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**SRNL-STI-2012-00070**

**Dynamic Mechanical Analysis Characterization  
of Glovebox Gloves**

**P.S. Korinko and Y. Breakiron**

**February 29, 2012**

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## Contents

List of Tables .....	4
List of Figures .....	4
Summary .....	5
Background .....	5
Experiment.....	6
Results and Discussion .....	7
Summary and Conclusions.....	12
Acknowledgements.....	13
References .....	13
Appendix A:.....	14
Appendix B:.....	16

## List of Tables

Table 1. Description of gloves and ID used for the testing. ....	7
Table 2. Average measured T <sub>g</sub> , Storage Modulus, and Tan $\delta$ for all the samples. ....	10

## List of Figures

Figure 1. Schematic of output for a DMA showing phase shift $\delta$ . ....	6
Figure 2. Schematic of DMA output indicating several physical changes in a polymer 6.....	6
Figure 3. DMA results for a Guardian 15 mil Butyl rubber test at three loading frequencies (a) linear and (b) log plots for the Moduli. ....	8
Figure 4. Determination of T <sub>g</sub> (-60.07) for the North Butyl at 1 Hz. ....	9
Figure 5. Graphical representation of the glass transition temperature gloves with single T <sub>g</sub> . ....	10
Figure 6. North 30 mil Butyl exhibiting an increase in the loss modulus between 50 and 75°C. ....	11
Figure 7. DMA results for Jung Butyl-Hypalon <sup>®</sup> with both Butyl and Hypalon <sup>®</sup> overlays. ....	12
Figure 8. DMA results for Jung Butyl-Viton <sup>®</sup> with both Butyl and Viton <sup>®</sup> overlays. ....	12



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# Dynamic Mechanical Analysis Characterization of Glovebox Gloves

### Summary

As part of the characterization of various glovebox glove material from four vendors, the permeability of gas through each type as a function of temperature was determined and a discontinuity in the permeability with temperature was revealed. A series of tests to determine the viscoelastic properties of the glove materials as a function of temperature using Dynamic Mechanical Analysis (DMA) was initiated. The glass transition temperature and the elastic and viscoelastic properties as a function of temperature up to maximum use temperature were determined for each glove material. The glass transition temperatures of the gloves were -60°C for butyl, -30°C for polyurethane, -16°C Hypalon®, -16°C for Viton®, and -24°C for polyurethane-Hypalon®. The glass transition was too complex for the butyl-Hypalon® and butyl-Viton® composite gloves to be characterized by a single glass transition temperature. All of the glass transition temperatures exceed the vendor projected use temperatures.

### Background

Butyl rubber gloves are the gloves currently used for containment in gloveboxes at Savannah River Site (SRS) in the Tritium Facility. As part of an overall characterization task (ref 1-4), the permeation properties and rates at room temperature were determined in air and hydrogen (3). In an effort to expand the data to temperatures other than room temperature, a North Butyl 30 mil glove was tested at room, 50, and 75°C. Based on thermally activated processes, it is expected that the permeation at elevated temperatures would scale linearly as logarithm of permeability versus reciprocal temperature, which is termed Arrhenius behavior. However, there appeared to be a non-linear discontinuity in the data plot of log permeability versus inverse temperature. Consequently, additional characterization was initiated using Dynamic Mechanical Analysis (DMA). Dynamic Mechanical Analysis is a technique where a small deformation is applied to a sample in a cyclic manner. This force application allows the material's elastic and viscoelastic response to stress, temperature, frequency and other values to be studied.

DMA measures stiffness and damping, which are reported as storage modulus (stiffness) and loss modulus and tan delta (damping). Because a sinusoidal force is applied, it is possible to express the modulus as an in-phase component, the storage modulus, and an out of phase component, the loss modulus, see Figure 1. The storage modulus, either  $E'$  or  $G'$ , is the measure of the sample's elastic behavior. The ratio of the loss to the storage is the tan delta ( $\tan \delta$ ) and is often called damping. It is a measure of the energy dissipation of a material.

Modulus values change with temperature and transitions in materials can be seen as changes in the  $E'$  or tan delta curves. This includes not only the glass transition and the melt, but also other transitions that occur in the glassy or rubbery plateau, shown in Figure 2. These transitions indicate more subtle

changes in the material. The samples were prepared and tested so that the properties from below the glass transition to the maximum use temperature were examined.

## Experiment

Gloves from four manufacturers, North, Piercan, Jungitec, and Guardian, with compositions based on butyl, Hypalon®, Viton®, and polyurethane comprised the test matrix, shown in Table 1. Several thicknesses as well as composition variations were tested.

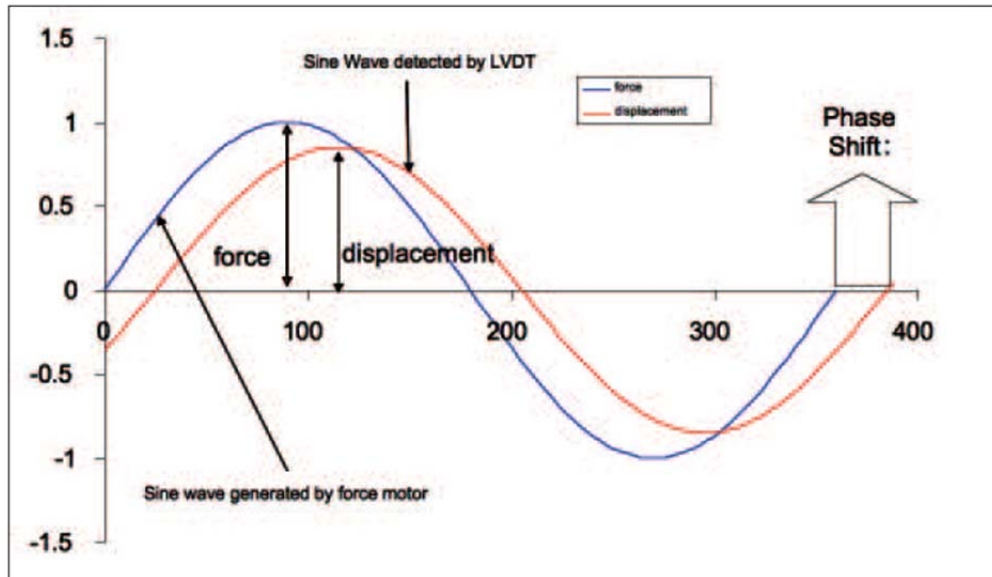


Figure 1. Schematic of output for a DMA showing phase shift 5.

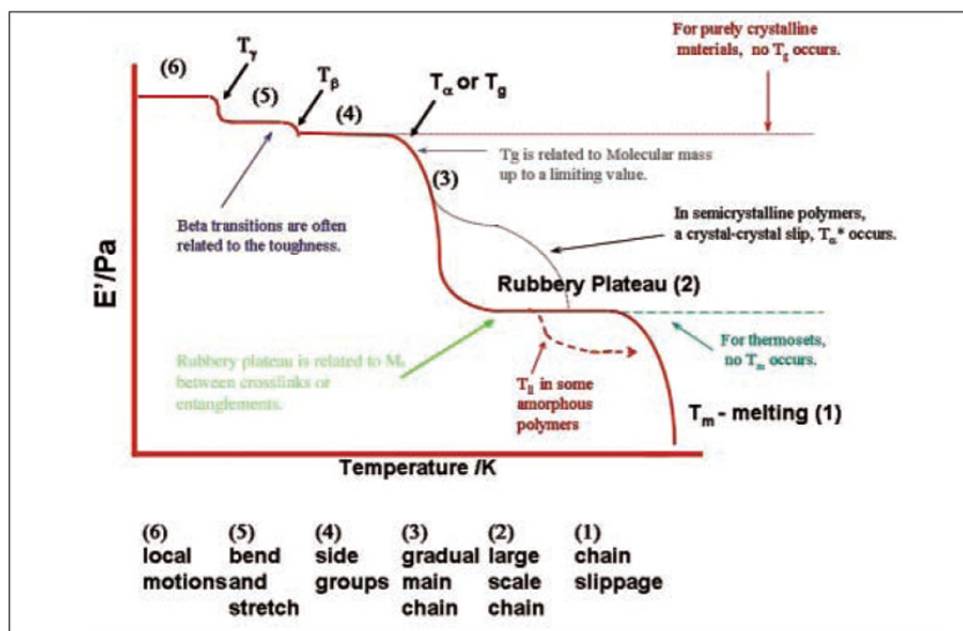


Figure 2. Schematic of DMA output indicating several physical changes in a polymer 5.

A TA Instruments 2950 Dynamic Mechanical Analyzer was used for this study. It is a forced oscillation, non-resonant, constant amplitude instrument. A tensile clamp, or sample holder was used due to the thickness of the sample and broad temperature range of interest.

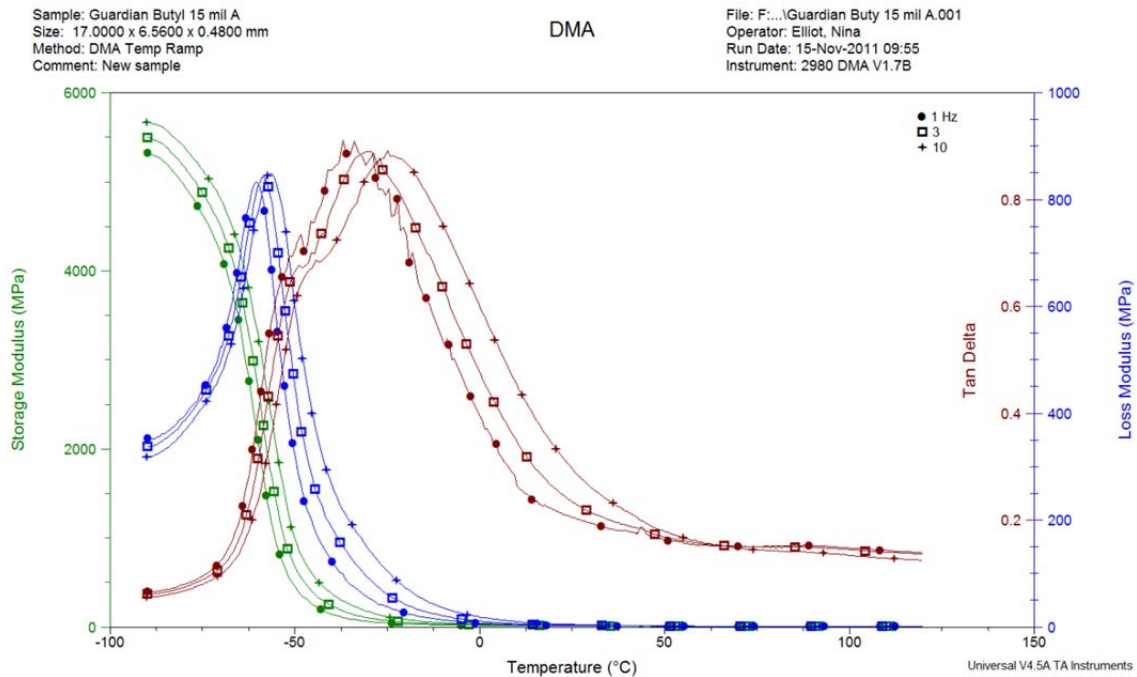
Rectangular test samples were cut from each glove. The samples were 6.4 mm (0.25") wide and 44.4 mm (1.5") long. Each sample being tested was installed on the tensile clamp. The test temperature was stabilized at the sub-ambient starting temperature (-90°C for most of the tests—but this was adjusted based on the observed  $T_g$ ), holding for ten minutes, and then increasing the temperature at 1°C/min to 120°C or the vendor recommended maximum temperature. The dynamic mechanical properties were continuously measured during the controlled heating. The TA 2980 has both electrical resistance heating and liquid nitrogen evaporative cooling using the Gas Cooling Accessory (GCA) and is controlled by the DMA software. The samples were loaded at frequencies of 1, 3, and 10 Hz. Up to three replicate tests were run, depending on the fidelity of the data. The DMA amplitude was set to be 40 microns, and the force track was 125%. The instrument was calibrated both at the beginning and the end of testing.

**Table 1. Description of gloves and ID used for the testing.**

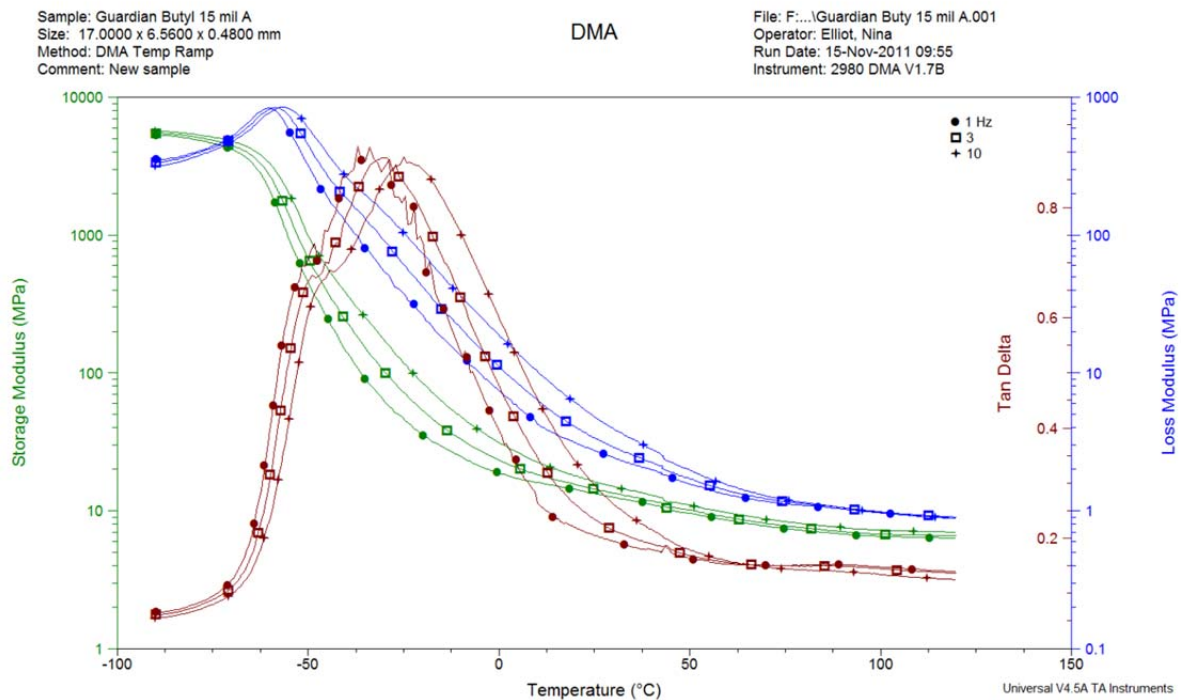
Vendor	Composition	Thickness (mils)	ID	Vendor	Composition	Thickness (mils)	ID
North	Butyl	15	NB15	North	Butyl	30	NB30
Piercan	Butyl	15	PB15	Piercan	Butyl	30	PB30
Piercan	Electrostatic Discharge Butyl	15	PESDB15	Piercan	Electrostatic Discharge Butyl	24	PESDB24
Guardian	Butyl	15	GB15	Guardian	Butyl	30	GB30
Jung	Butyl-Hypalon <sup>®</sup>	27	JBH27	Jung	Butyl-Viton <sup>®</sup>	20	JBV20
Jung	Viton <sup>®</sup>	24	JV24	Jung	Viton <sup>®</sup>	31	JV31
Piercan	Polyurethane	15	PU15	Piercan	Polyurethane-Hypalon <sup>®</sup>	20	PUY20
Piercan	Hypalon <sup>®</sup>	25	PY25				

## Results and Discussion

The DMA results for Guardian Butyl at 15 mil thickness tested at 1, 3, and 10 Hz are shown in Figure 3. These results indicate consistent mechanical and physical property response over this frequency range. The increase in all three measured properties, storage modulus, loss modulus, and tan delta, with increasing frequency is expected and can even be used to account for time – temperature trade-offs, although that was not completed as part of this study. The glass transition temperature,  $T_g$ , was obtained from the 1 Hz data for all the samples, a practice which is consistent with the approach taken in the literature.



(a) Linear



(b) Log

Figure 3. DMA results for a Guardian 15 mil Butyl rubber test at three loading frequencies (a) linear and (b) log plots for the Moduli.

The  $T_g$  was determined using the TA instruments data analysis software (6). A plot of the resultant analysis is shown in Figure 4. The tangent and slopes are used in the analysis program to determine the

$T_g$ . The average  $T_g$  values for the butyl rubber, Viton<sup>®</sup>, polyurethane, Hypalon<sup>®</sup>, and polyurethane-Hypalon<sup>®</sup> samples are listed in Table 2, and the individual values are available in Appendix A. The data for the approximate  $T_g$  for butyl rubber exhibit very little scatter and have an average of -60.5°C and a standard deviation of 0.71°C. The other gloves have significantly higher  $T_g$ s at -30°C for polyurethane and approximately -16°C for Hypalon<sup>®</sup> and Viton<sup>®</sup>. The composite glove of polyurethane-Hypalon<sup>®</sup> has a  $T_g$  that is intermediate between polyurethane and Hypalon<sup>®</sup>, as shown in Figure 5; the other composite gloves (BV and BH) exhibited unexpected behavior and will be discussed subsequently. The individual plots for the 1 Hz data for all of the samples are presented in Appendix B.

