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14. ABSTRACT
Polyhedral Oligomeric Silsesquioxanes (POSS) are inorganic/organic cages that are used as high-tech additives to enhance the thermal, mechanical and surface properties of many polymeric systems. A useful analogy is that a POSS is nano-sized particle of silica solubilzed with organic modifiers (RSiO1.5)n; the organic periphery determines how well the POSS can interact with any host polymer, while the siliceous core adds thermoxidative stability. Beginning in 1993 and continuing today, there has been extensive research in understanding how POSS incorporation into various materials affects polymer properties. Herein is reported a synthesis strategy to produce POSS-containing, thermosetting phenylethynyls to yield high temperature materials with extreme thermo-oxidative stability, low moisture uptake and good mechanical properties.

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Phenylethynyl silsesquioxanes: Monomer synthesis, characterization, thermolysis and thermal properties

14 Dec 2016

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Integrity ★ Service ★ Excellence

Outline

- High-temperature/performance Polymers
- Synopsis of earlier work
- Strategy to make an imide-free monomer
- Thermal data comparisons

Acknowledgements:

Air Force Office of Scientific Research,

Air Force Research Laboratory

Presentation Outline

- Introduction to Aromatic Imide Polymers
  - Pros & cons motivating research
  - Research strategy
- Material Synthesis and Characterization
  - Short oligimide macromers
  - BisphenylethynyldimidePOSS
  - BisphenylethynylPOSS,
- Oligomer Processing and Thermal Analyses
  - DSC, TGA-MS, Rheology, Humidity Exposure, TMA
- Moisture Uptake Results
- Conclusions

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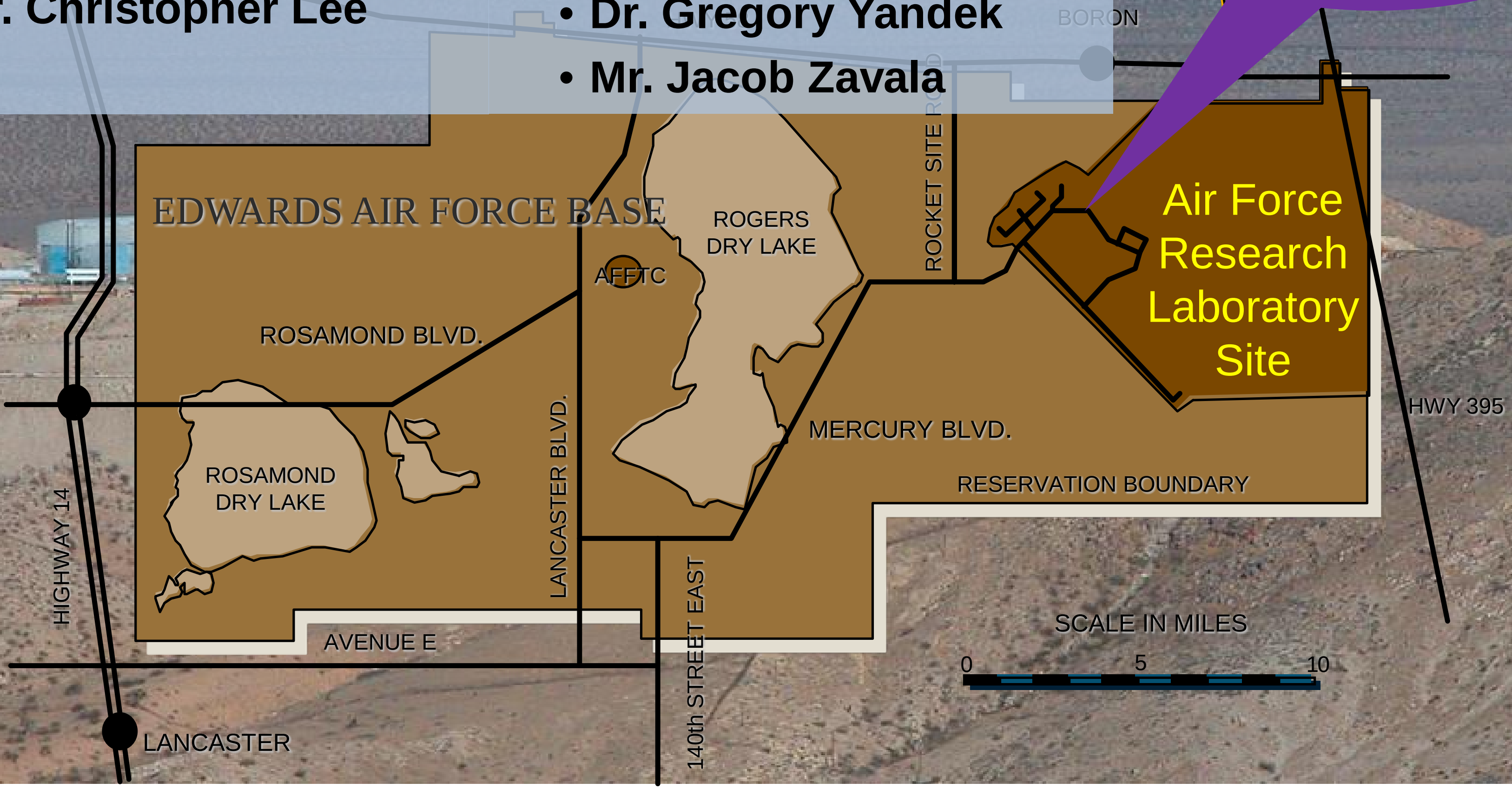


Air Force Research Laboratory Site

Introduction to Polyimides

STRENGTHS

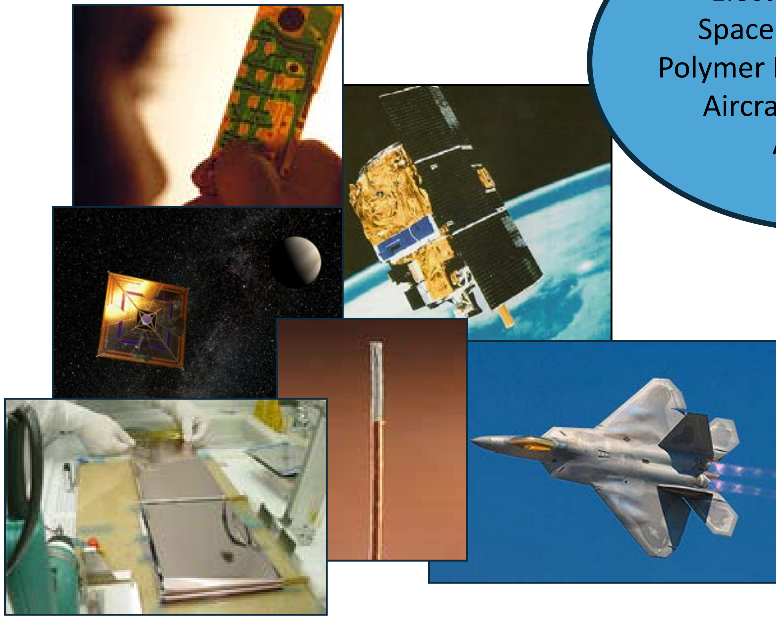
Mechanical Properties
Electrical Properties
Temperature Capabilities
Chemical Resistance
Low Permeability
Tailorability

