	Trial 2 (°C)	Trial 3 (°C)	Average (°C)
20	-8.53	-5.44	-5.65	-6.54
40	-45.60	-29.23	-26.91	-33.91
60	-100.99	-100.40	-100.28	-100.56
65	-100.05	-101.85	-98.90	-100.27
70	-98.22	-98.86	-98.99	-98.69
75	-98.21	-97.31	-98.48	-98
80	-98.90	-97.19	-96.26	-97.45
85	-94.75	-95.35	-95.13	-95.08
88.7	-92.59	-92.29	-92.50	-92.46

Table 1. Summary of TMA solid-liquid transitions

Wt% Ethylene Glycol	Tg (°C) Luyet	T_D (°C) Luyet	T_M (°C) Luyet	TMA Solid-Liquid Transition Temperature (°C)	Cordray Liquidus (°C)
0			0	0	0
10			-3		-3.6
20			-8	-6.54	-8.4
30	-135	-125	-15		-15
35	-135.5	-124			
40	-134.5	-121	-24	-33.91	-24.1
45	-133.5	-114			
50	-132.5	-103	-35		-36.8
55	-131.5	-94			

60	-130	-78	-48	-100.56	-49.2
65				-100.27	-46.9
70	-127			-98.69	-44.6
75				-98	-43.2
80	-124.5		-47	-97.45	-43.2
85				-95.08	-37.7
88.7				-92.46	
90	-122	-60	-29		-29.2
100	-119	-82	-13		-13.2

Table 2. Summarized literature and experimental transitions[1,4]

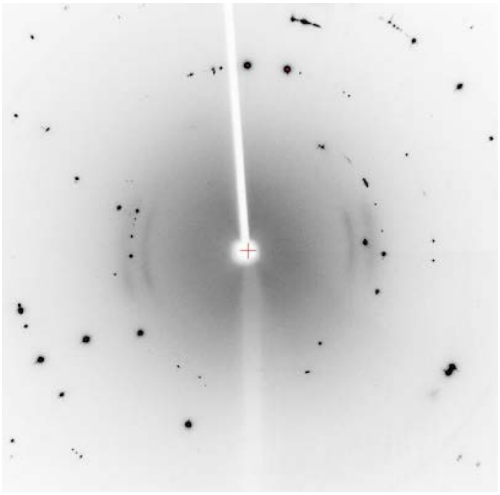

Figure 24. X-ray studies of frozen pure water show evidence of crystal structure via the dark spots in the image.

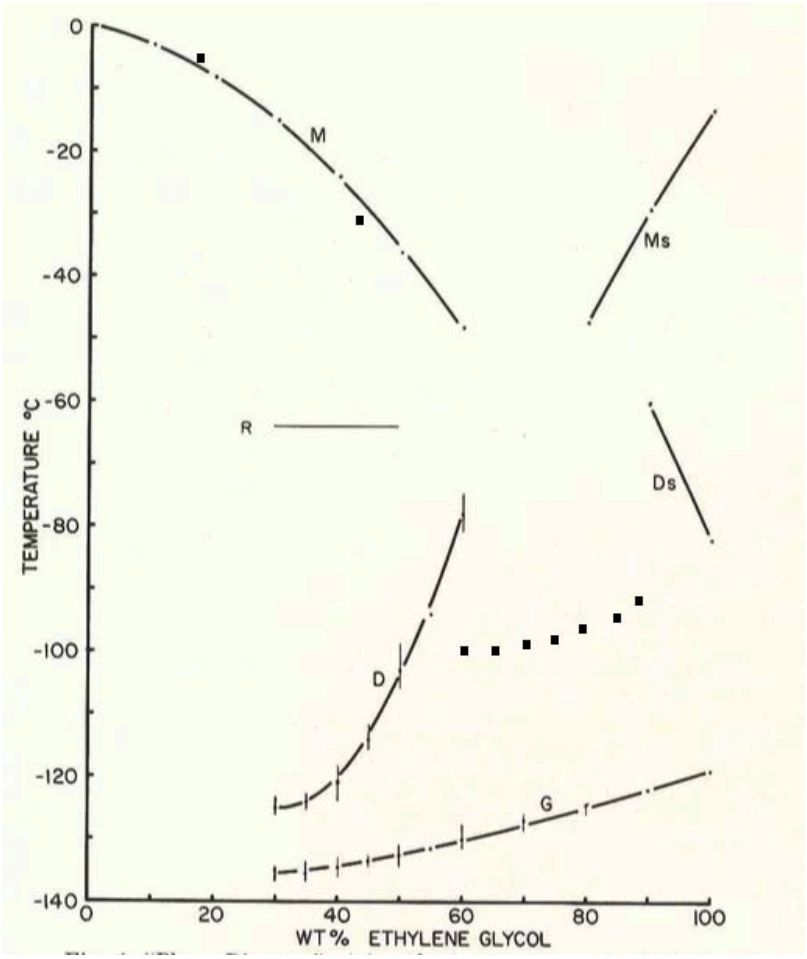

Figure 25. The solid-liquid transition points (■) fall above the glass transition in the 60 to 90 % wt. EG-water region, while following a similar upward trend. M labels melting, D labels devitrification, R labels recrystallization, and G labels the glass transformation.[4]

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