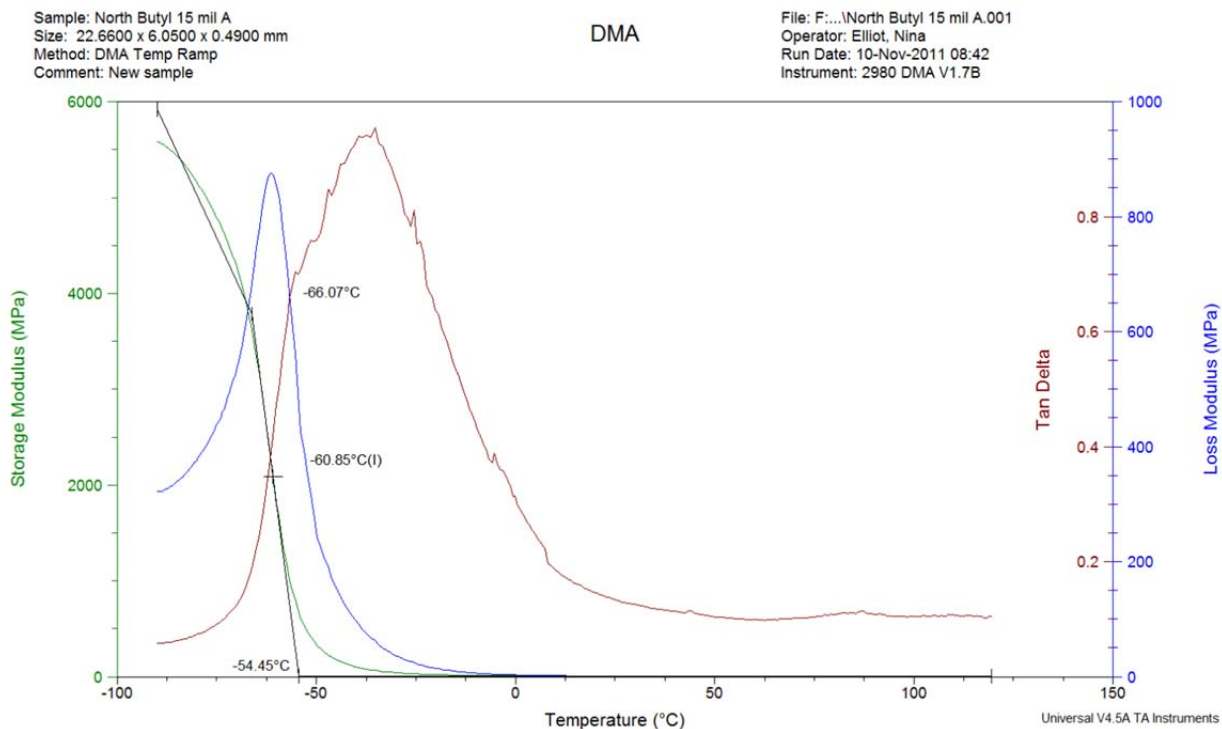
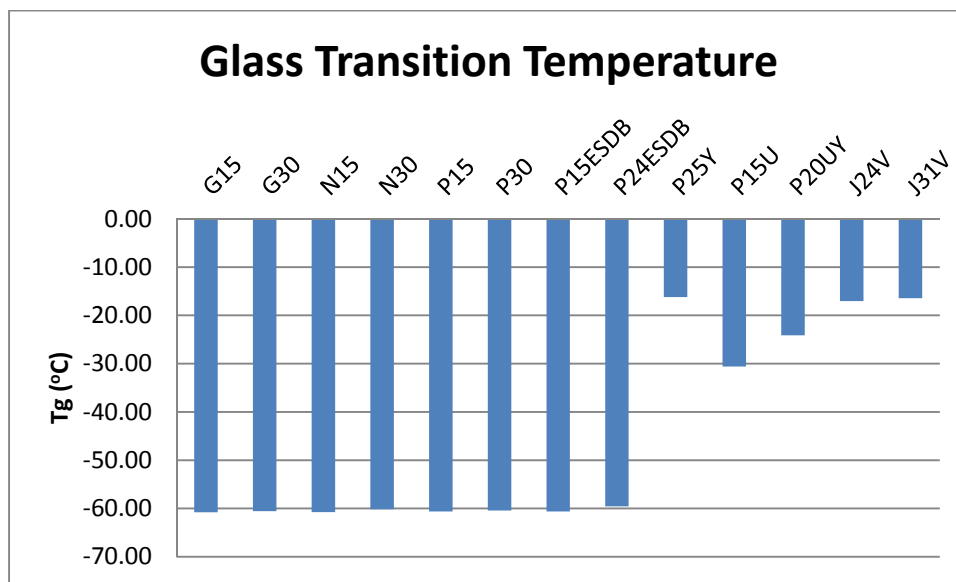


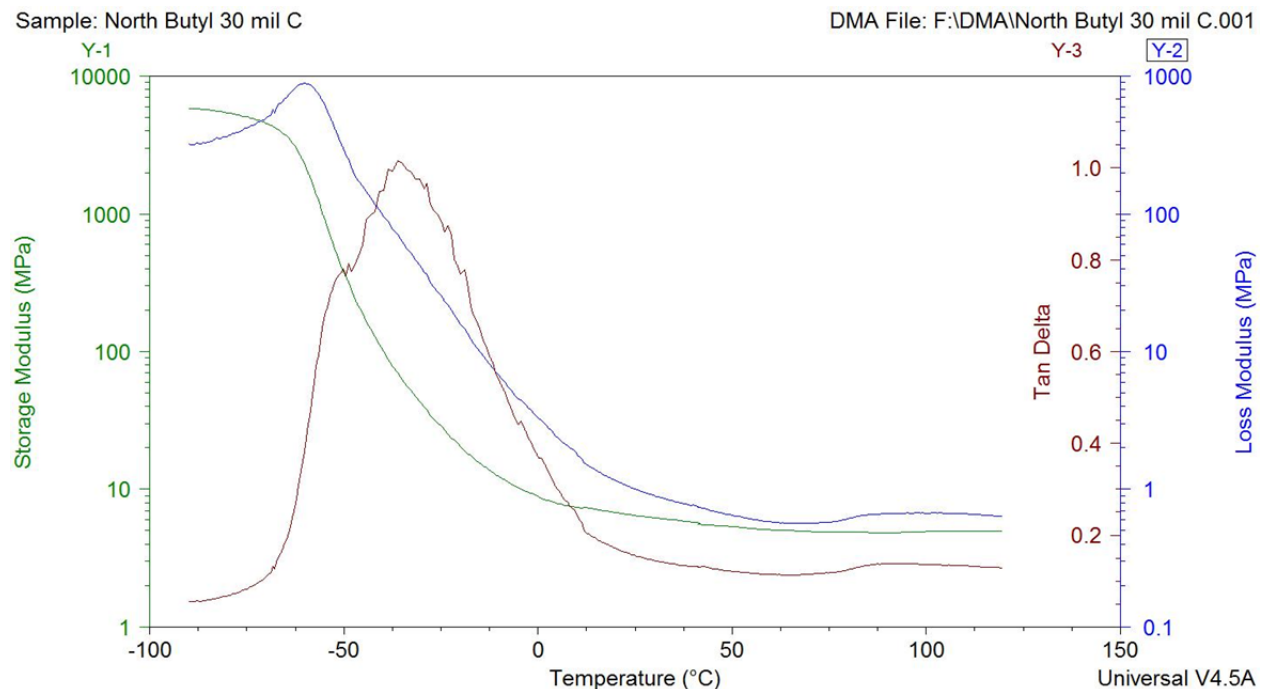
Figure 4. Determination of  $T_g$  (-60.07) for the North Butyl at 1 Hz.

Examination of the DMA data plots over the entire temperature range tested may indicate physical and structural property changes. For instance, there is an unexpected change in the  $\tan \delta$  of the North butyl rubber between 50 and 75°C, Figure 4. The storage modulus decreases with increasing temperature across the entire test range; however, the loss modulus and consequently, the  $\tan \delta$  values exhibit an increase, as shown in Figure 6. This deviation from the expected continuous reduction in loss modulus with increasing temperature coincides with an observed discontinuity of the permeability of butyl rubber (3) over the range of 25 to 75°C. Thus, the non-Arrhenius relationship of permeation rate with temperature appears to be related to a real structural change in the polymer. The change in physical or structural properties measured across the potential use temperature suggests that additional testing may be warranted to better characterize the temperature effects on permeability and structure/properties.

Table 2. Average measured  $T_g$ , Storage Modulus, and  $\tan \delta$  for all the samples.

	~Approx $T_g$ ( $^{\circ}\text{C}$ )	Actual $T_g$ ( $^{\circ}\text{C}$ )	Storage Modulus at $T_g$ (Mpa)	$\tan \delta$ at $T_g$
G15	-60.8	-68.6	4201	0.137
G30	-60.6	-68.7	4397	0.133
N15	-60.7	-68.4	3795	0.140
N30	-60.2	-68.1	4306	0.130
P15	-60.6	-68.4	2794	0.137
P30	-60.4	-68.7	4288	0.132
P15ESDB	-60.6	-68.4	3739	0.136
P24ESDB	-59.6	-69.4	3566	0.136
P25Y	-16.2	-25.1	1489	0.067
P15U	-30.6	-39.2	1852	0.068
P20UY	-24.1	-35.3	1935	0.055
J24V	-17.0	-24.5	2738	0.089
J31V	-16.5	-24.4	2749	0.100

Figure 5. Graphical representation of the glass transition temperature gloves with single  $T_g$ .



Jung Butyl-Hypalon® (JBH) and Jung Butyl-Viton® (JBV) gloves were also evaluated using DMA. These materials exhibited more complex behaviors and have more than one glass transition temperature. The DMA results for Butyl-Hypalon® are shown in Figure 7, and those of Butyl-Viton® in Figure 8. The plots are far more complex than the previous test results. The JBH27.5 exhibits multiple transitions at about -60, -20, and 2°C. The first two  $T_g$  values are consistent with the base polymers of this composite and the third is not. Additional testing to determine the cause would be required. The JBV20 sample exhibits dual  $T_g$ s, one at -60 and one at -9°C. The -60°C is consistent with butyl while the -9°C is close to that observed for Viton®, i.e., -16°C. No intermediate  $T_g$  was detected suggesting that there is no interaction between the butyl and Viton®, while there may be an interaction between butyl and Hypalon®.



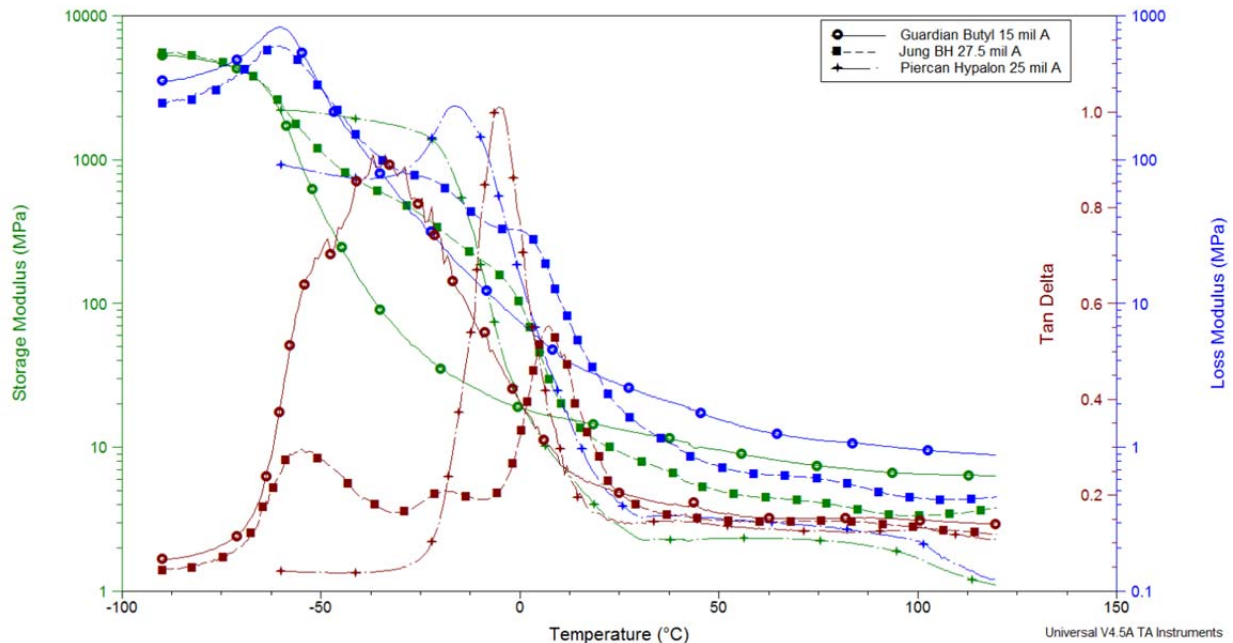


Figure 7. DMA results for Jung Butyl-Hypalon<sup>®</sup> with both Butyl and Hypalon<sup>®</sup> overlays.

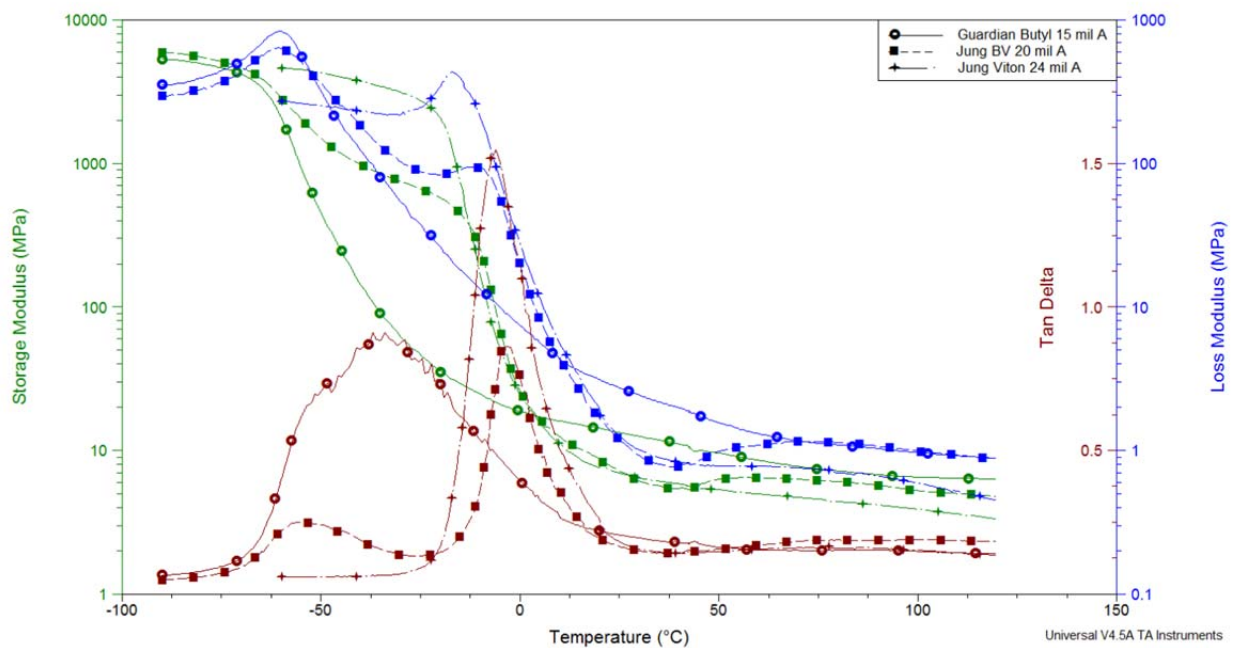


Figure 8. DMA results for Jung Butyl-Viton<sup>®</sup> with both Butyl and Viton<sup>®</sup> overlays.

## Summary and Conclusions

The glass transition temperature,  $T_g$ , of the materials used in gloves of interest for SRS Tritium Facility glovebox use was determined. The  $T_g$  of all the butyl gloves was consistent and about  $-60^{\circ}\text{C}$ . The composite gloves exhibited three different behaviors: the Polyurethane-Hypalon<sup>®</sup> had a  $T_g$  that was intermediate between Polyurethane and Hypalon<sup>®</sup>; the butyl-Viton<sup>®</sup> glove exhibited two distinct  $T_g$ s, at



approximately the same values as the component constituents; while the butyl-Hypalon<sup>®</sup> composite material exhibited three distinct  $T_g$ s; one that was at approximately each of the constituent materials and a final one that was about 2°C.

All of the  $T_g$  are well below room temperature and so brittle behavior is not expected for any of the glove materials in Tritium Facility operating conditions. Even the highest measured  $T_g$  of 2°C for the Jung butyl-Hypalon<sup>®</sup> is well below room temperature.

The North butyl exhibits an increase in the loss modulus at temperatures between 50 and 75°C. The reason for this increase was not evaluated; however, the temperature range that this deviation was observed in is consistent with the observed change in the plot of log permeation rate as a function of reciprocal temperature.

## Acknowledgements

The authors would like to thank Tritium Operations, Tritium Engineering, and Tritium Extraction Facility for technical and financial support.

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## Appendix A:

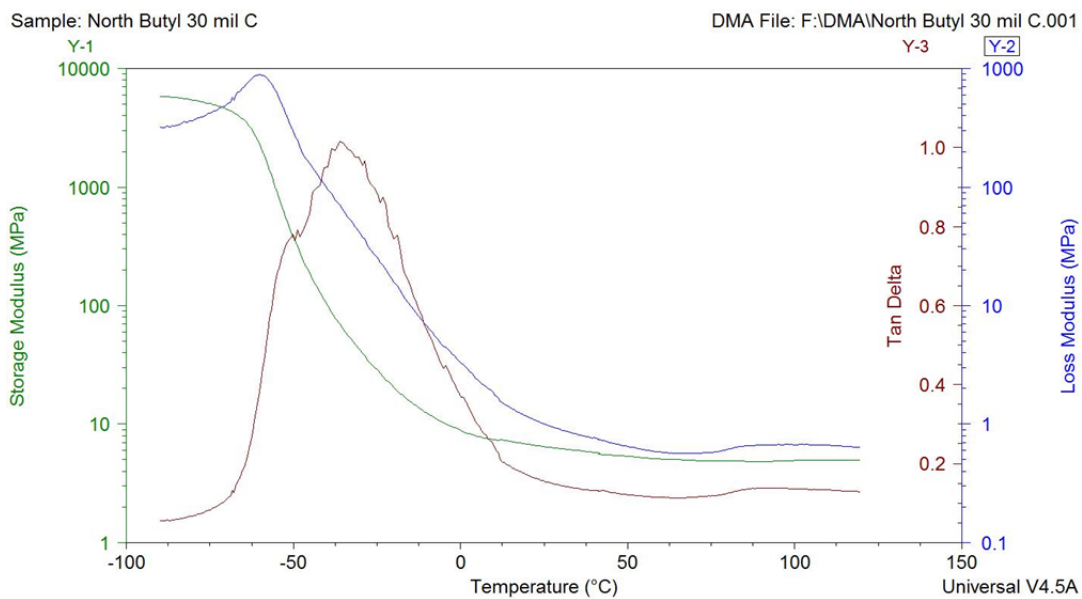
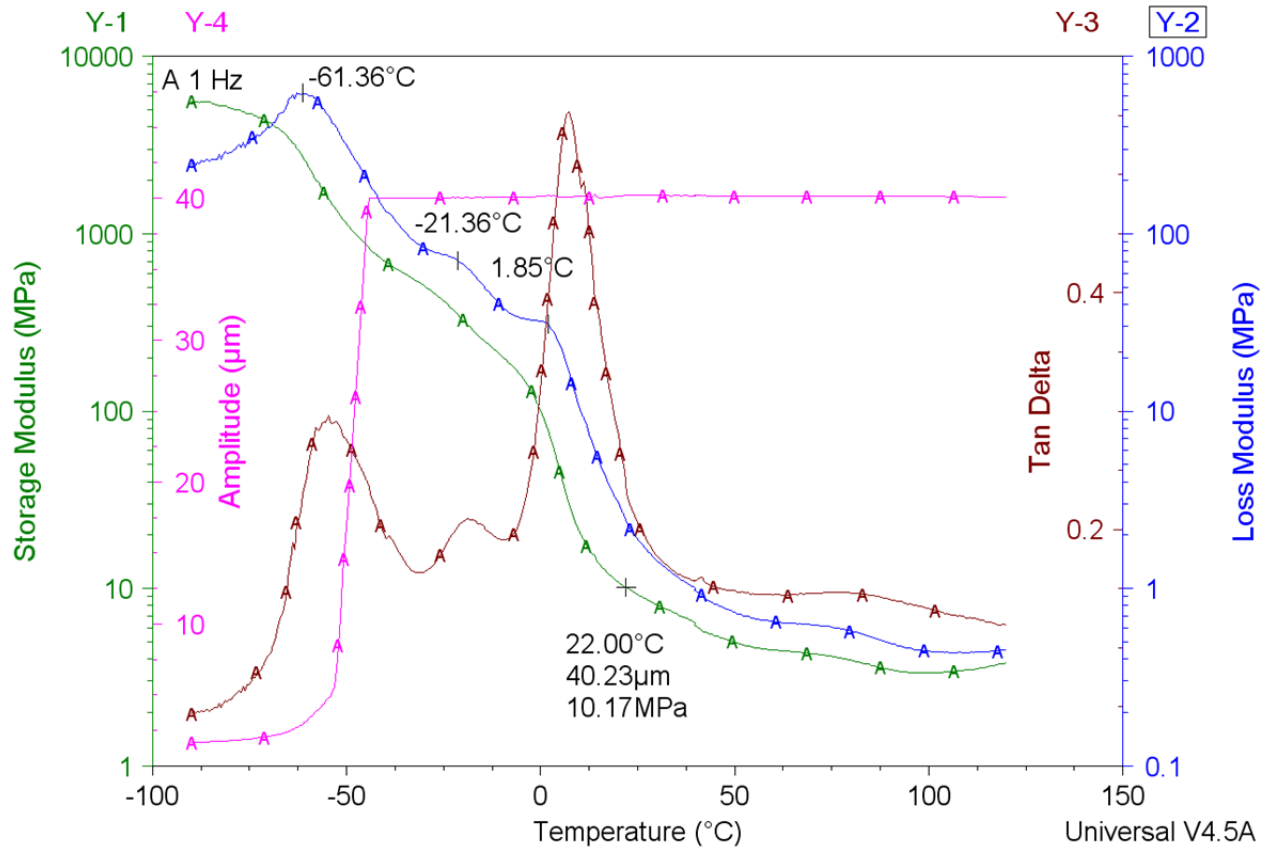
	~Approx imated T <sub>g</sub> (°C)	Actual T <sub>g</sub> (°C)	Storage Modulus at T <sub>g</sub> (MPa)	Tan δ at T <sub>g</sub>	Storage Modulus at 22 °C (MPa)	Storage Modulus at 50 °C (MPa)	Storage Modulus at 75 °C (MPa)	Storage Modulus at 120 °C (MPa)	Tan δ at 22°	Tan δ at 50°	Tan δ at 90°	Tan δ at 120°
G15BA	-60.5	-68.4	3993	0.141	13.87	9.59	6.77	6.33	0.213	0.162	0.152	0.140
G15BB	-61.1	-68.8	4409	0.133	15.15	10.67	7.59	7.11	0.210	0.162	0.153	0.141
G30BA	-60.9	-68.6	4527	0.137	15.16	10.65	7.62	6.93	0.191	0.154	0.144	0.131
G30BB	-60.3	-68.8	4266	0.130	14.93	10.78	8.04	7.65	0.216	0.177	0.179	0.169
N15BA	-61.3	-68.8	4095	0.140	8.70	6.26	1.73	1.64	0.141	0.104	0.109	0.105
N15BB	-60.2	-68.1	3495	0.141	9.54	8.34	6.50	5.29	0.170	0.127	0.150	0.143
N30BA	NA	NA	NA	NA	NA	6.55	5.78	5.45	NA	0.120	0.168	0.156
N30BB	NA	NA	NA	NA	6.88	6.01	5.10	4.60	0.207	0.157	0.190	0.167
N30BC	-60.2	-68.1	4306	0.130	6.63	5.35	4.82	4.97	0.164	0.121	0.138	0.129
P15BA	-60.0	-68.2	2779	0.137	1.91	1.16	0.48	NA	0.149	0.098	0.089	NA
P15BB	-60.4	-68.3	2695	0.137	0.90	0.52	0.50	NA	0.141	0.094	0.082	NA
P15BC	-61.5	-68.7	2907	0.136	1.24	1.07	0.70	0.73	0.157	0.121	0.116	0.063
P30BA	-60.0	-68.6	4395	0.136	13.31	9.64	7.10	6.71	0.200	0.157	0.141	0.128
P30BB	-60.8	-68.8	4180	0.129	11.32	8.15	6.03	5.50	0.213	0.165	0.147	0.130
P15ESDBA	-60.6	-68.3	3913	0.141	7.33	5.04	2.77	2.71	0.189	0.151	0.154	0.138
P15ESDBB	-60.6	-68.6	3565	0.131	5.44	2.87	1.02	1.02	0.185	0.141	0.142	0.181
P24ESDBA	-60.7	-68.6	3765	0.135	7.53	4.66	2.47	0.68	0.170	0.138	0.147	0.134
P24ESDBB	-58.4	-70.2	3366	0.136	7.24	4.97	2.61	2.26	0.185	0.147	0.151	0.136
P25YA	-15.9	-24.9	1527	0.070	3.28	2.33	2.01	1.10	0.148	0.138	0.124	0.108
P25YB	-16.5	-25.4	1450	0.064	4.93	1.61	2.07	1.68	0.158	0.137	0.113	0.093
P15UA	-30.3	-39.4	1876	0.066	12.53	6.60	0.73	NA	0.134	0.091	0.152	NA
P15UB	-30.9	-38.9	1827	0.071	10.20	3.03	1.06	NA	0.146	0.099	0.154	NA
P20UYA	-26.0	-35.8	1922	0.054	14.09	5.19	1.09	NA	0.143	0.120	0.181	NA
P20UYB	-22.3	-34.8	1948	0.055	11.45	6.69	0.95	NA	0.143	0.124	0.184	NA

J24VA	-17.1	-24.3	2679	0.094	7.43	5.33	4.17	3.34	0.207	0.147	0.159	0.135
J24VB	-17.0	-24.8	2797	0.084	5.61	3.45	1.47	1.32	0.229	0.142	0.149	0.128
J31VA	-16.5	-24.2	2544	0.111	8.83	6.30	4.24	3.44	0.207	0.159	0.180	0.160
J31VB	-16.7	-24.8	2850	0.095	5.71	3.61	2.08	1.84	0.200	0.153	0.193	0.168
J31VC	-16.2	-24.3	2854	0.094	9.70	6.89	4.39	3.56	0.207	0.160	0.179	0.158

Sample ID	T <sub>g1</sub> (°C)	T <sub>g1</sub> (°C)	Storage Modulus at T <sub>g1</sub> (MPa)	Tan $\delta$ at T <sub>g1</sub>	T <sub>g2</sub> (°C)	T <sub>g2</sub> (°C)	Storage Modulus at T <sub>g2</sub> (MPa)	Tan $\delta$ at T <sub>g2</sub>	T <sub>g3</sub> (°C)	T <sub>g3</sub> (°C)	Storage Modulus at T <sub>g3</sub> (MPa)	Tan $\delta$ at T <sub>g3</sub>
J275BHA	-61.4	-69.3	4173	0.099	-21.4	-26.0	436.9	0.1771	1.8	1.4	83.4	0.376
J275BHB	-60.8	-71.1	4287	0.086	-21.4	-26.4	498.2	0.1671	2.2	-3.0	154.9	0.231
J20BVA	-60.9	-70.5	4705	0.091	-9.0	-13.9	415.7	0.2231	NA	NA	NA	NA
J20BVB	-61.4	-71.0	4844	0.086	-9.7	-15.5	500.9	0.1832	NA	NA	NA	NA

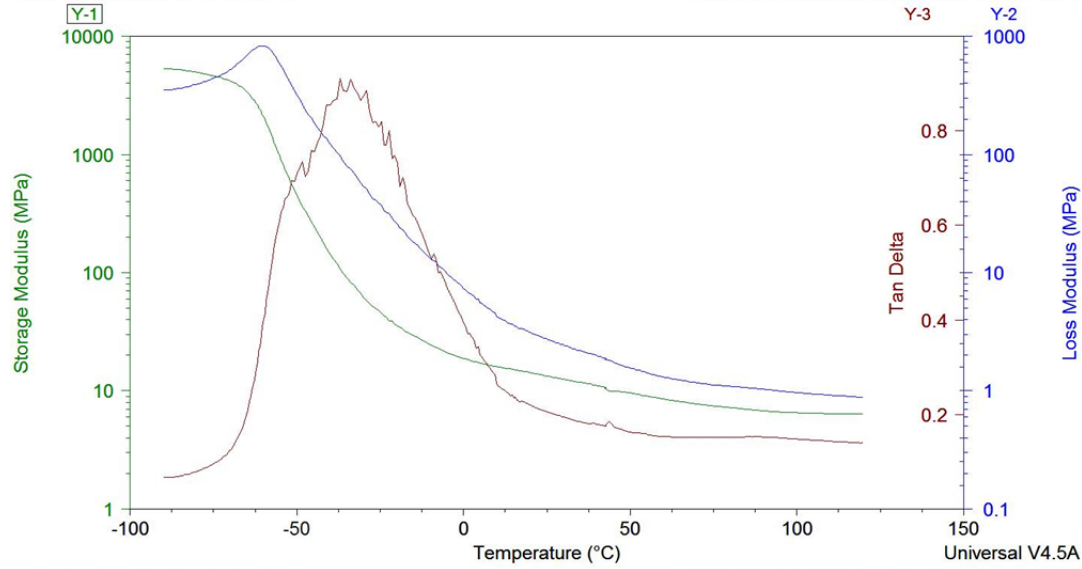
	Storage Modulus at 22°C (MPa)	Storage Modulus at 50°C (MPa)	Storage Modulus at 75°C (MPa)	Storage Modulus at 120°C (MPa)	Tan $\delta$ at 22	Tan $\delta$ at 50°C	Tan $\delta$ at 90°C	Tan $\delta$ at 120°C
J275BHA	10.17	4.97	3.49	3.80	0.236	0.148	0.141	0.119
J275BHB	8.48	3.44	2.65	2.89	0.244	0.139	0.133	0.110
J20BVA	8.03	6.32	5.72	4.76	0.180	0.156	0.187	0.184
J20BVB	8.68	6.35	6.07	5.01	0.190	0.152	0.189	0.185

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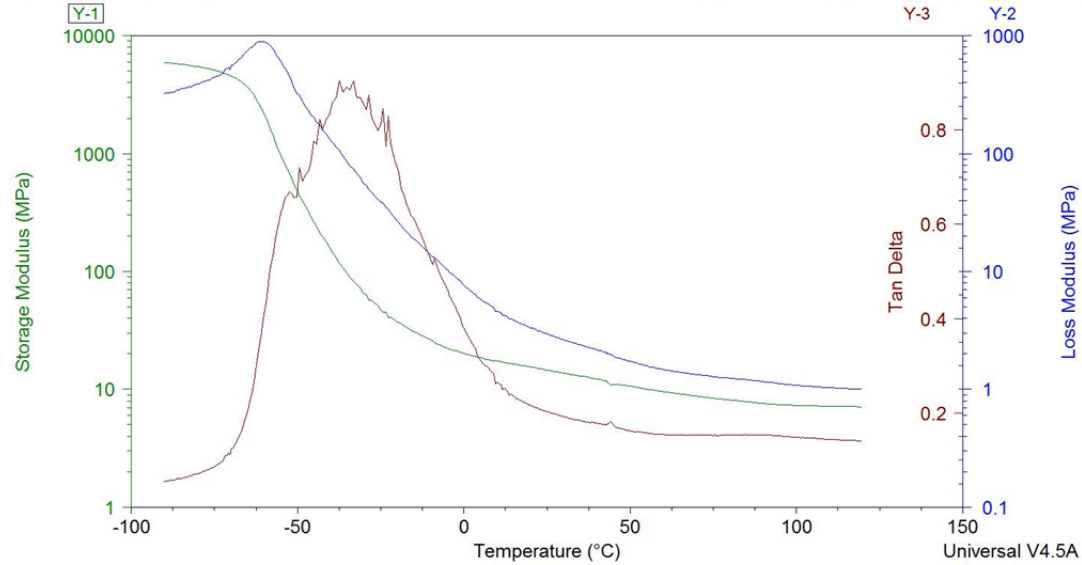
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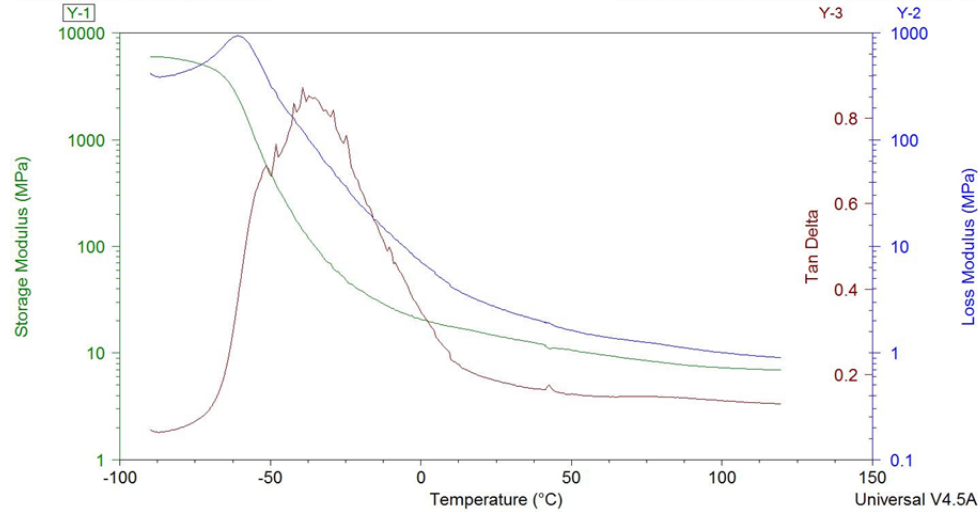
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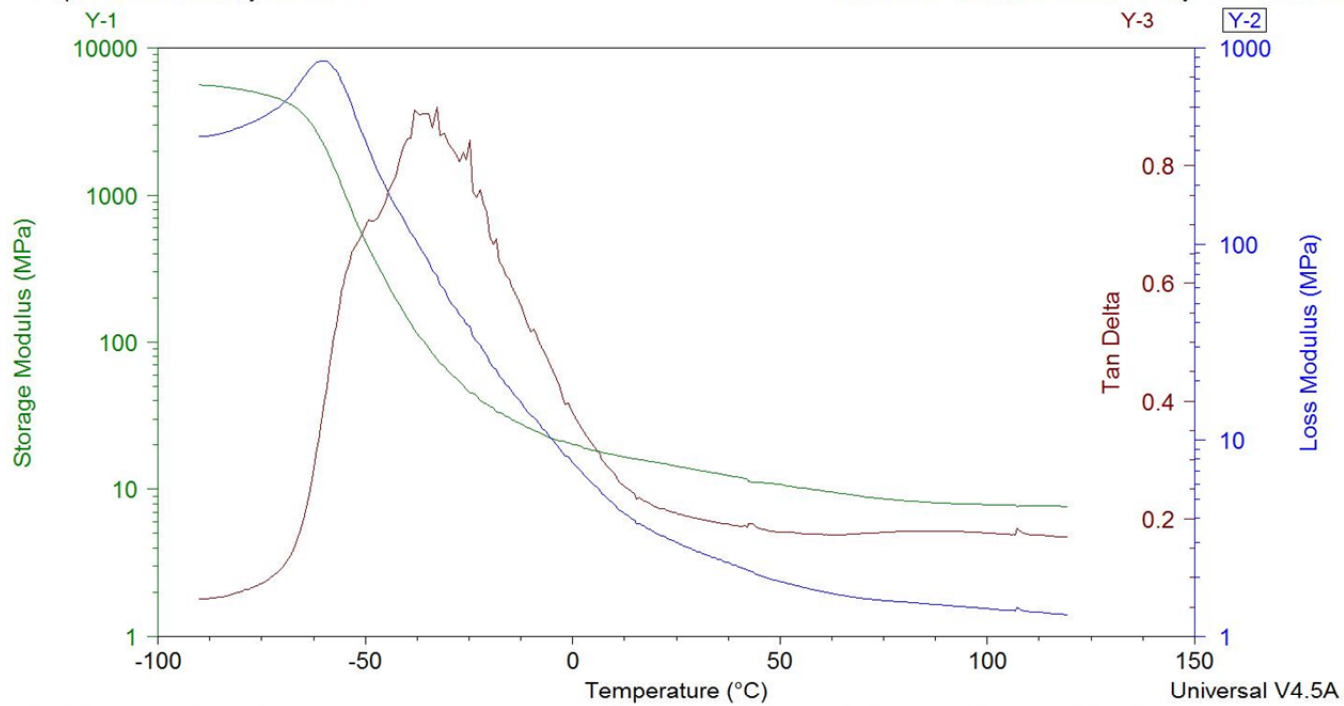
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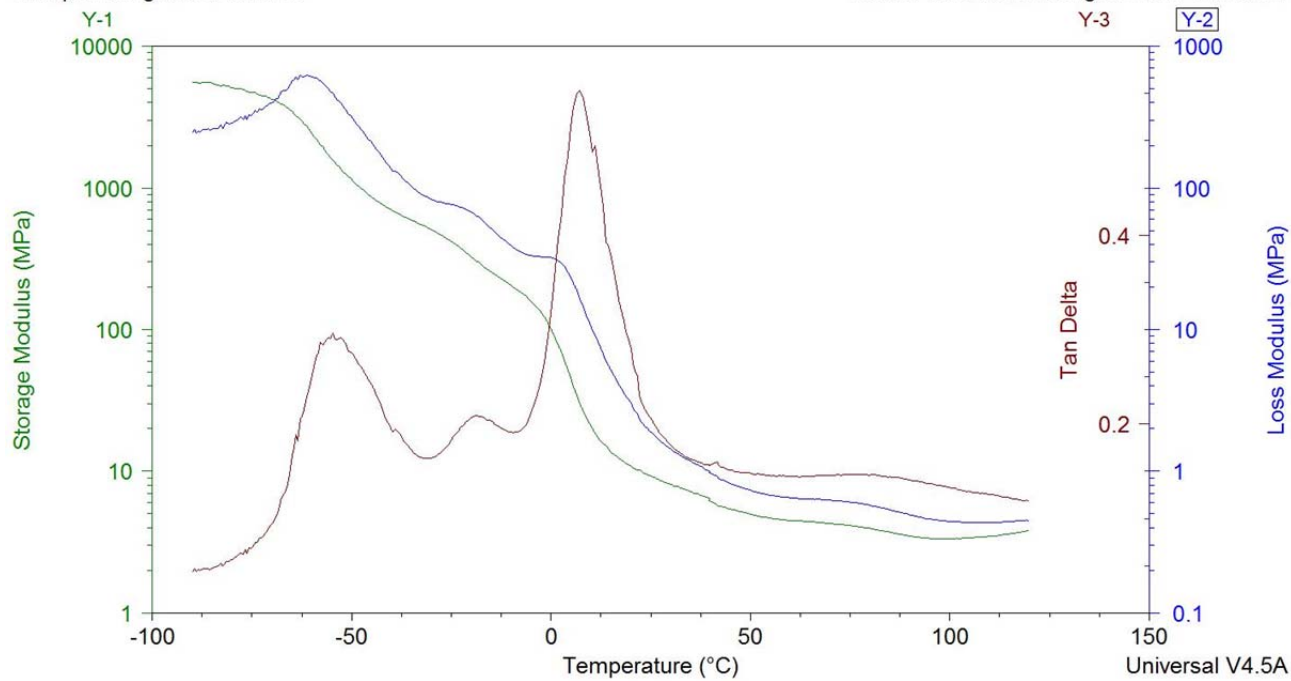
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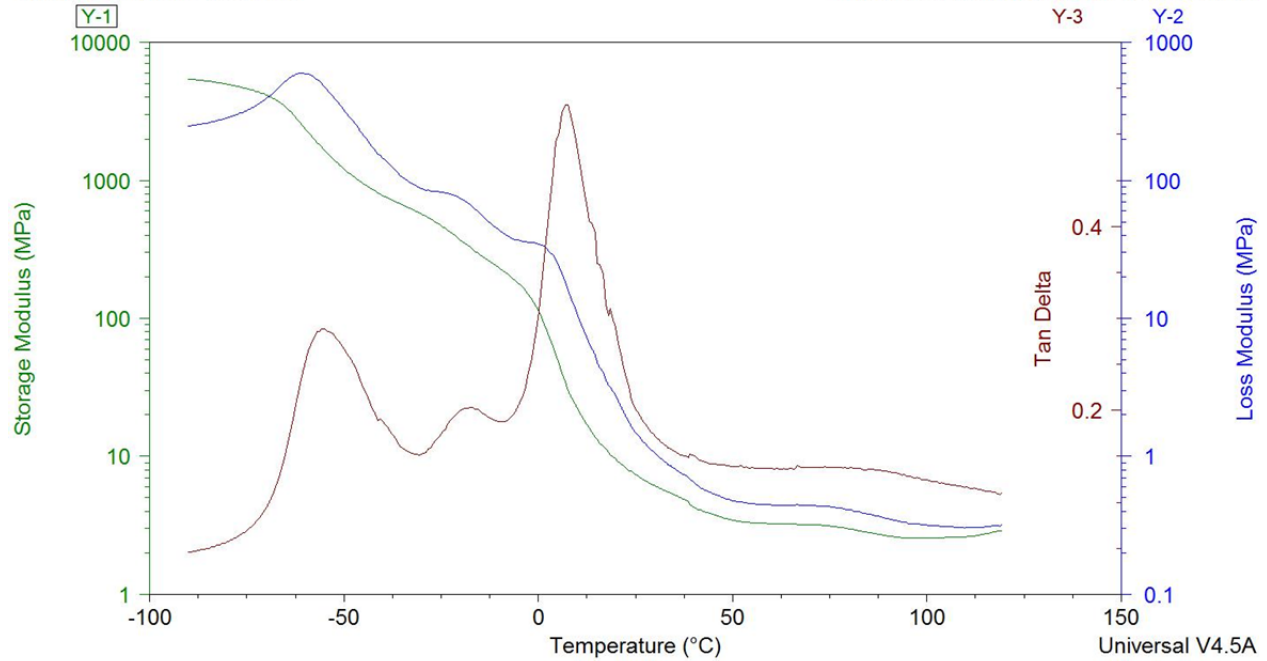
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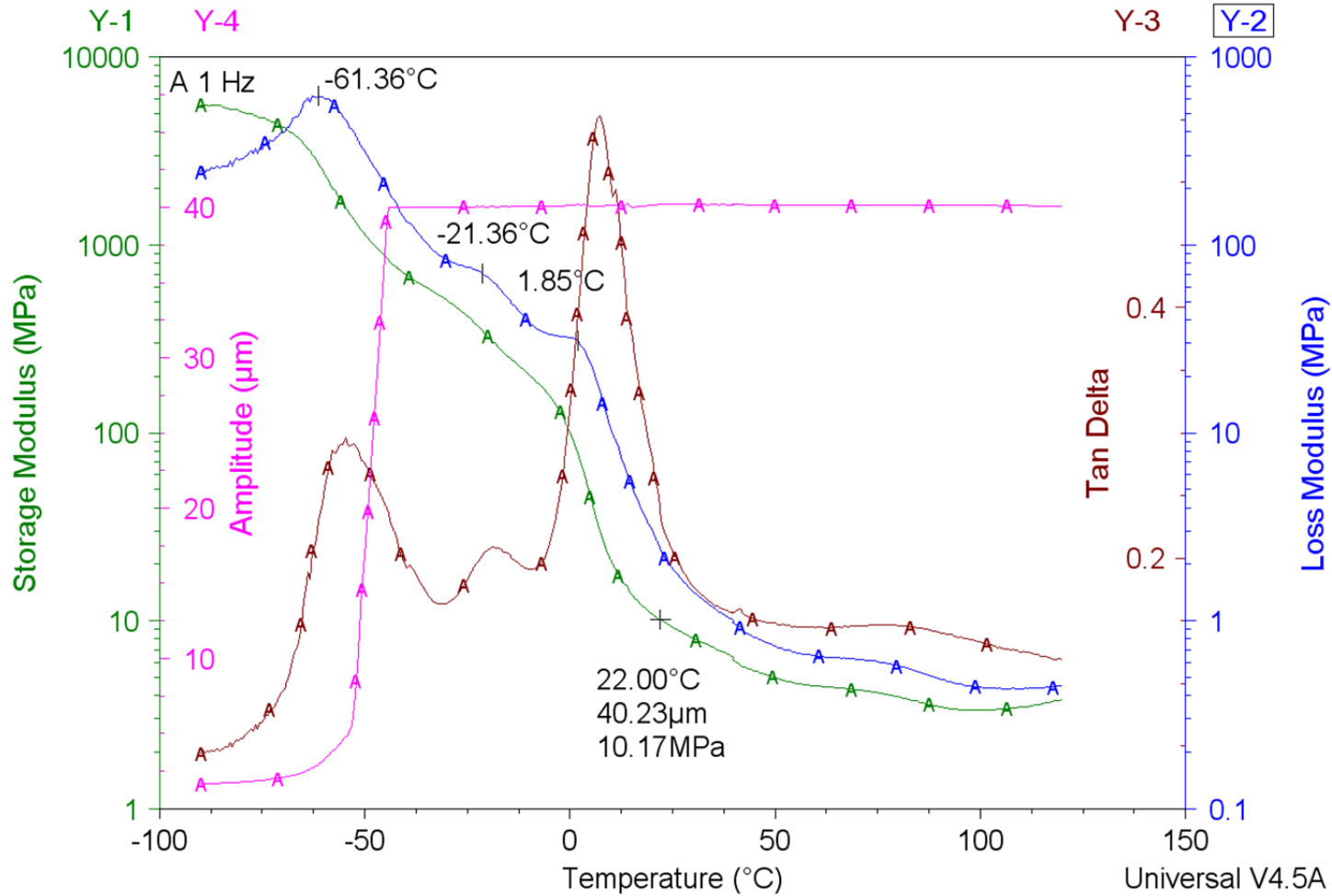
Sample: Jung BH 27.5 mil B