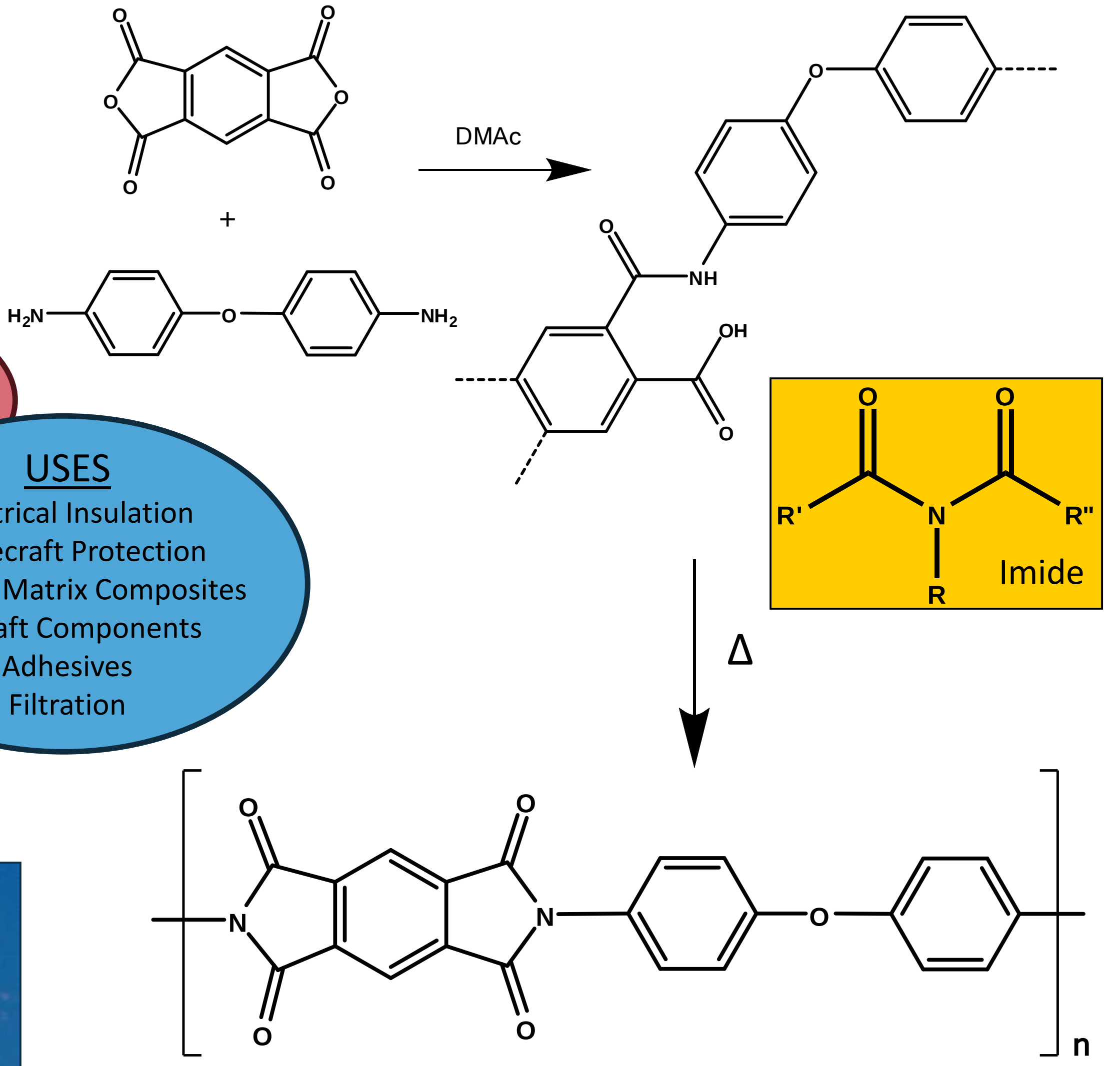
WEAKNESSES

Processability
Moisture Uptake

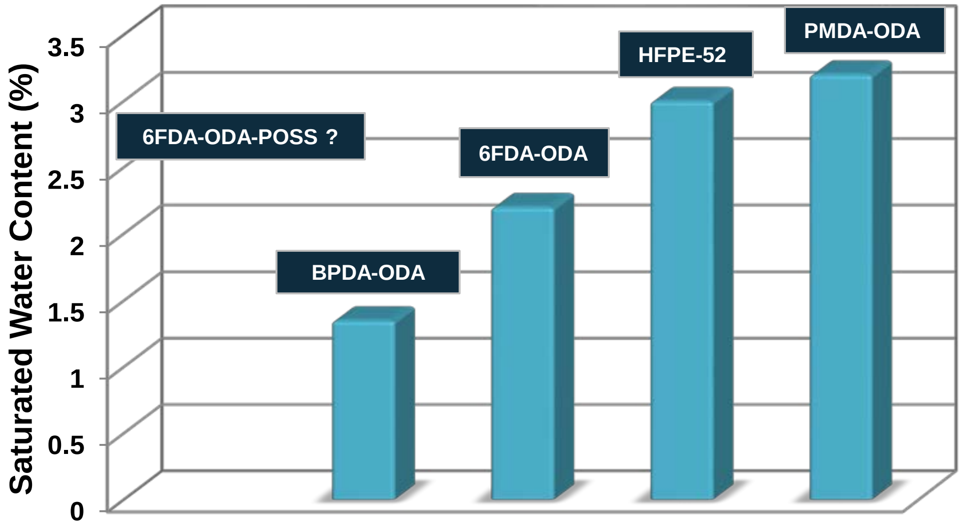
USES

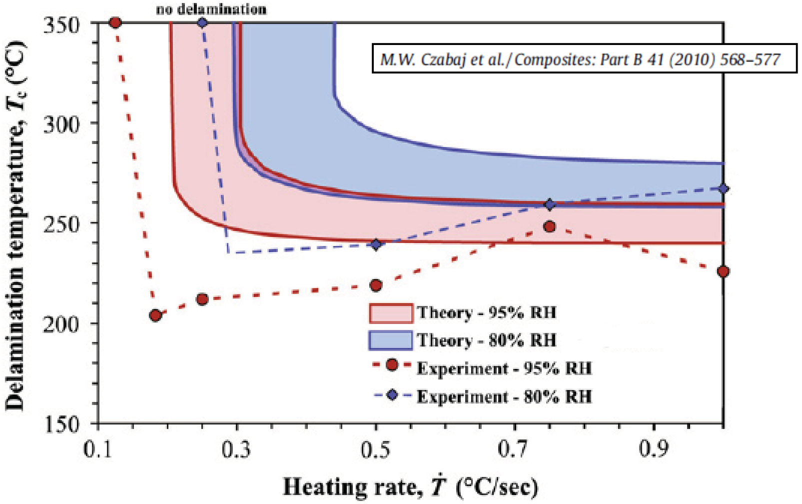
Electrical Insulation
Spacecraft Protection
Polymer Matrix Composites
Aircraft Components
Adhesives
Filtration





Moisture Uptake in Polyimides





Moisture uptake by the resin matrix in PiMCs currently prevents their application to rocket propulsion structures

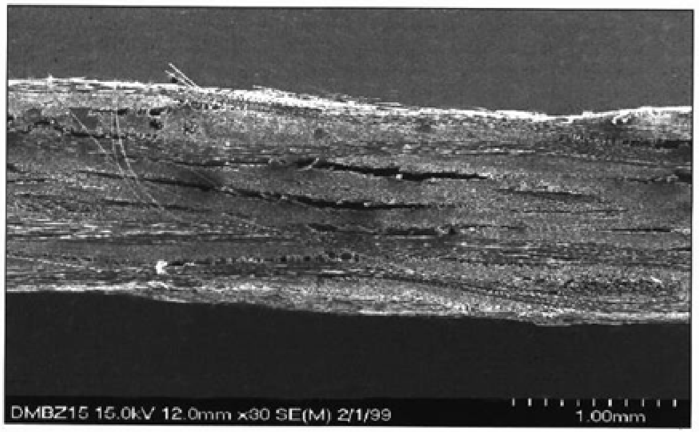
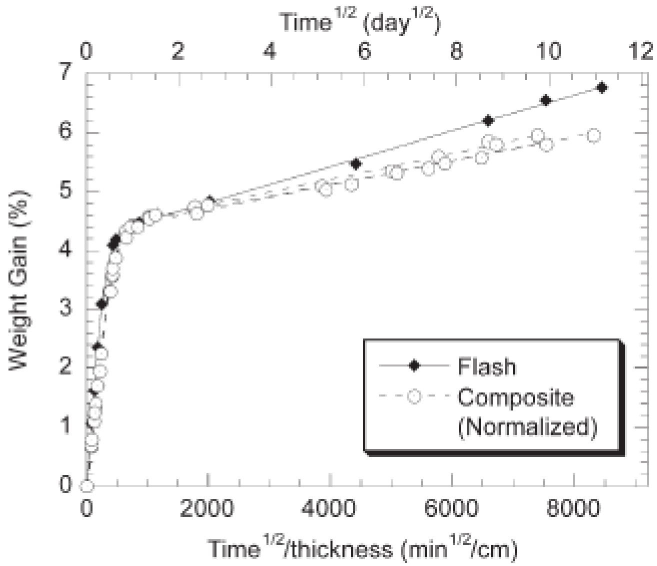
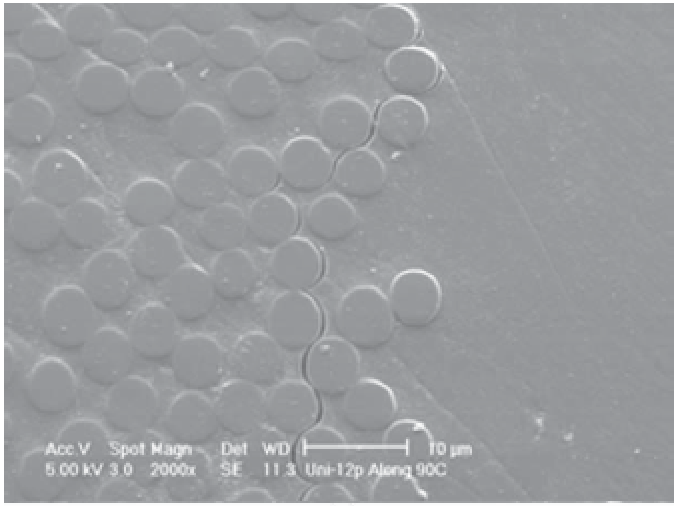


Image of PiMC laminate post-exposed to RH and subsequent rapid heating
Courtesy of NAWCWD

Moisture Uptake Mechanism in OMCs





- **Local segmental polymer motion is responsible for small molecule diffusion (Fick’s)**
- **Once Fick’s plateau is reached, large scale network relaxation accommodates more water**
- **Fiber confines relaxation in OMCs**
- **Stress build-up in matrix causes delamination and cracking**

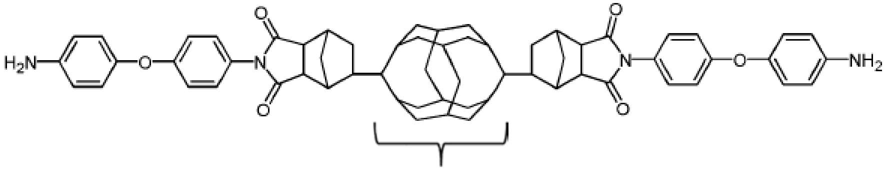
Bao, L.-R.; Yee, A.F. *Composites Science and Technology* **2002**, *62,* 2099–2110

Mitigation Strategies for PiMC Moisture Uptake

	Barrier Materials	Modify Oligomer Chemistry	Blend/IPN
Mechanism	Integration of impermeable layer	Reduce backbone polarity	Free volume occupation
Potential Drawbacks	Greater number of interfaces Increased weight Monitoring Repair	Reduction in thermal and mechanical properties	Mechanical property knockdown

Can the moisture resistance of polyimide networks formed from thermosetting oligomers be improved without sacrificing other key properties?

POSS-Modified Polyimides



$Si_{10}O_{14}Ph_8Me_2$

Table 4. Mechanical Properties of POSS−PIs 8a−e

POSS−PI	tensile strength (MPa)	elongation (%)	initial modulus (GPa)
8b	74.1	6.0	2.15
8b[a]	65.9	3.8	1.96

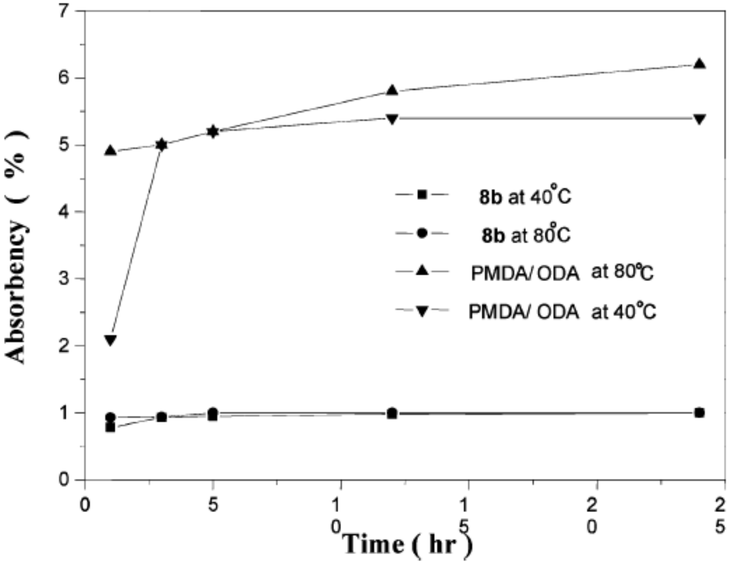
PI	T_g[a] (°C)	T_{d5}[b] (°C)	T_{d10}[c] (°C)	density (g/cm^3)	contact angle (deg)
PMDA/ODA	362	470	530	1.44	54
8b	261	501	538	1.42	86