DMA File: F:\DMA\Jung BH 27.5 mil B.001



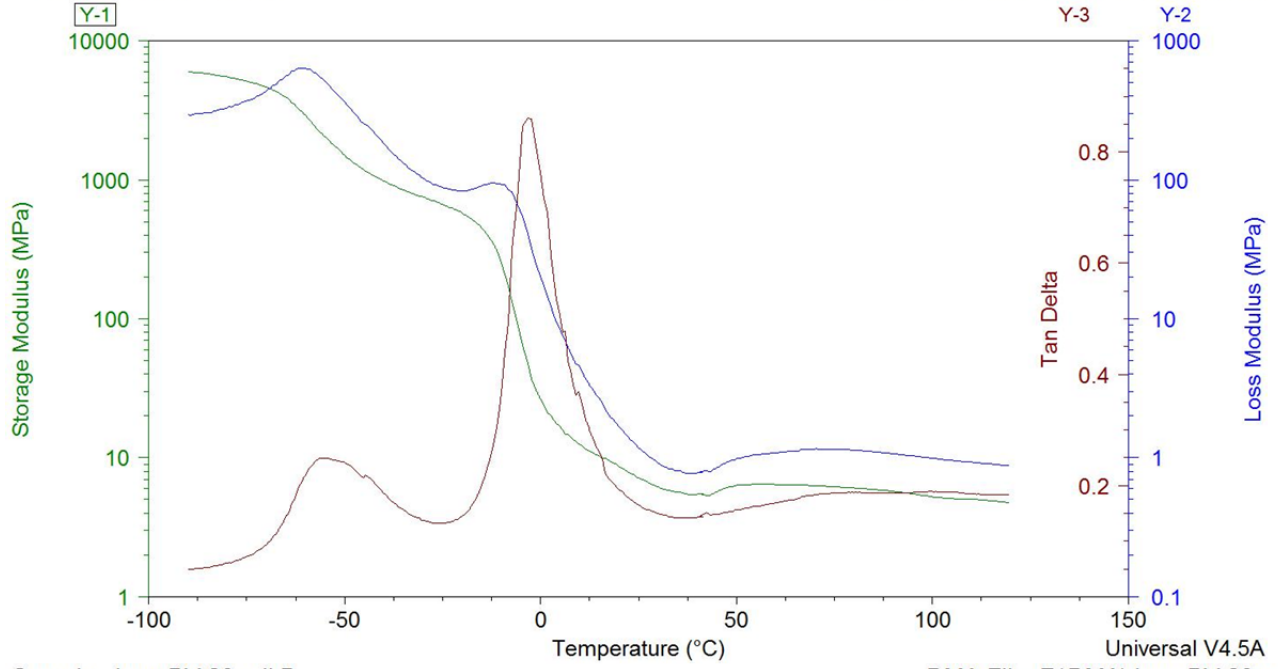
Sample: Jung BH 27.5 mil A

DMA File: C:\...DMA\Nina Breakiron\Jung BH 27.5 mil A.001



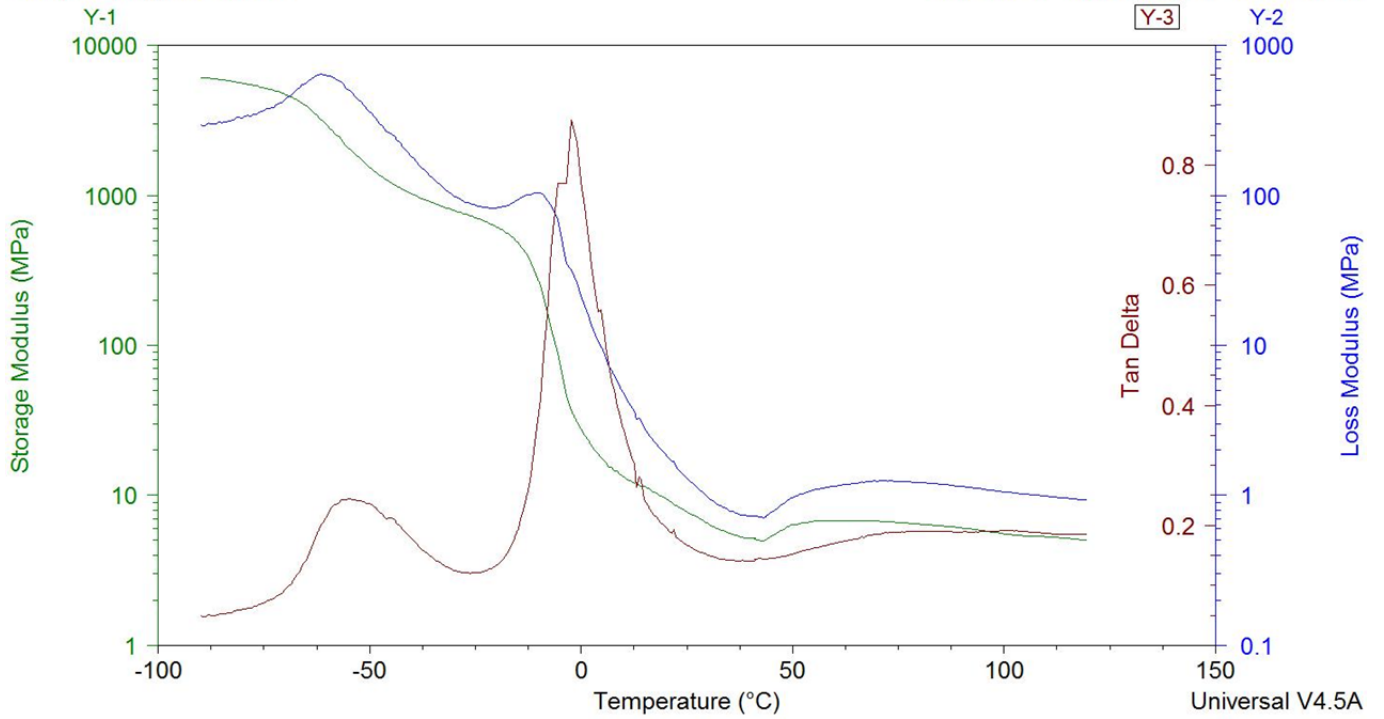
Sample: Jung BV 20 mil A

DMA File: F:\DMA\Jung BV 20 mil A.001



Sample: Jung BV 20 mil B

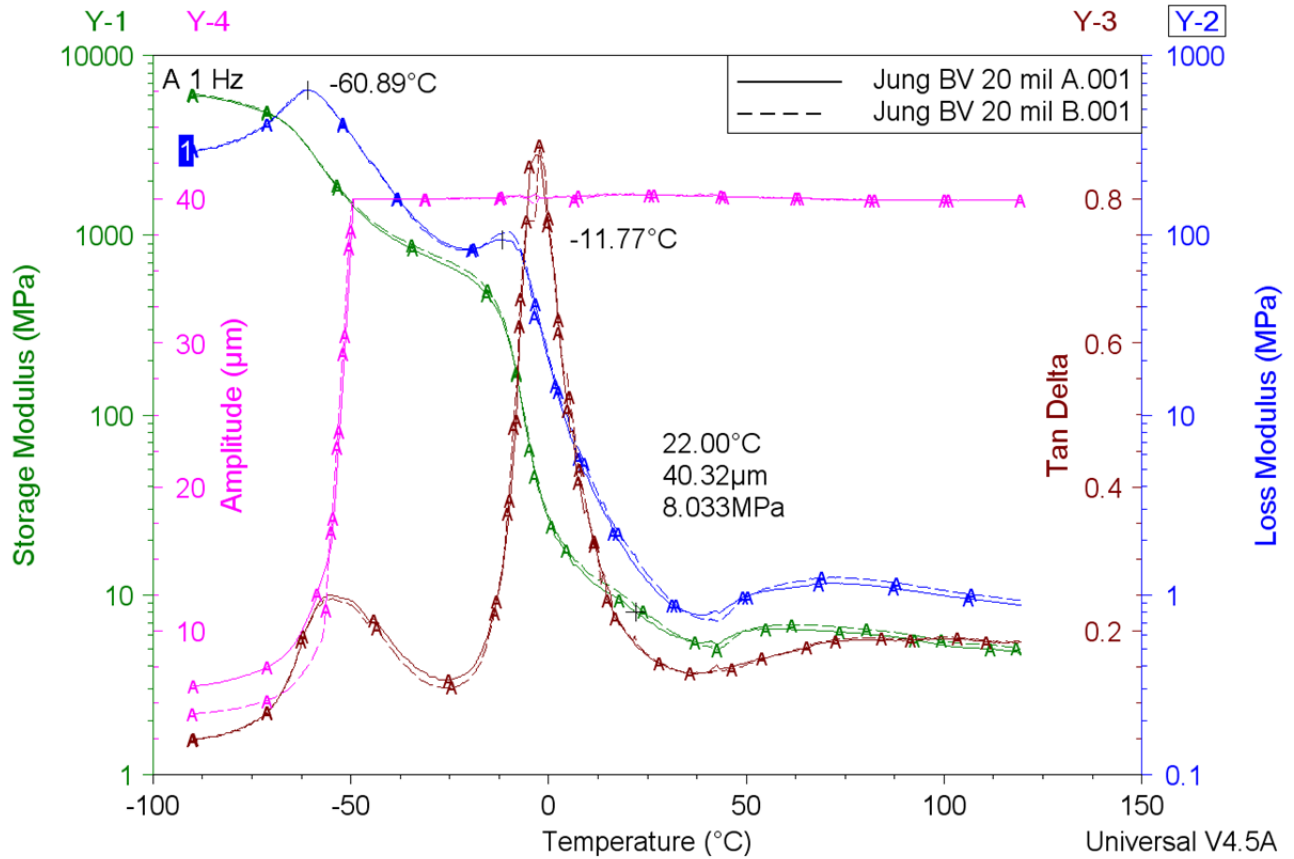
DMA File: F:\DMA\Jung BV 20 mil B.001





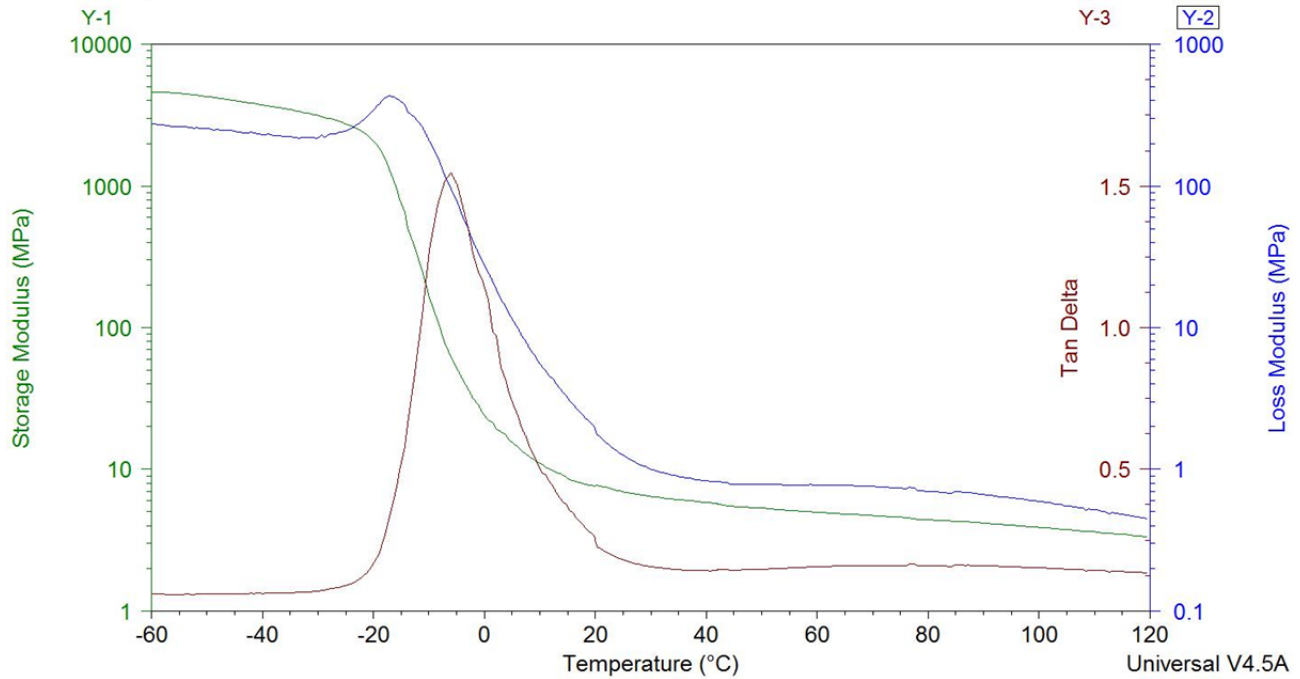
Curve 1: Jung BV 20 mil A

DMA File: C:\TA\Data\DMA\Nina Breakiron\Jung BV 20 mil A.001



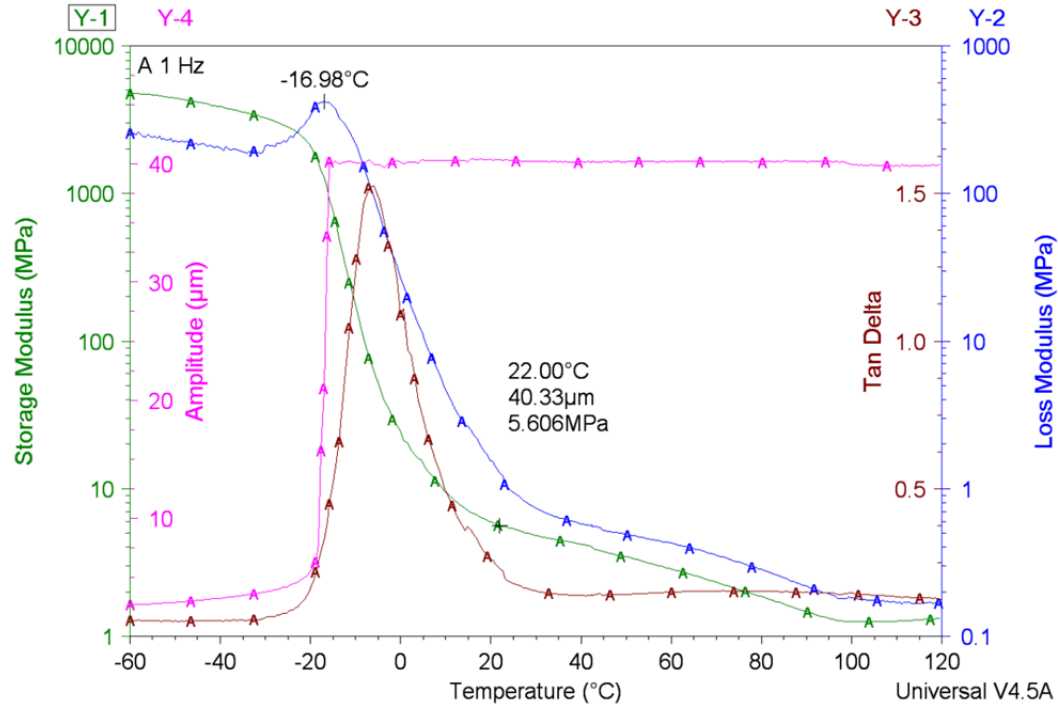
Sample: Jung Viton 24 mil A

DMA File: F:\DMA\Jung Viton 24 mil A.001



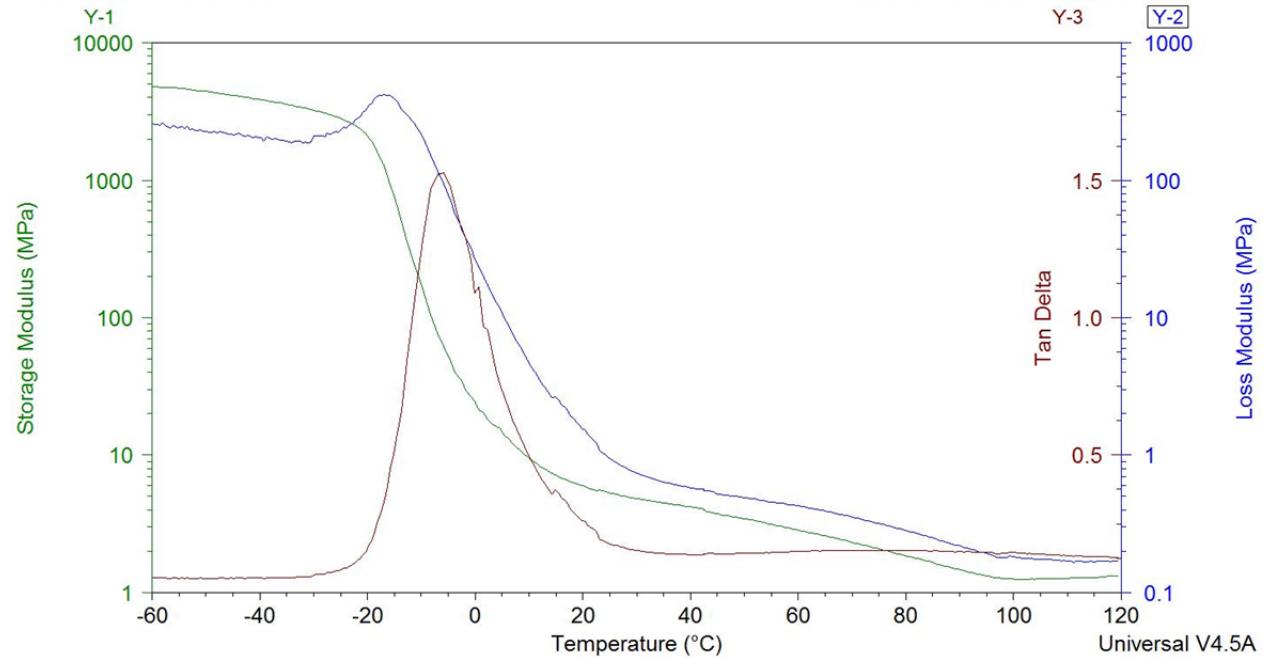