Table 5. Dielectric Constant of Polyimides

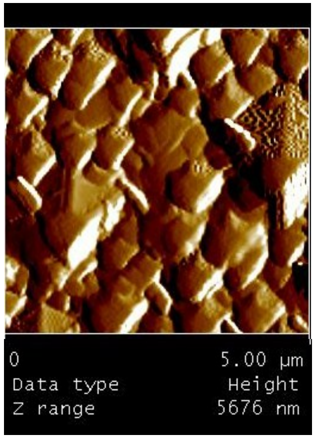
PI	10 kHz	100 kHz	1 MHz
PMDA/ODA	3.46	3.44	3.39
8b	2.69	2.65	2.63

Kakimoto, et al. *Macromolecules* **2007**, 40, 5698 – 5705.

- **Copolymerization of DDSQ and PMDA improves moisture resistance, dielectric properties, and thermal stability**
- **Reduces Tg and mechanical properties**

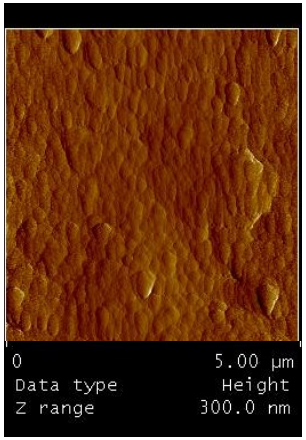


POSS-Modified Polyimides



Kapton

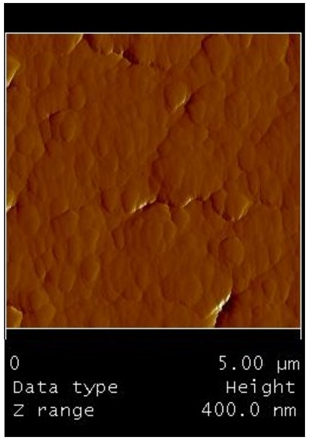
TOTAL EROSION



Kapton-Main Chain POSS

Erosion Depth:

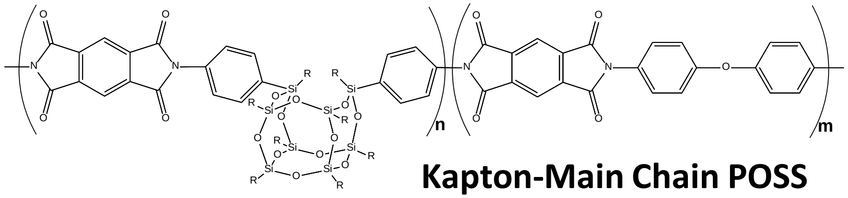
0.885 ± 0.0785



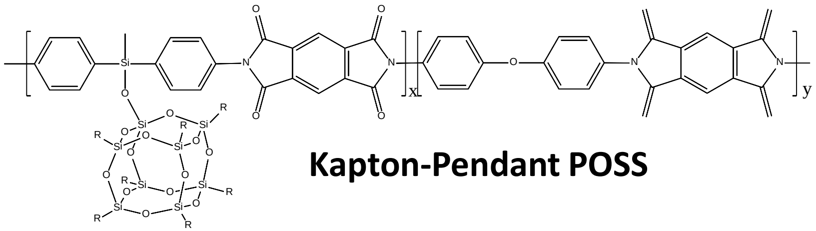
Kapton-Pendant POSS

Erosion Depth:

0.793 ± 0.0669



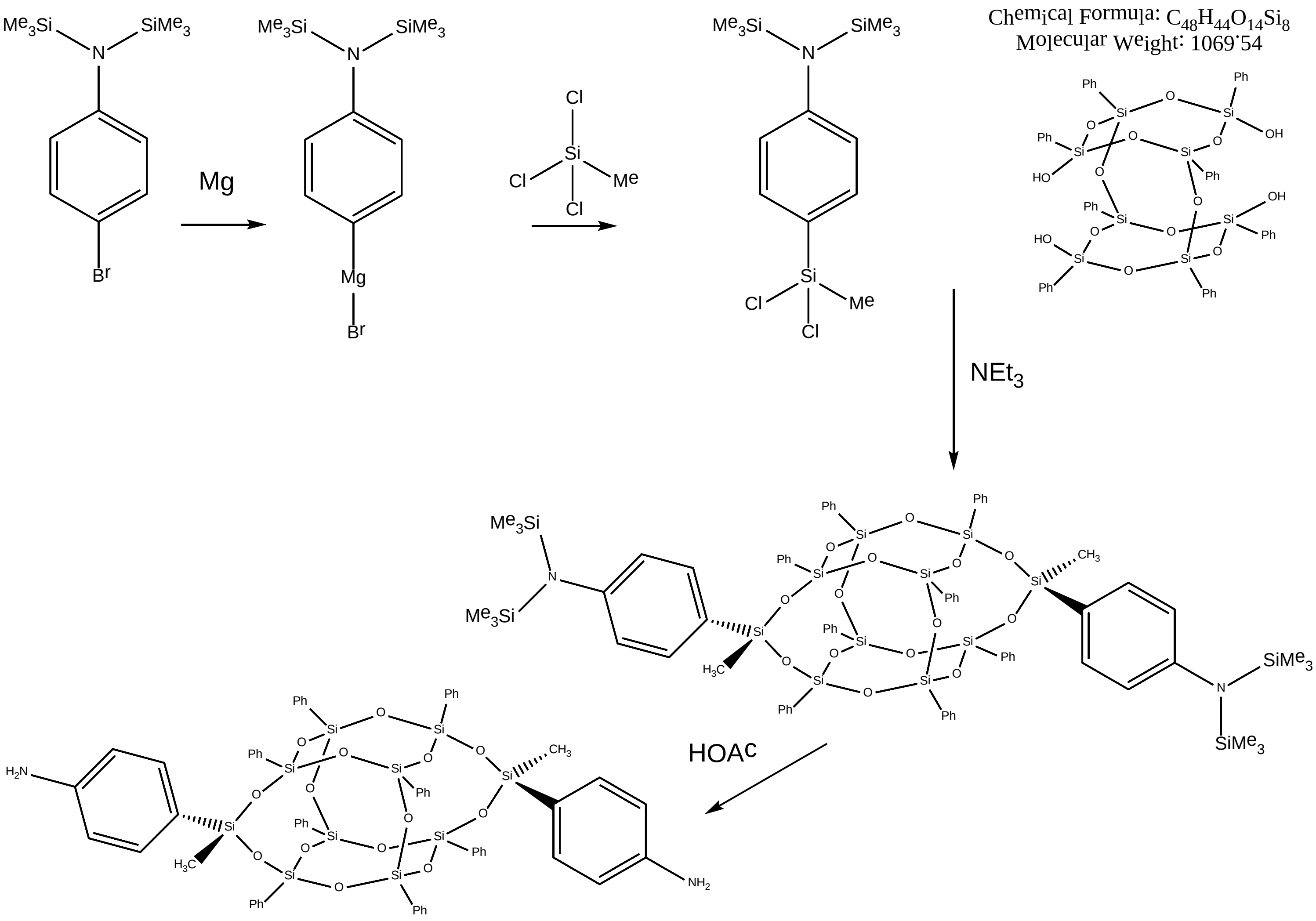
Kapton-Main Chain POSS



Kapton-Pendant POSS

- **POSS modification of polyimides has been shown to drastically reduce erosion induced by atomic oxygen in LEO**
- **Formation of protective layer of silicon dioxide prevents further attack**

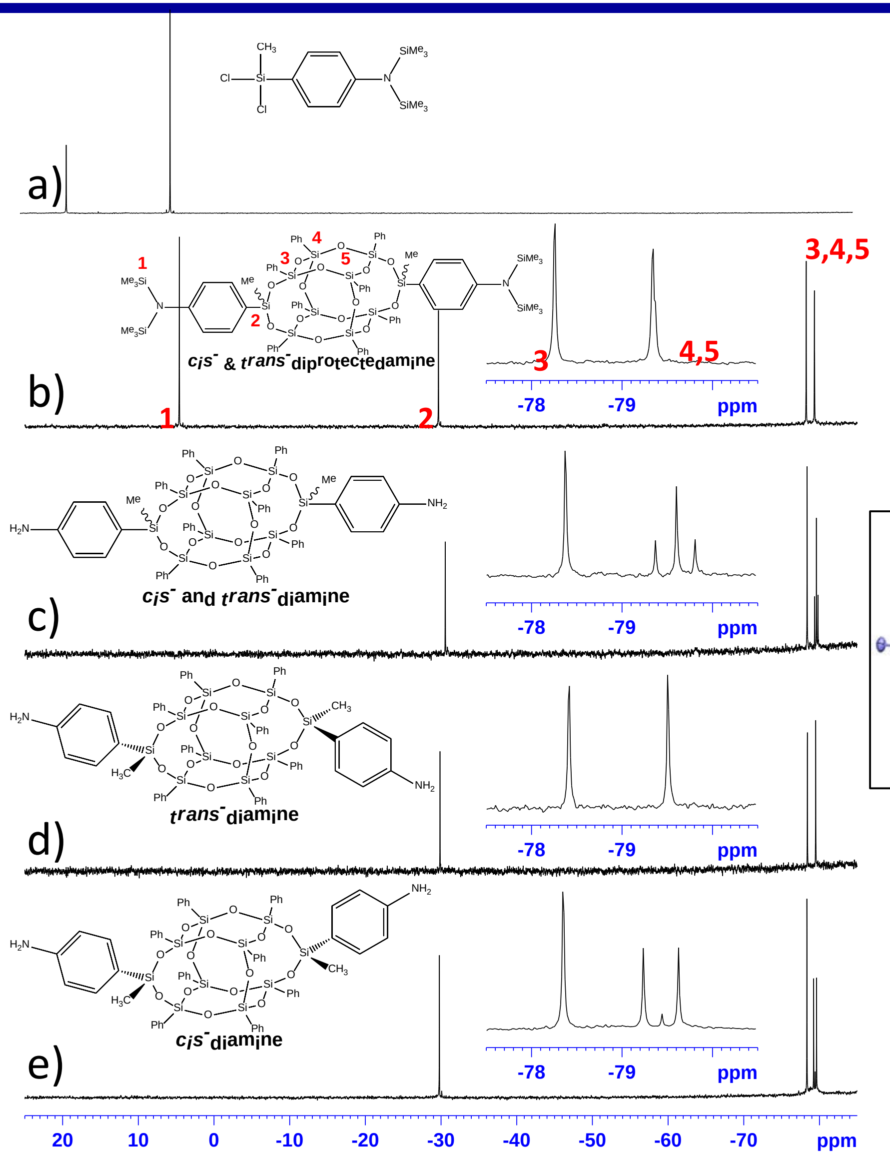
POSS-diamine Synthesis

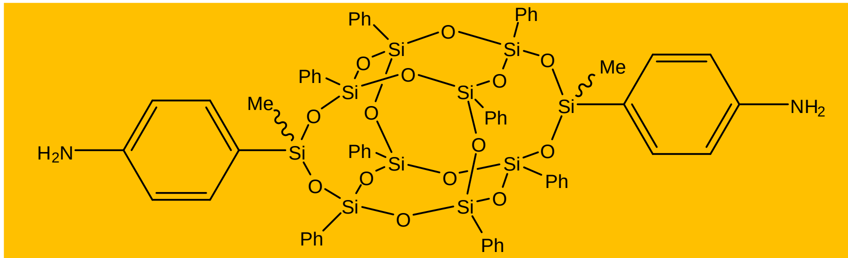


86 % yield from tetraol

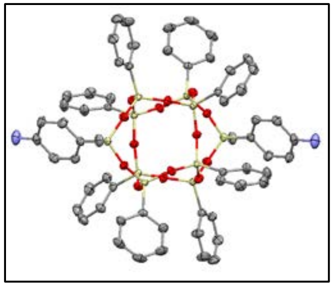
½ pound scale
In a 3 L flask

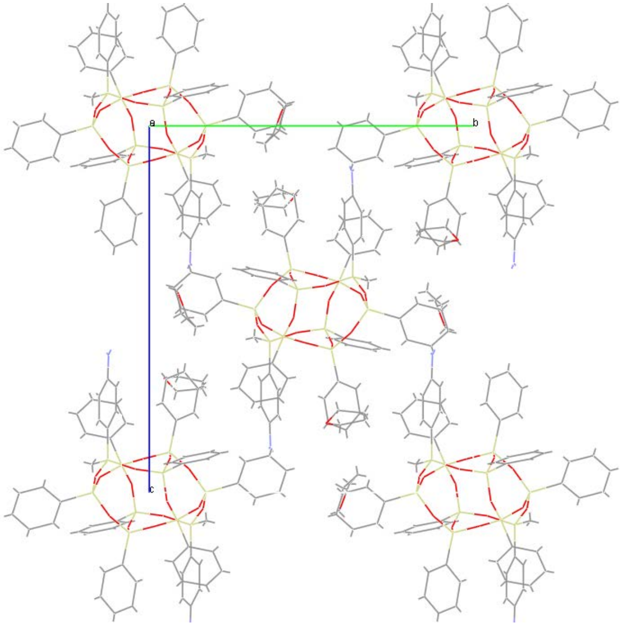
Bis(aniline, methyl)siloxy-Octaphenyl Silsesquioxane





- Well defined inorganic cage
- Peripherally aromatic
- Bi-reactive functionality
- Isomer suite

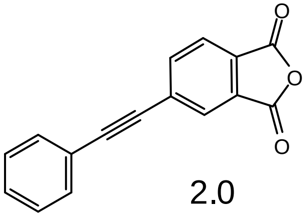




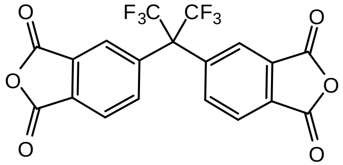
Crystal System - Monoclinic
Space group – P 2(1)/c
Cell Volume = 4034.0 (3)
Cell Dimensions
a = 10.9135 (4)   a = 90.00
b = 18.0703(7)   b = 99.7840 (10)
c = 20.7573 (8)   g = 90.00
R_1 = 0.038

Courtesy of Dr. Sean Ramirez

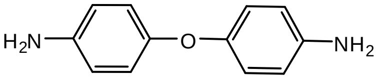
Oligomer Architectures



2.0

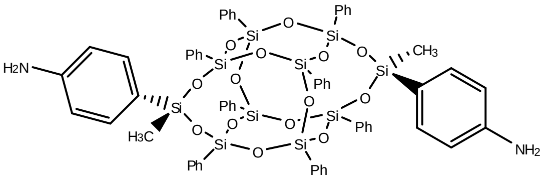


4.0

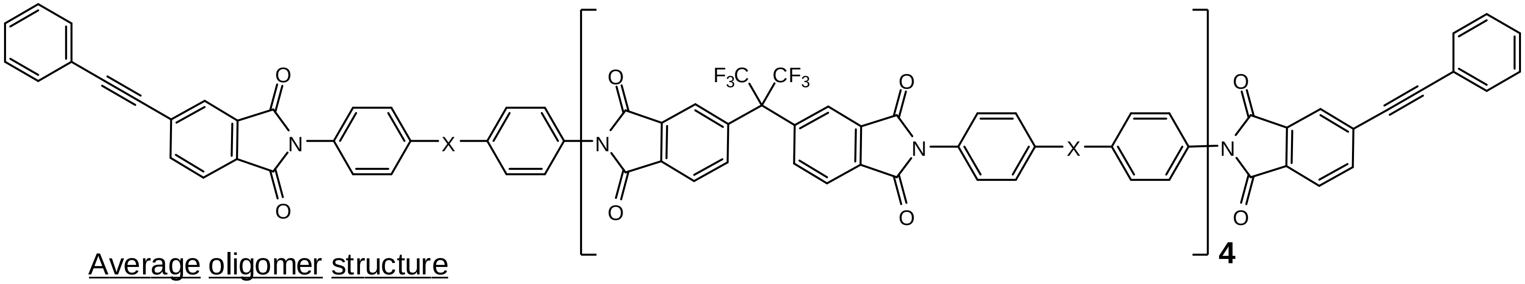


5.0 - 0.0

TetraSilanolPhenyl POSS
100 g/220 USD

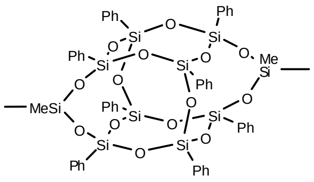


0.0 - 5.0



Average oligomer structure

X = oxygen or



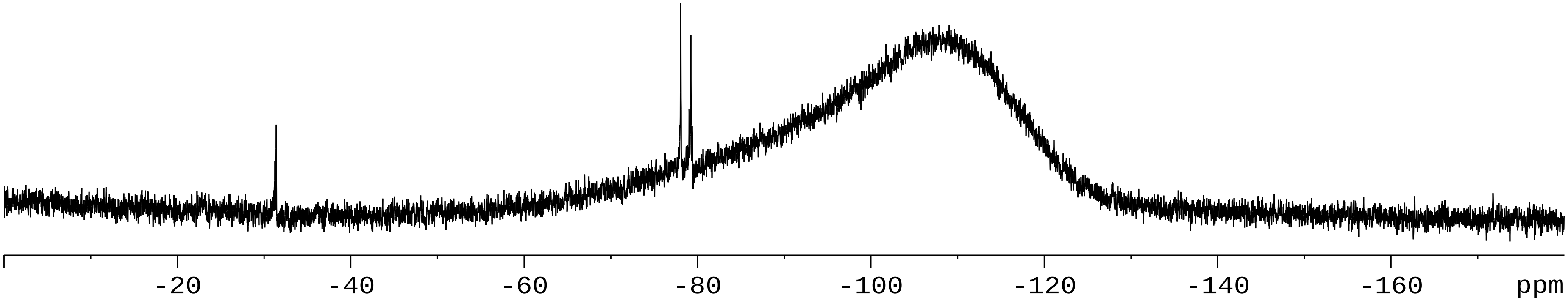
Oligomer ID	Avg Mol Wt (g mol^{-1})
0 POSS	3094
1 POSS	4230
2 POSS	5366
3 POSS	6502
4 POSS	7637
5 POSS	8773