Sample: Jung Viton 24 mil B

DMA File: C:\...DMA\Nina Breakiron\Jung Viton 24 mil B.001



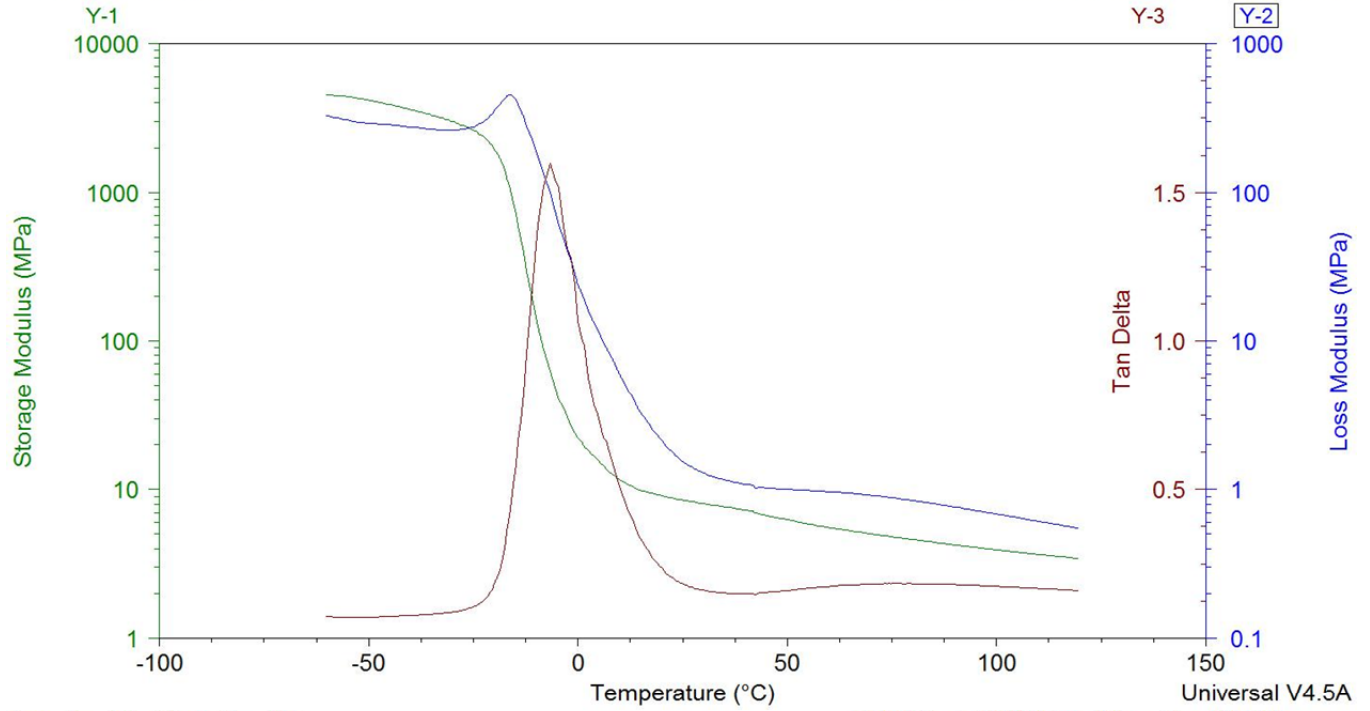
Sample: Jung Viton 24 mil B

DMA File: F:\DMA\Jung Viton 24 mil B.001



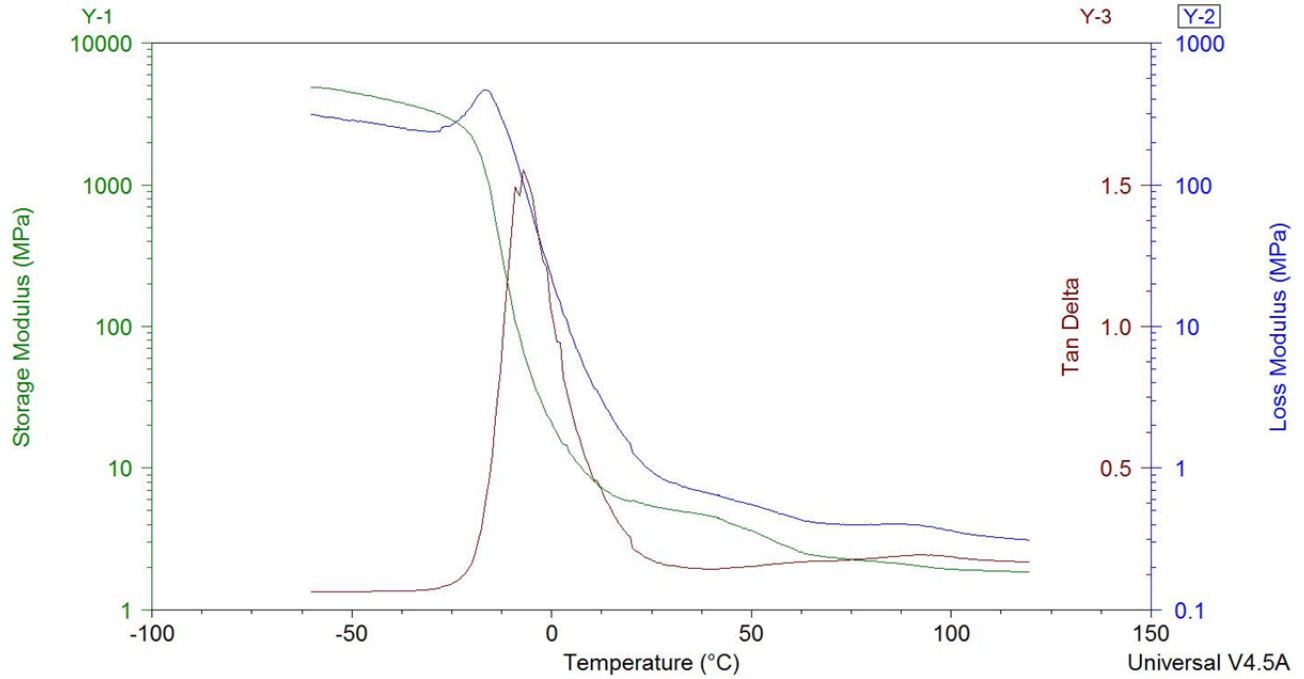
Sample: Jung Viton 31 mil A

DMA File: F:\DMA\Jung Viton 31 mil A.001



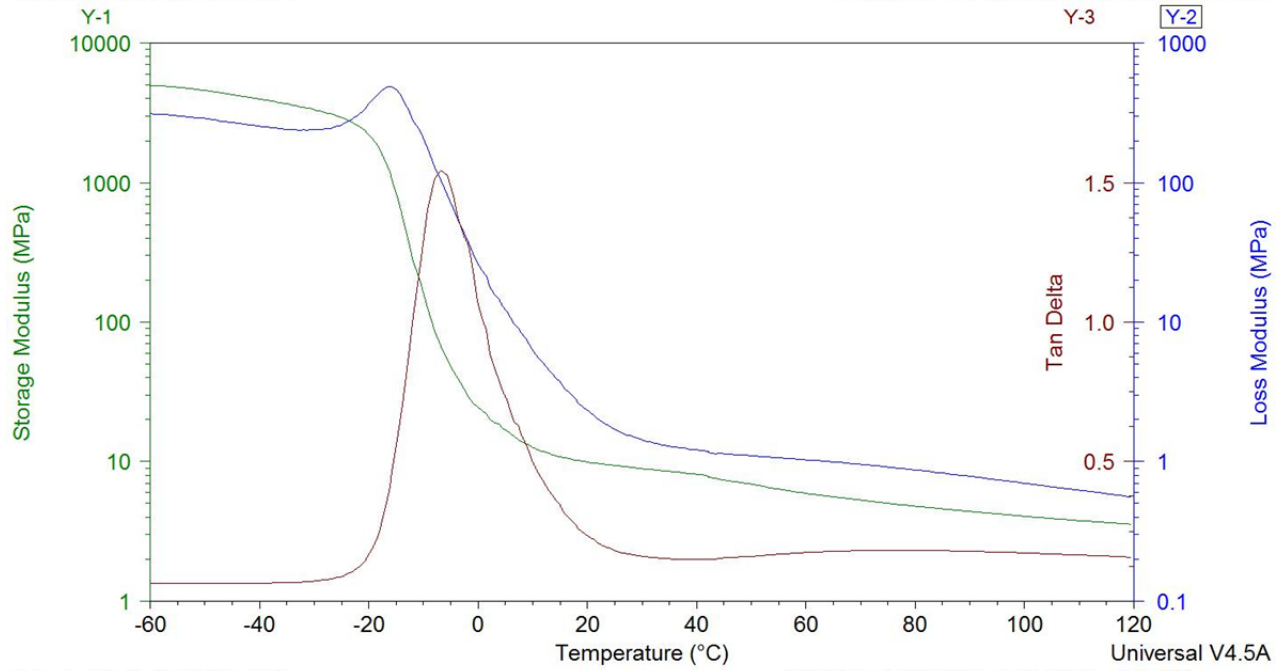
Sample: Jung Viton 31 mil B

DMA File: F:\DMA\Jung Viton 31 mil B.001



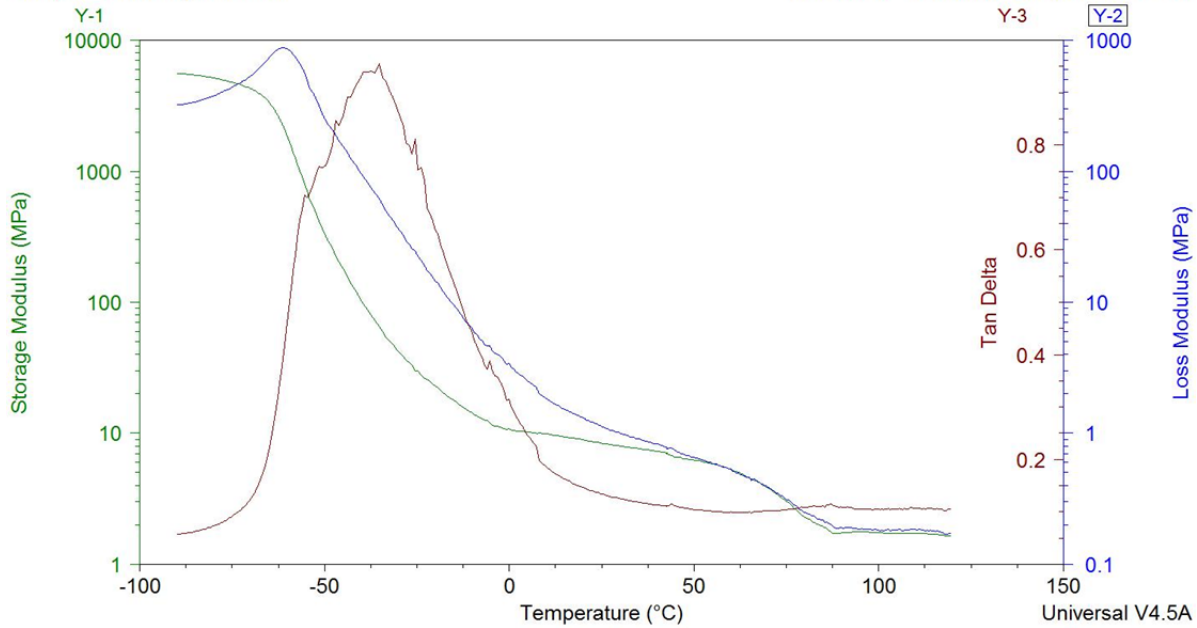
Sample: Jung Viton 31 mil C

DMA File: F:\DMA\Jung Viton 31 mil C.001



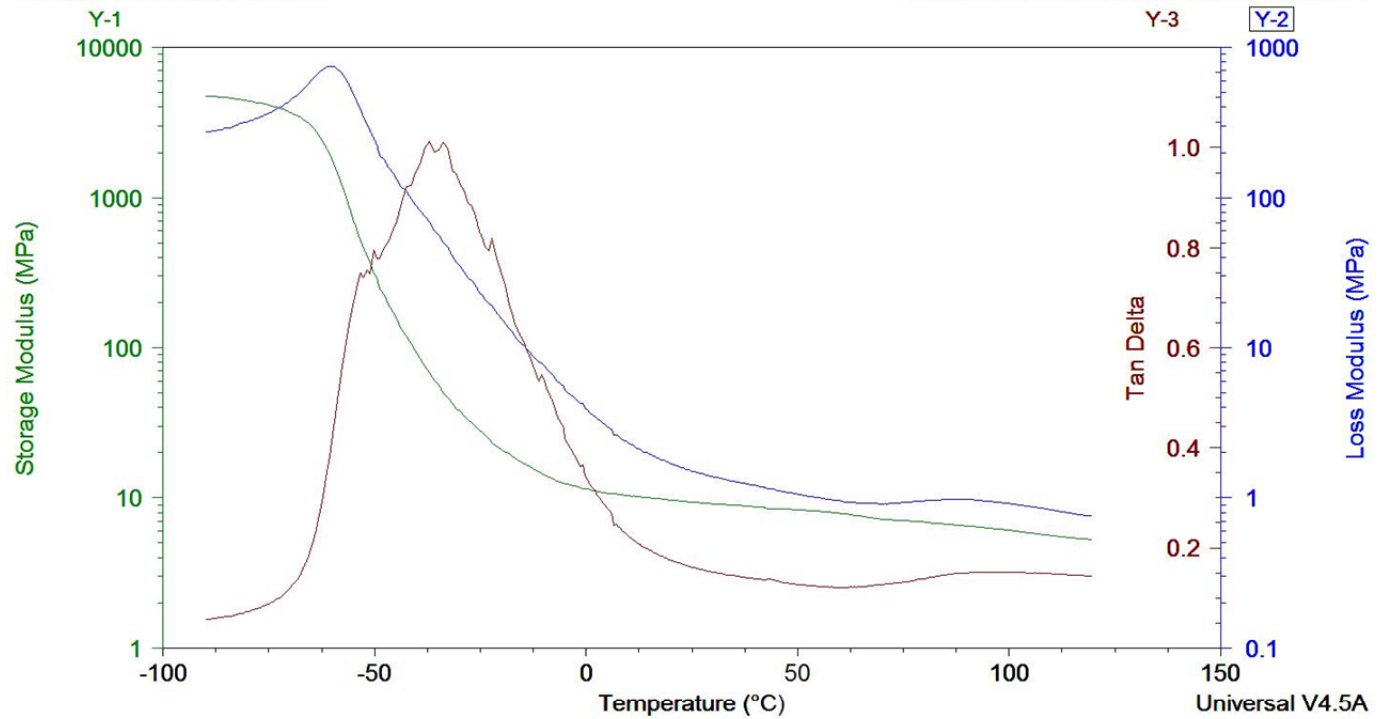
Sample: North Butyl 15 mil A

DMA File: F:\DMA\North Butyl 15 mil A.001



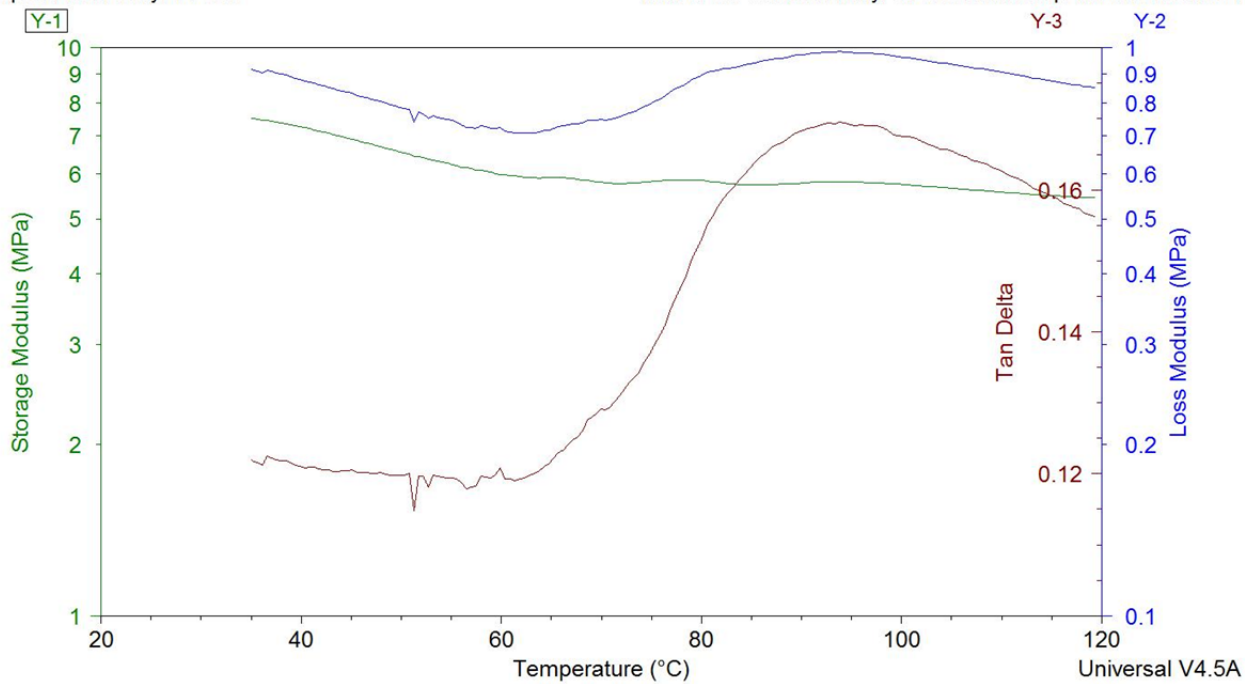
Sample: North Butyl 15 mil B

DMA File: F:\DMA\North Butyl 15 mil B.001



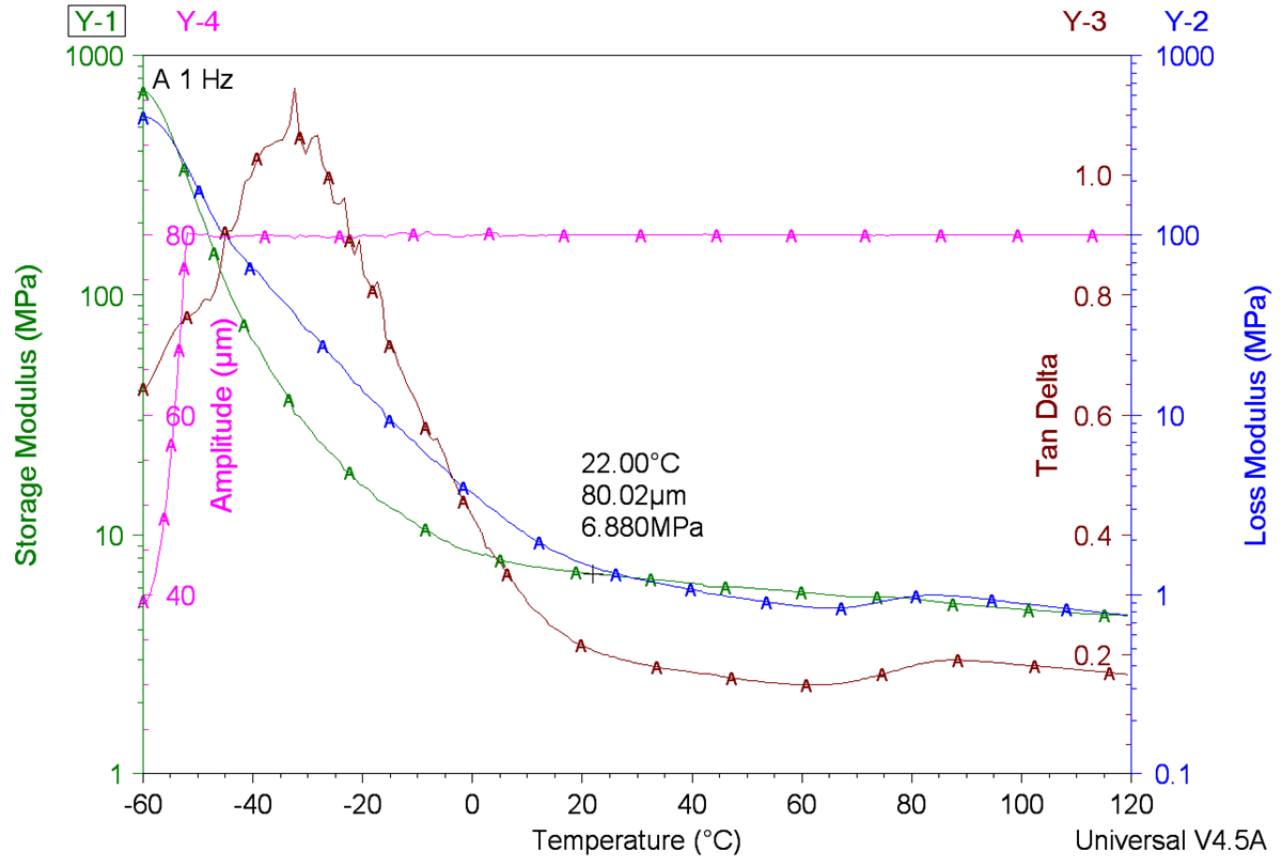
Sample: North Butyl 30 mil

DMA File: ...North Butyl 30 mil multi-freq DMA tension.001



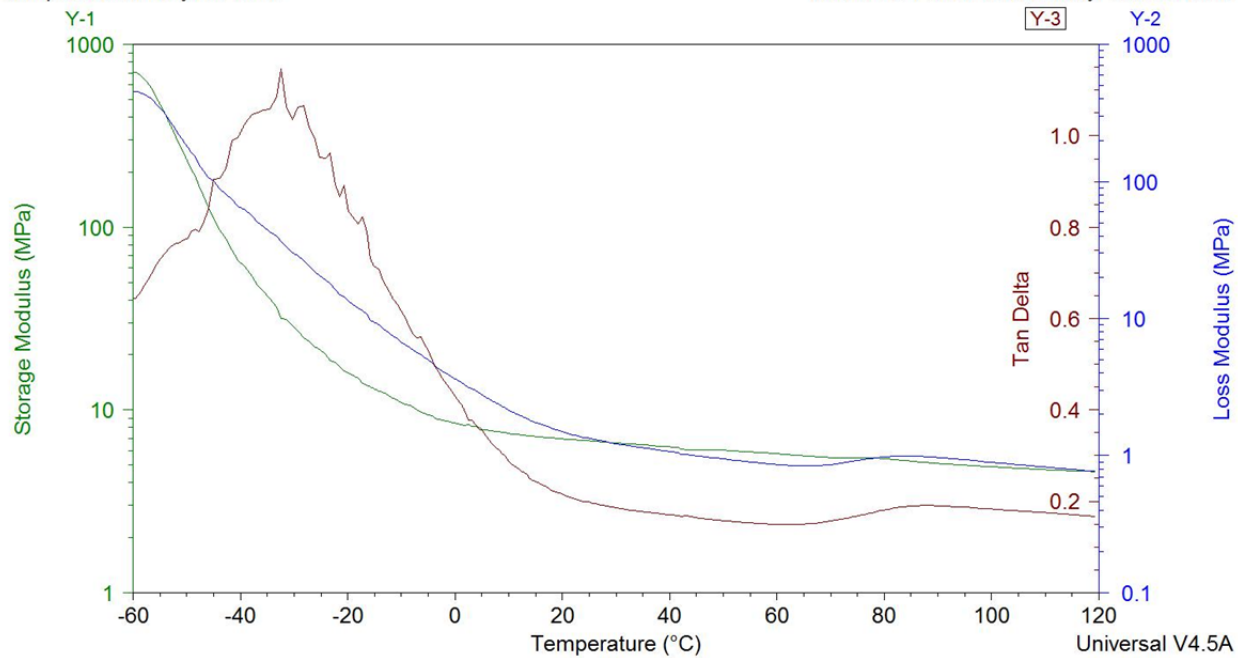
Sample: North Butyl 30 mil B

DMA File: C:\...Nina Breakiron\North Butyl 30 mil B.001



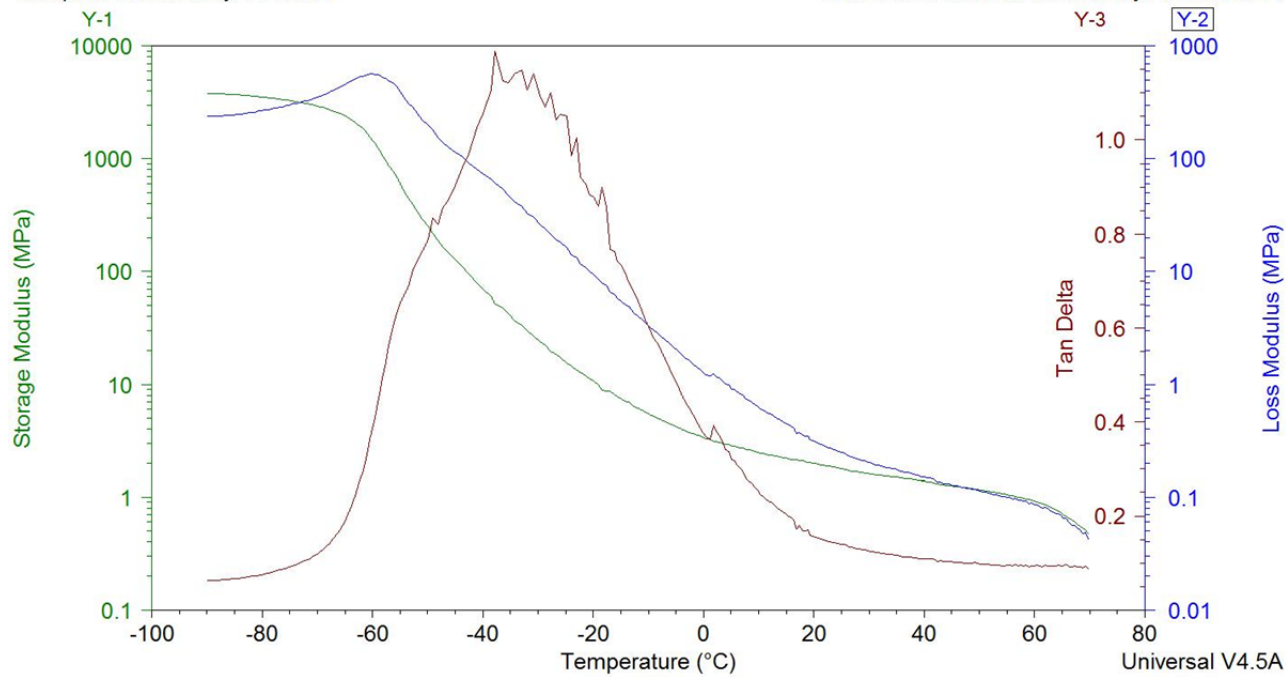
Sample: North Butyl 30 mil B

DMA File: F:\DMA\North Butyl 30 mil B.001



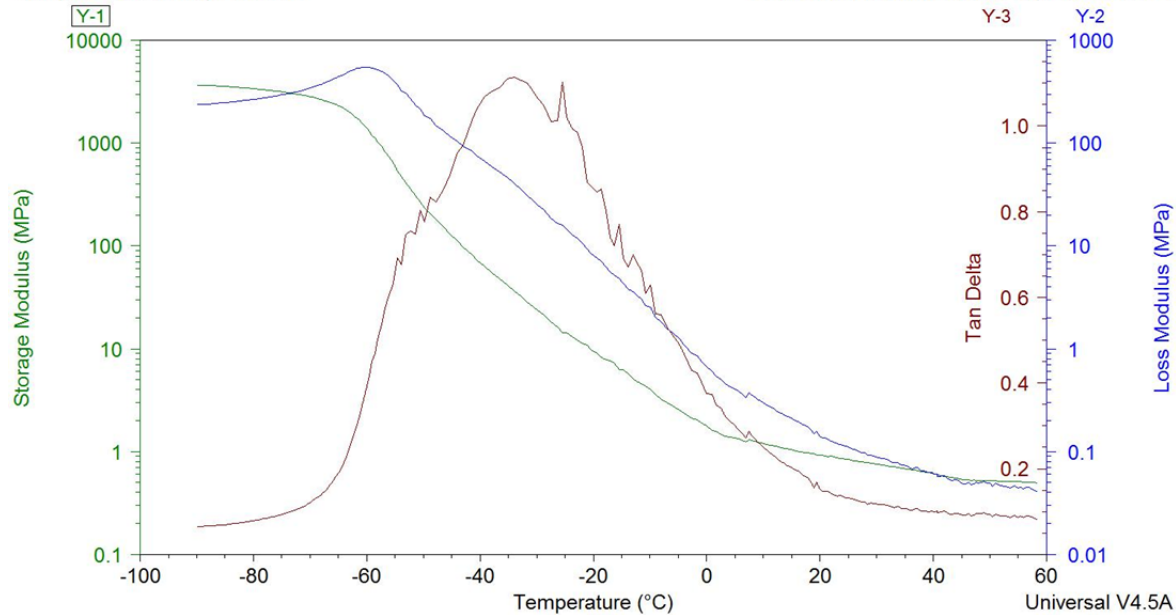
Sample: Piercan Butyl 15 mil A

DMA File: F:\DMA\Piercan Butyl 15 mil A.001



Sample: Piercan Butyl 15 mil B

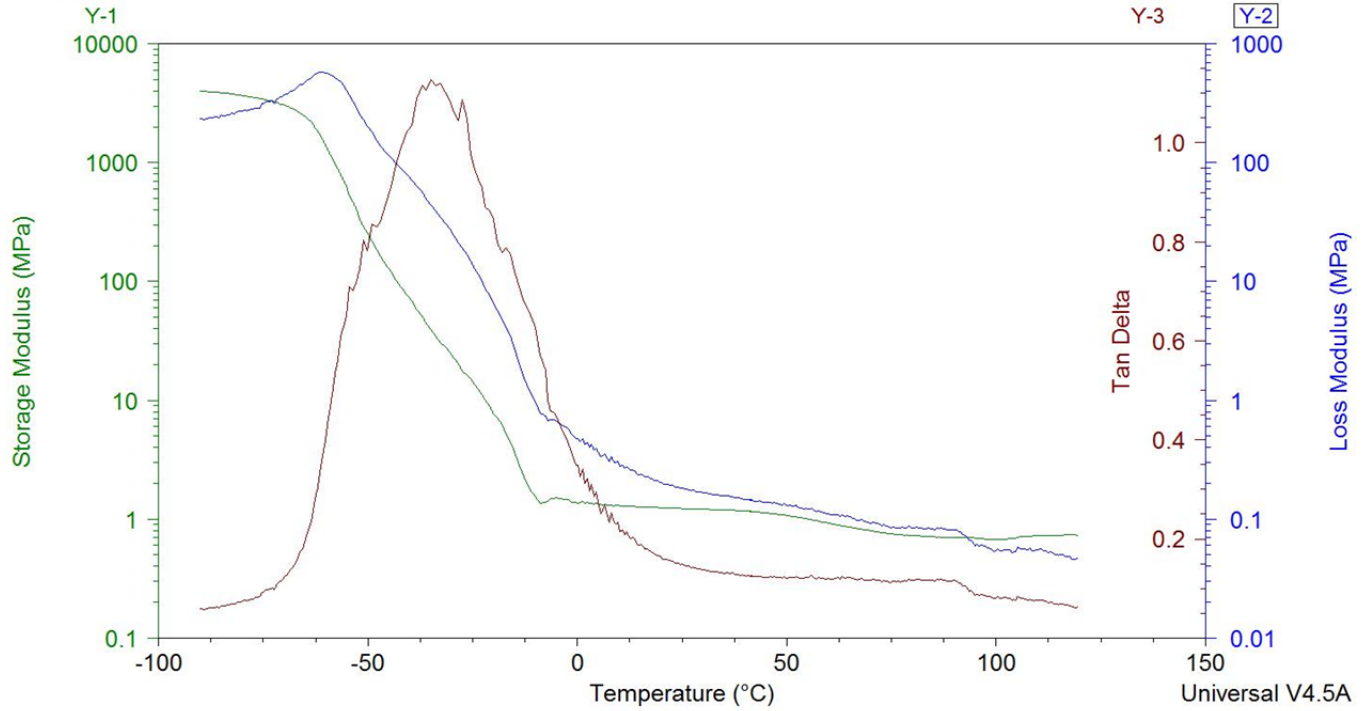
DMA File: F:\DMA\Piercan Butyl 15 mil B.001