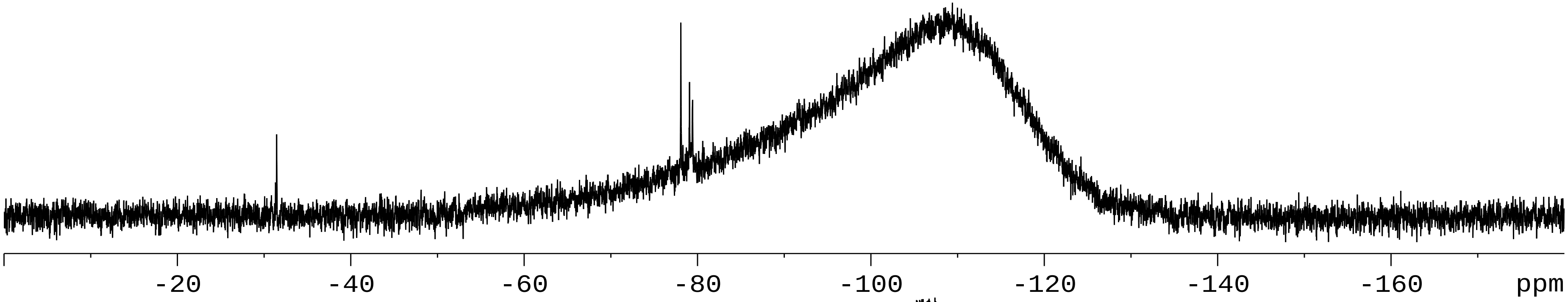
Oligomer Architectures

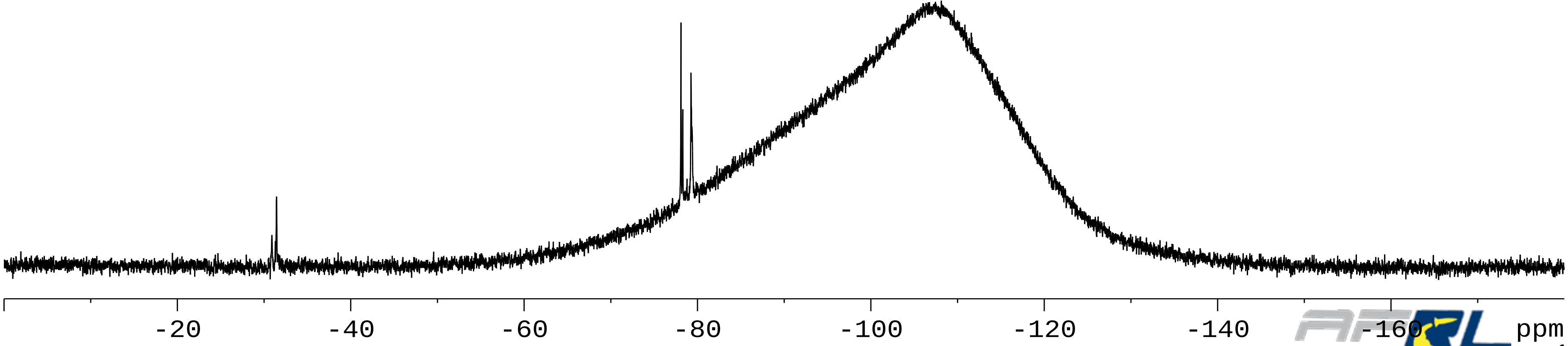
Compound Mol. Wt. Formula	PEPA 248.23 $C_{16}H_8O_3$	6-FDA 444.24 $C_{19}H_6F_6O_6$	ODA 200.24 C12H12N2O	POSS 1335.98 $C_{62}H_{58}N_2O_{14}Si_{10}$
Control	2	4	5	0
1 POSS	2	4	4	1
2 POSS	2	4	3	2
3 POSS	2	4	2	3
4 POSS	2	4	1	4
5 POSS	2	4	0	5

Oligomer	Mol. Wt. (g/mol)	Mmoles in 7.00 g	PE (g)	6-FDA (g)	ODA (g)	POSS (g)	Wt % $Si_{10}O_{14}$
$C_{168}H_{80}F_{24}N_{10}O_{25}$	3094.45	2.2624	1.1228	4.0194	2.2645	0.000	0.0%
$C_{218}H_{126}F_{24}N_{10}O_{38}Si_{10}$	4230.19	1.6548	0.8218	2.9407	1.3251	2.2106	8.33%
$C_{268}H_{172}F_{24}N_{10}O_{51}Si_{20}$	5365.94	1.3048	0.6475	2.3184	0.784	3.4853	13.16%
$C_{318}H_{218}F_{24}N_{10}O_{64}Si_{30}$	6501.69	1.0766	0.5348	1.9131	0.262	4.3148	16.31%
$C_{368}H_{264}F_{24}N_{10}O_{77}Si_{40}$	7637.44	0.9163	0.4550	2.3289	0.1834	4.8979	18.48%
$C_{418}H_{310}F_{24}N_{10}O_{90}Si_{50}$	8773.18	0.798	0.3962	1.4175	0.000	5.3298	20.16%

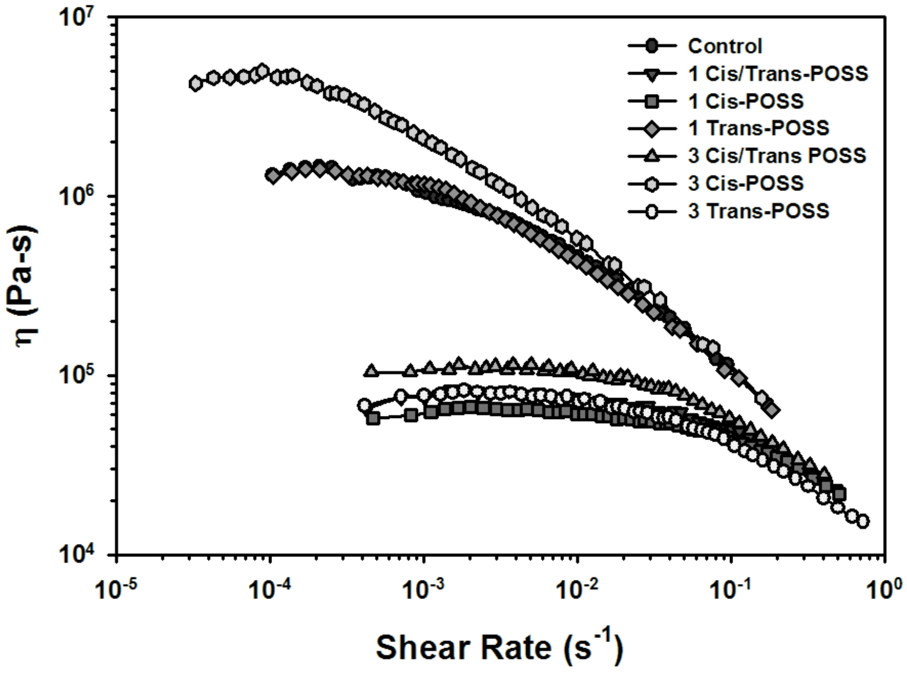
^{29}Si NMR of 1POSS Cis&Trans, 1POSS Cis, and 1POSS Trans Oligoimides in $CDCl_3$