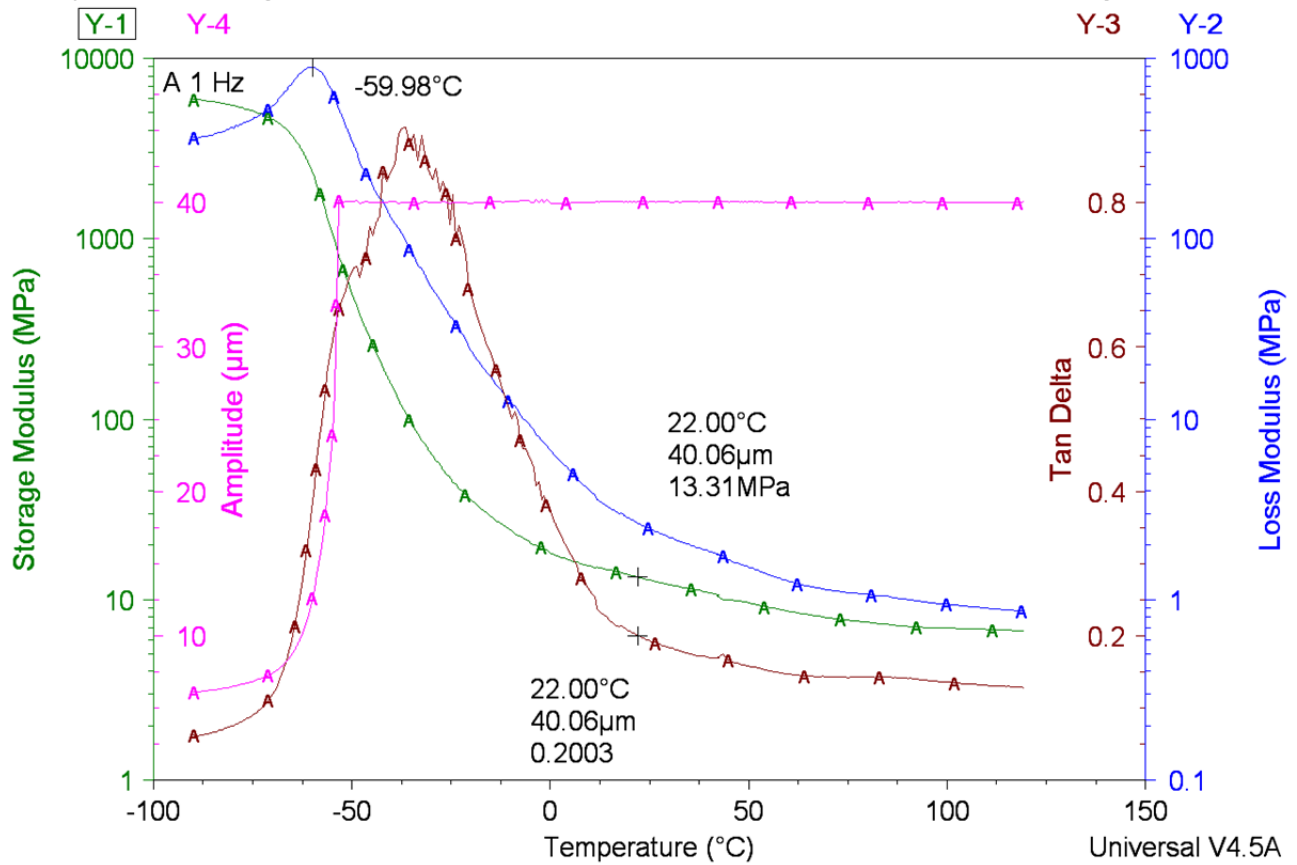
Sample: Piercan Butyl 15 mil C

DMA File: F:\DMA\Piercan Butyl 15 mil C.001



Sample: Piercan Butyl 30 mil A

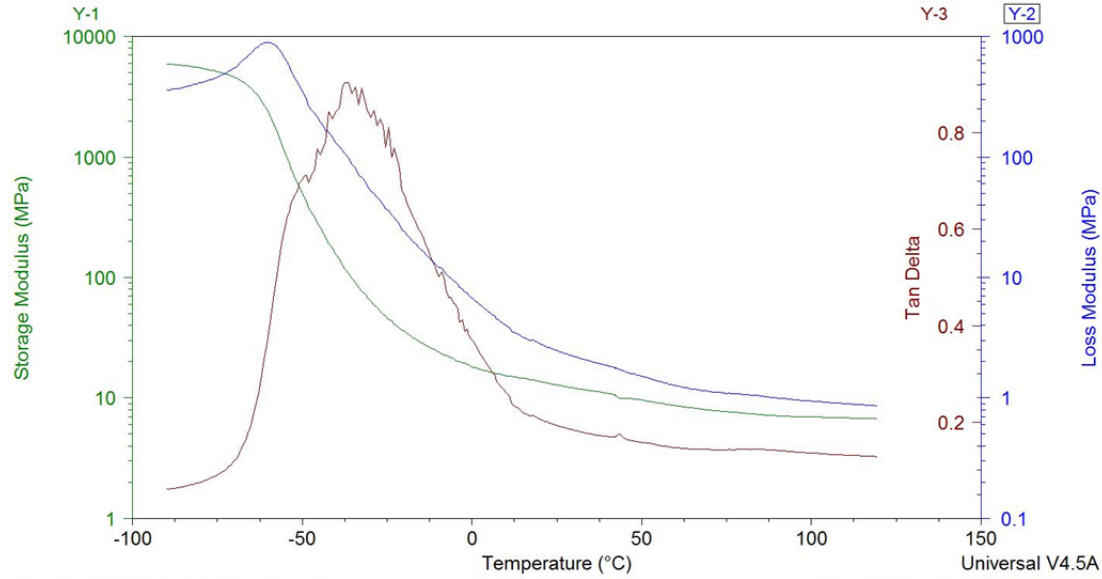
DMA File: C:\...Piercan Butyl 30 mil A.001





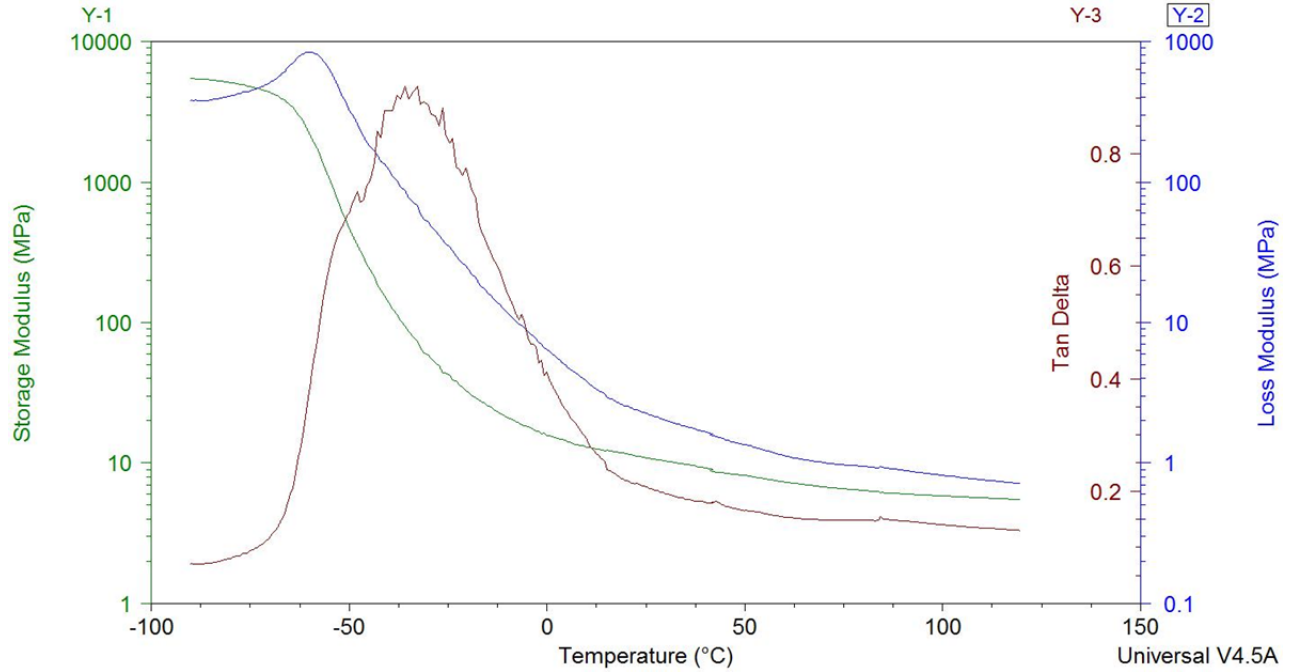
Sample: Piercan Butyl 30 mil A

DMA File: F:\DMA\Piercan Butyl 30 mil A.001



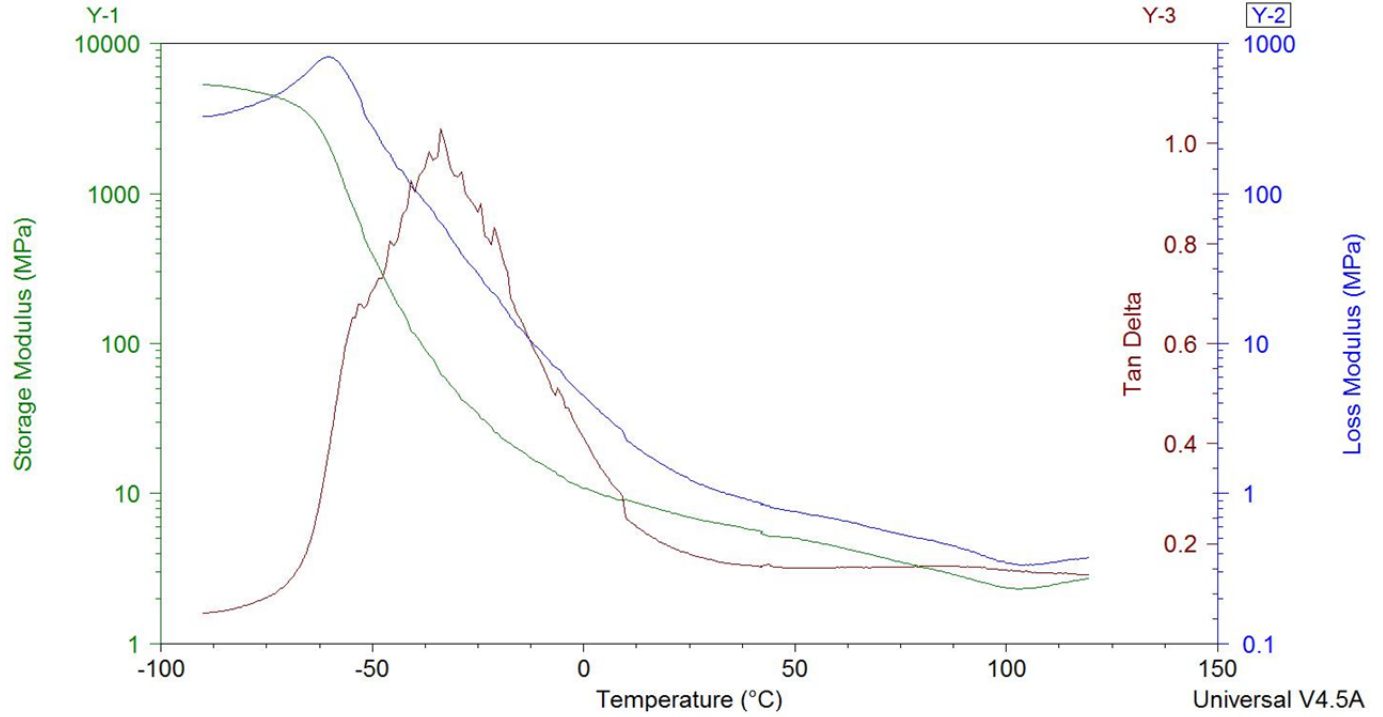
Sample: Piercan Butyl 30 mil B

DMA File: F:\DMA\Piercan Butyl 30 mil B.001



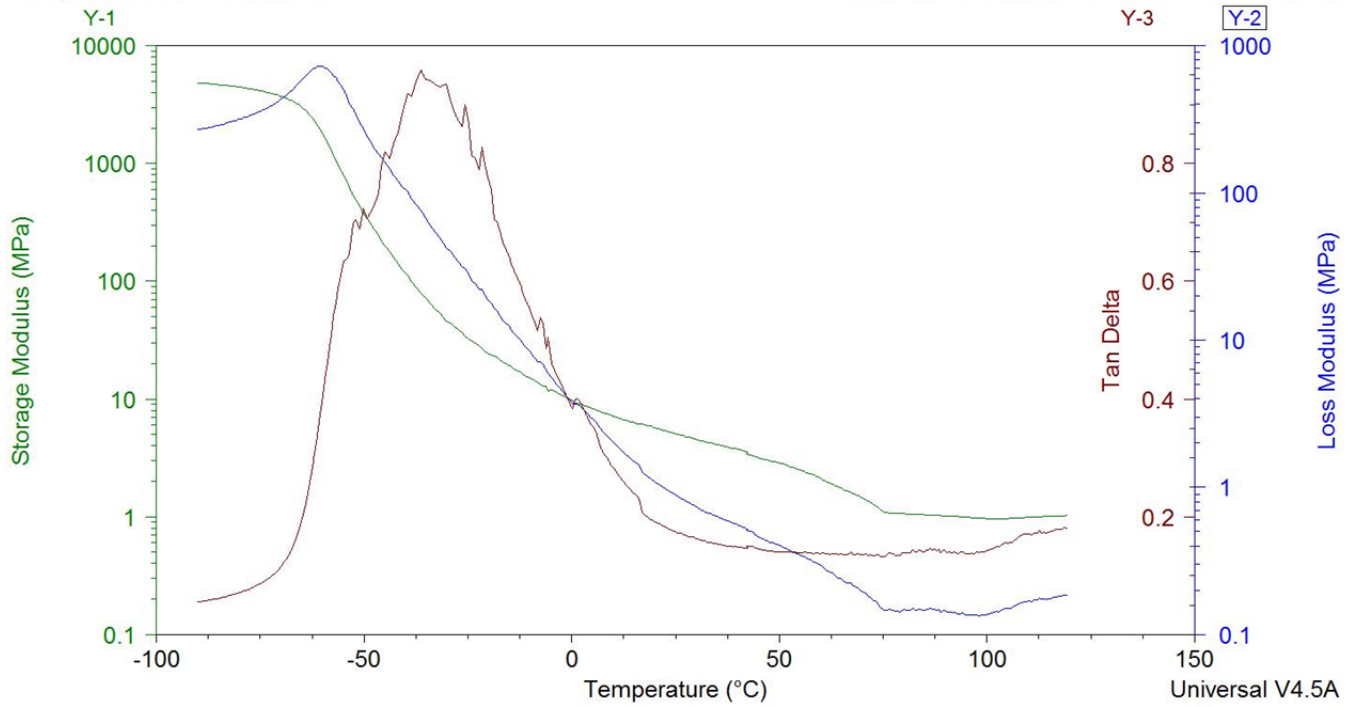
Sample: Piercan ESDB 15 mil A

DMA File: F:\DMA\Piercan ESDB 15 mil A.001



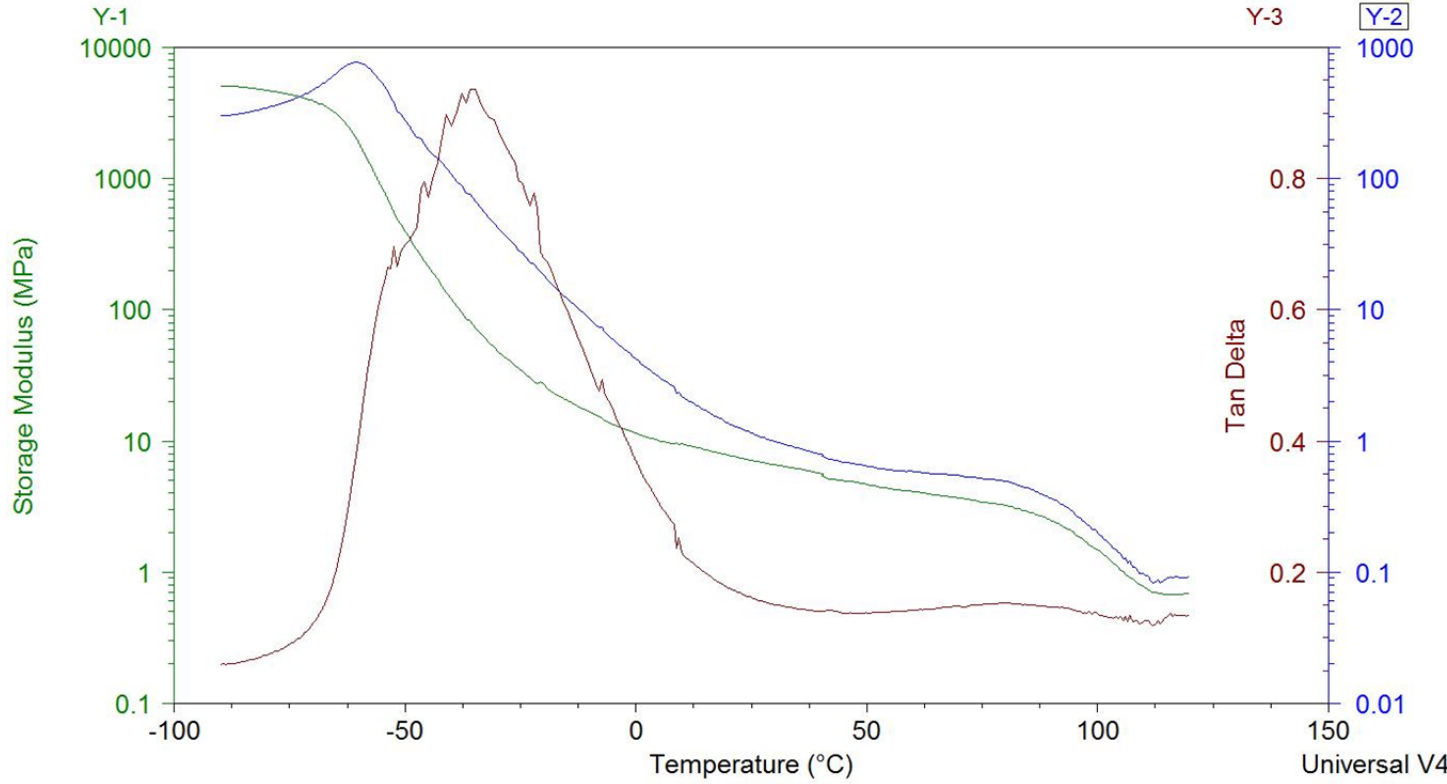
Sample: Piercan ESDB 15 mil B

DMA File: F:\DMA\Piercan ESDB 15 mil B.001



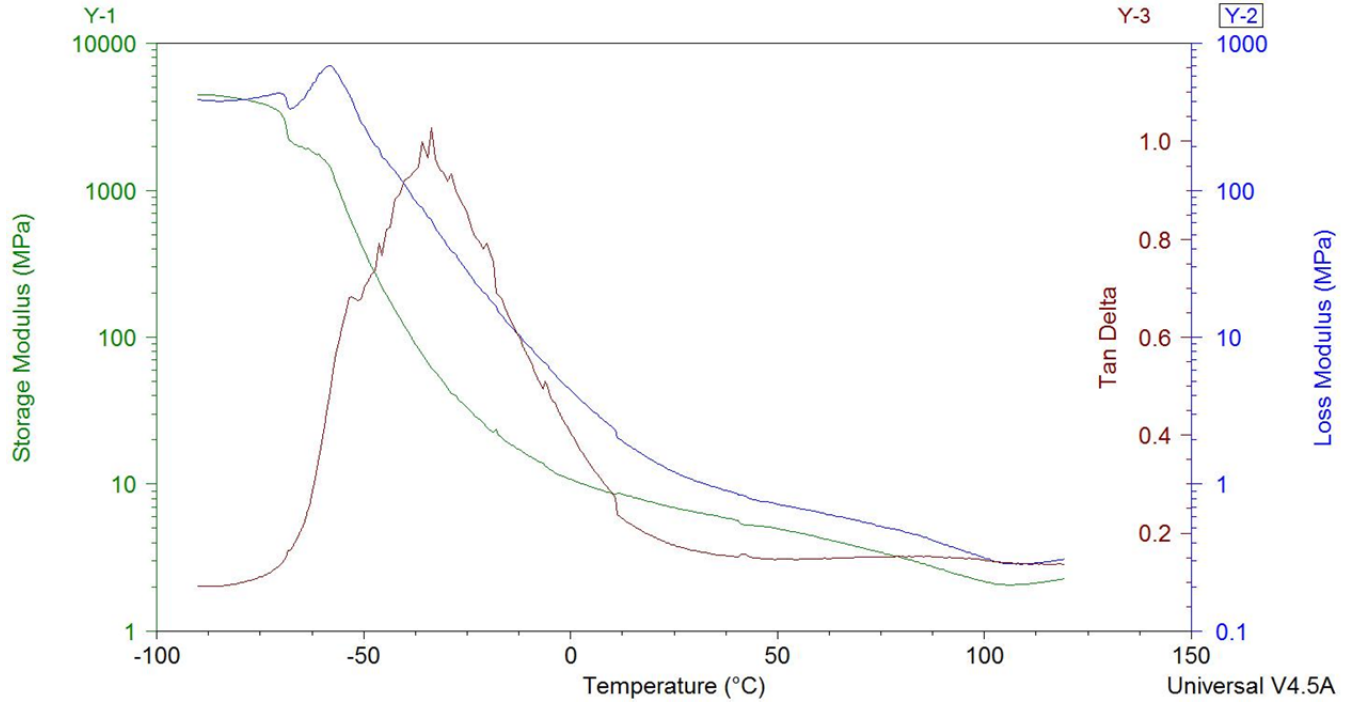
Sample: Piercan ESDB 24 mil A

DMA File: F:\DMA\Piercan ESDB 24 mil A.



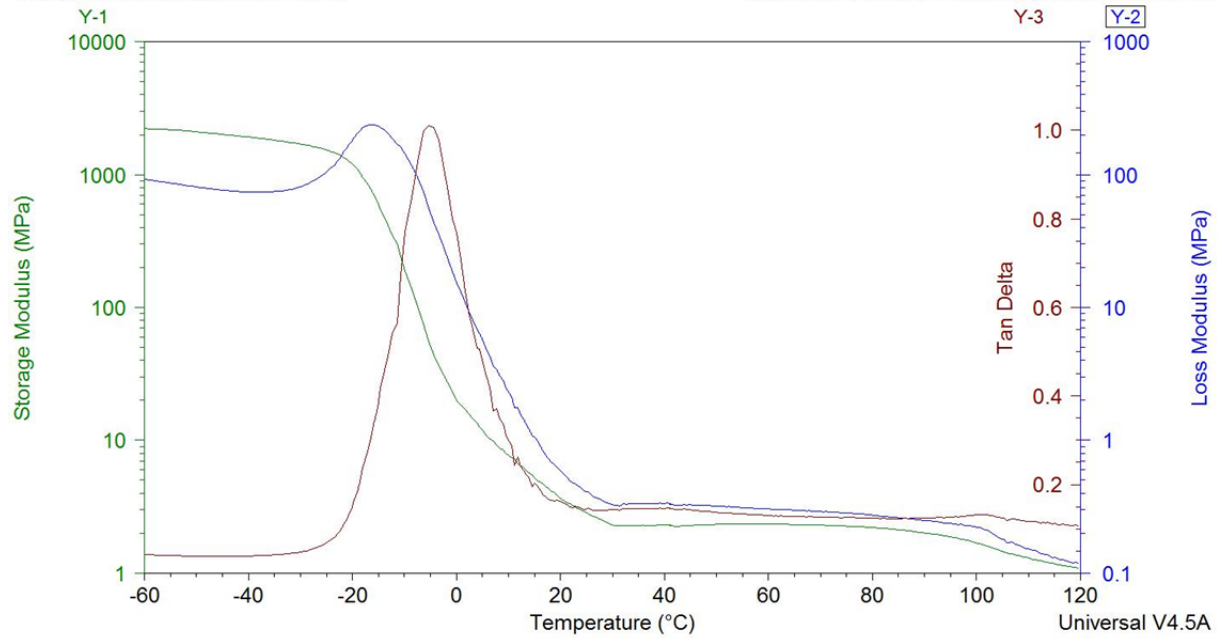
Sample: Piercan ESDB 24 mil B

DMA File: F:\DMA\Piercan ESDB 24 mil B.001



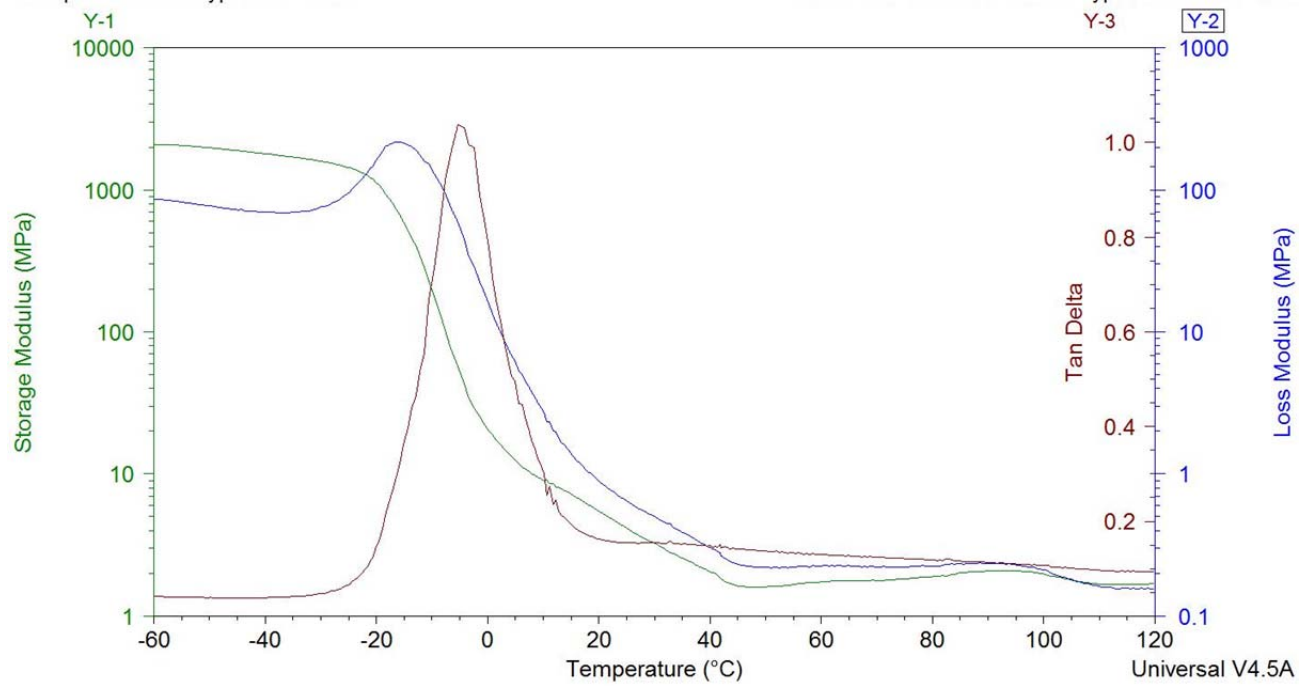
Sample: Piercan Hypalon 25 mil A

DMA File: F:\DMA\Piercan Hypalon 25 mil A.001



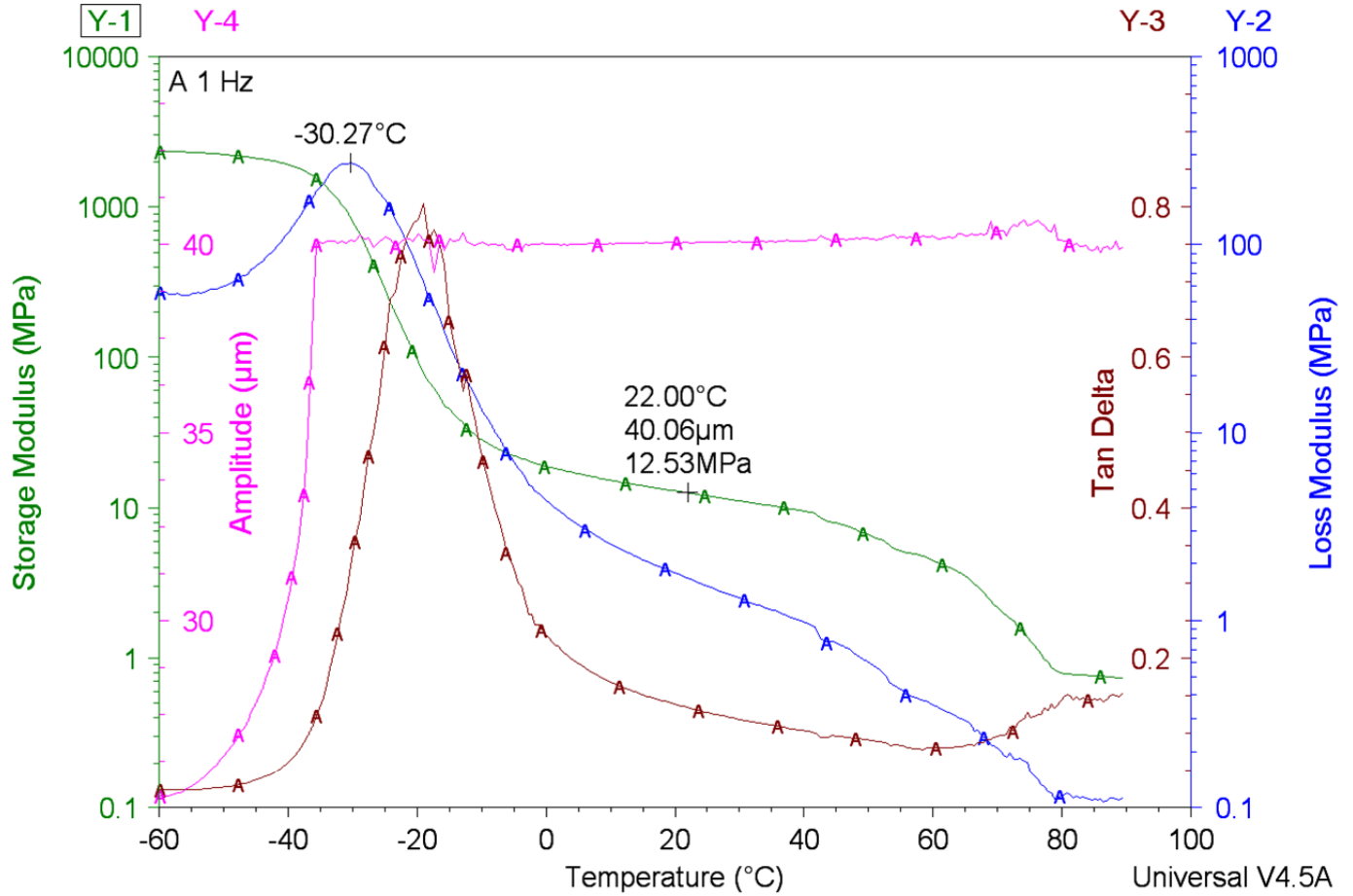
Sample: Piercan Hypalon 25 mil B

DMA File: F:\DMA\Piercan Hypalon 25 mil B.001



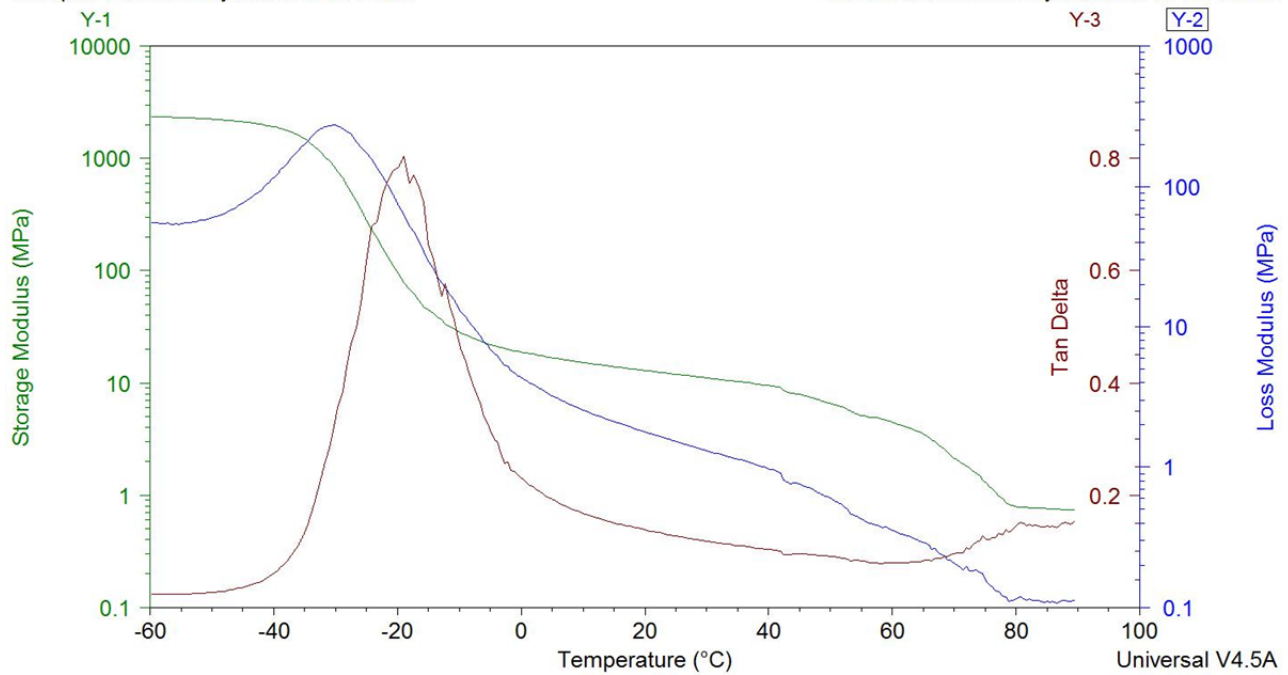
Sample: Piercan Polyurethane 15 mil A

DMA File: Piercan Polyurethane 15 mil A.001



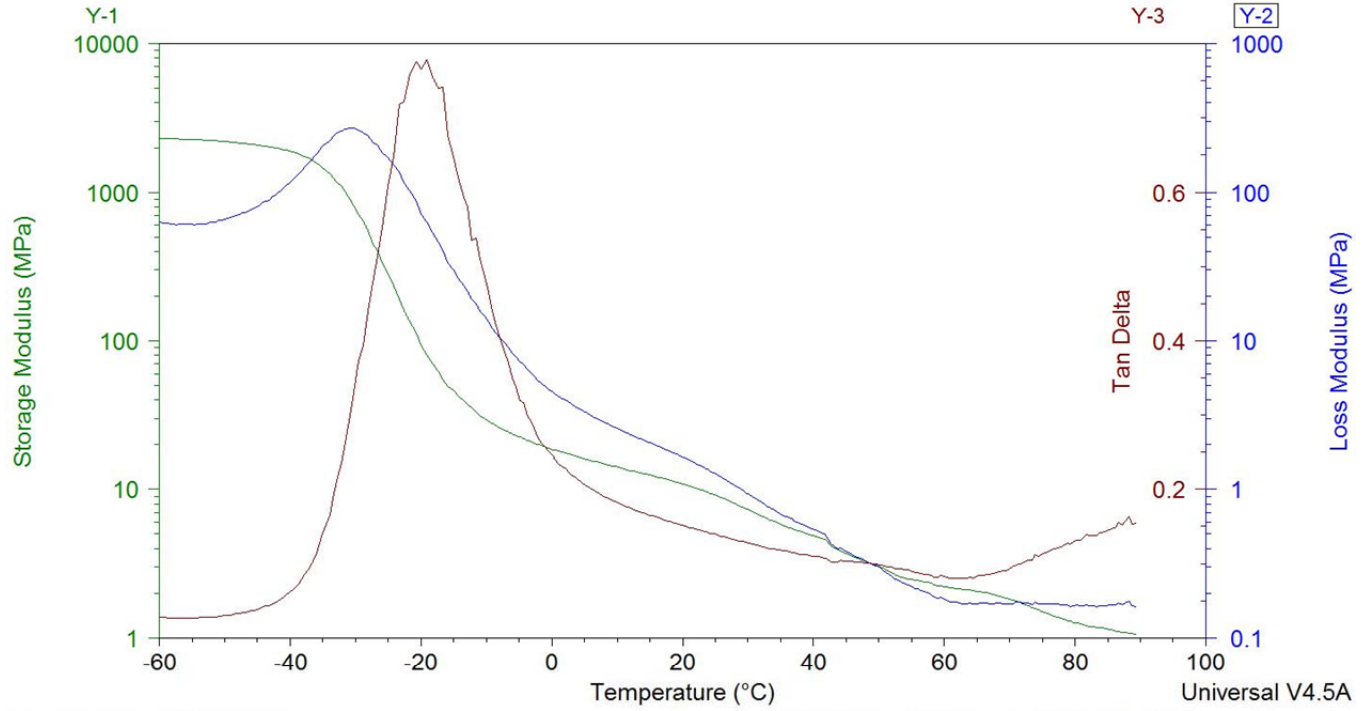
Sample: Piercan Polyurethane 15 mil A

DMA File: Piercan Polyurethane 15 mil A.001



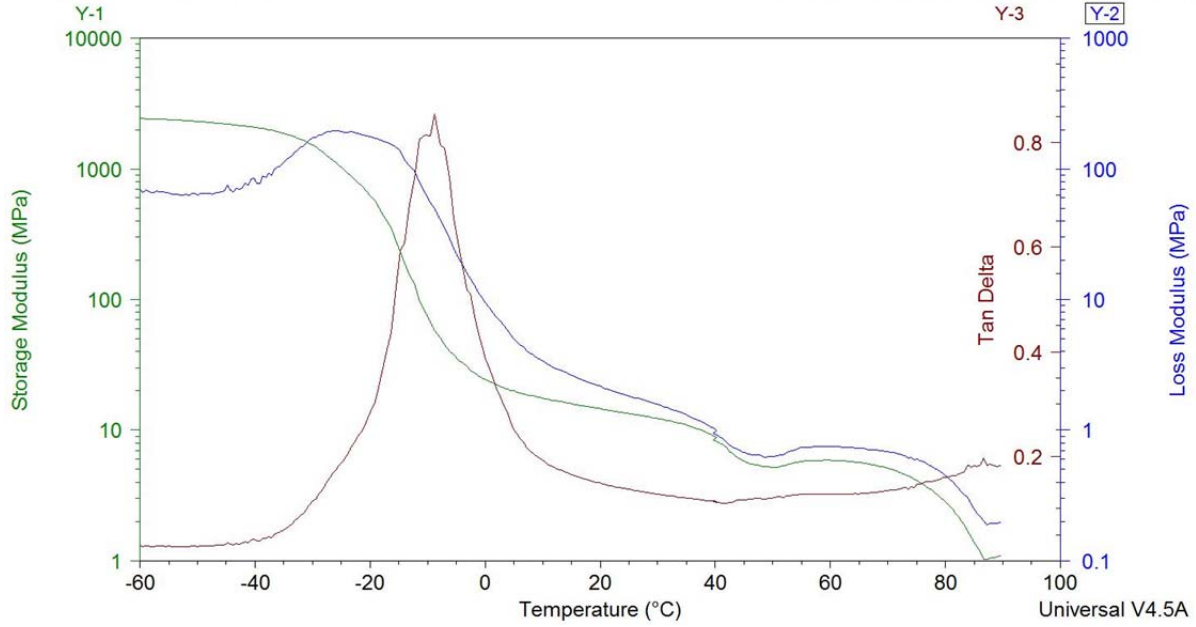
Sample: Piercan Polyurethane 15 mil B

DMA File: Piercan Polyurethane 15 mil B.001



Sample: Piercan UY 20 mil A

DMA File: F:\DMA\Piercan UY 20 mil A.001



Sample: Piercan UY 20 mil B

DMA File: F:\DMA\Piercan UY 20 mil B.001