Steady Shear Rheology

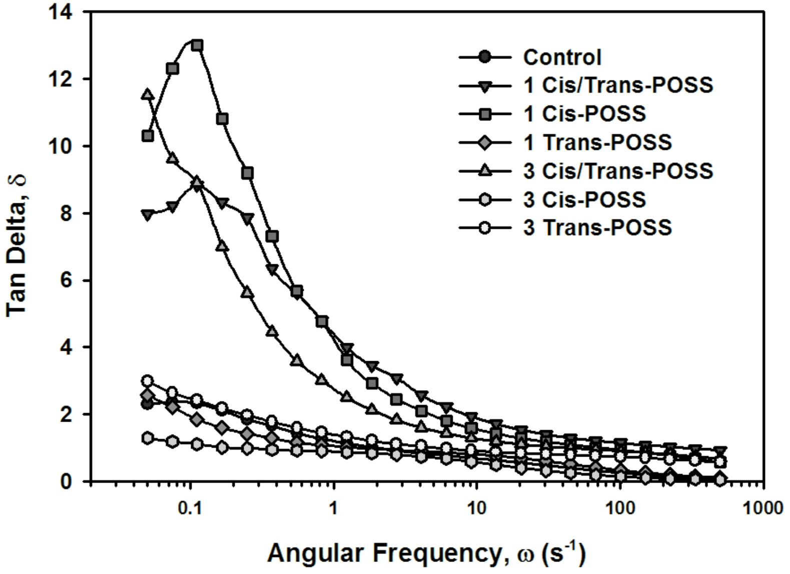


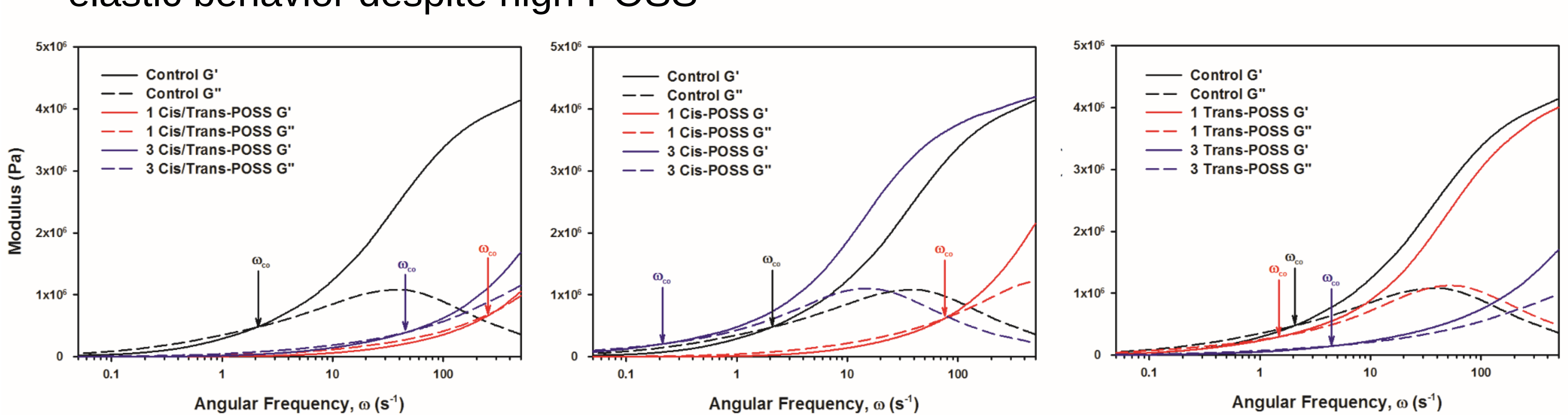
Oligomer	M_n (theor.)	M_n (obs.)
Control	3095	3700
1 cis/trans-POSS	4230	4006
1 cis-POSS	4230	4494
1 trans-POSS	4230	4934
3 cis/trans-POSS	6502	5870
3 cis-POSS	6502	6244
3 trans-POSS	6502	7100

- **Experiment performed at 250 °C and 0.7 mm gap**
- **For this set of oligomers, POSS isomer type exerts greater effect on steady shear viscosity than MW and PDI**
- **Mixture of cis and trans isomers in backbone is most effective at reducing viscosity**
- **3 cis-POSS exhibits highest viscosity and shortest linear response, perhaps due to oligomer chain conformation**

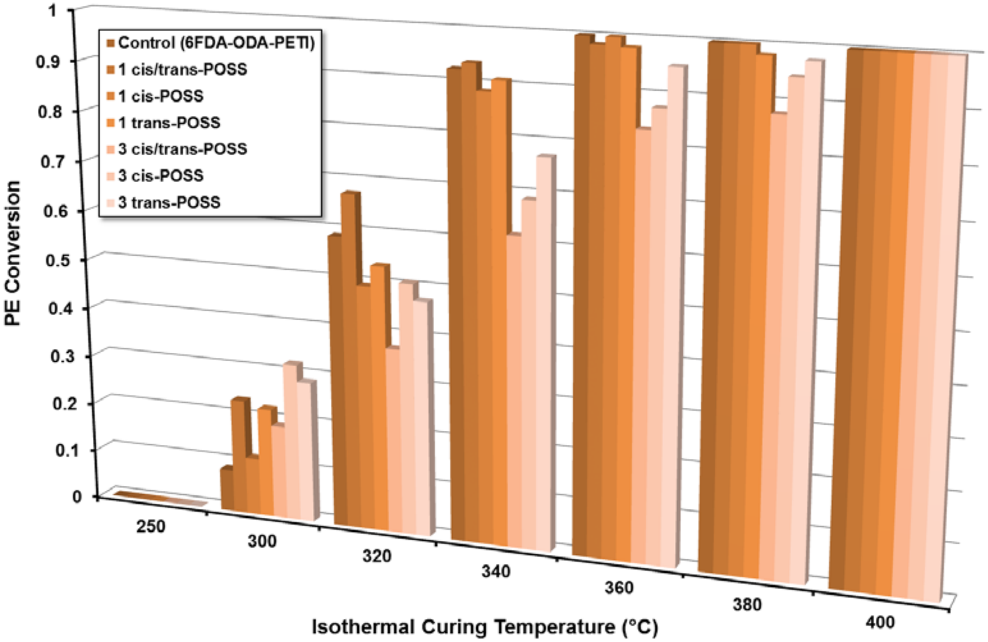
Complex Rheology

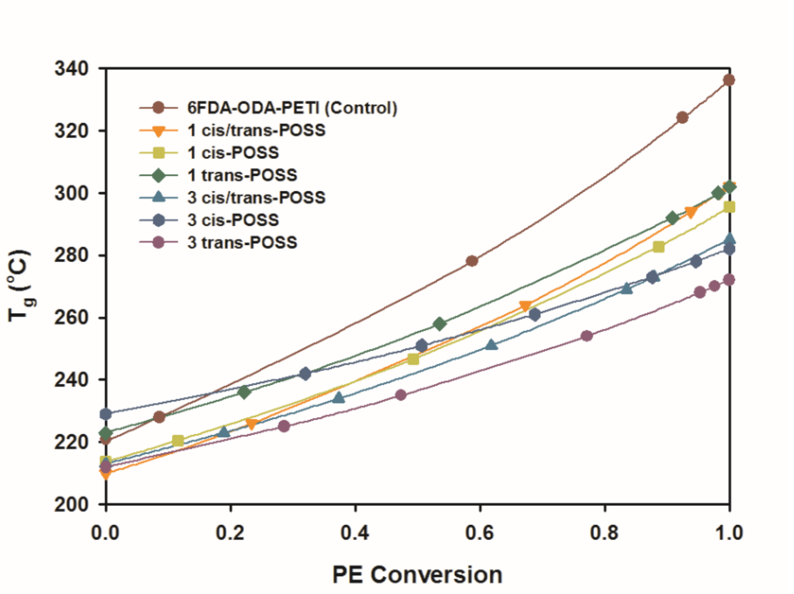
- Peaks in tan delta occur at low frequency for cis-POSS containing oligomers (except 3 cis-POSS)
- Control and 1 trans-POSS are very similar rheologically
- PDI effects are negligible
- Copolymerization of POSS generally shifts crossover point to higher frequency
- 3 cis-POSS is unusual due to its highly elastic behavior despite high POSS





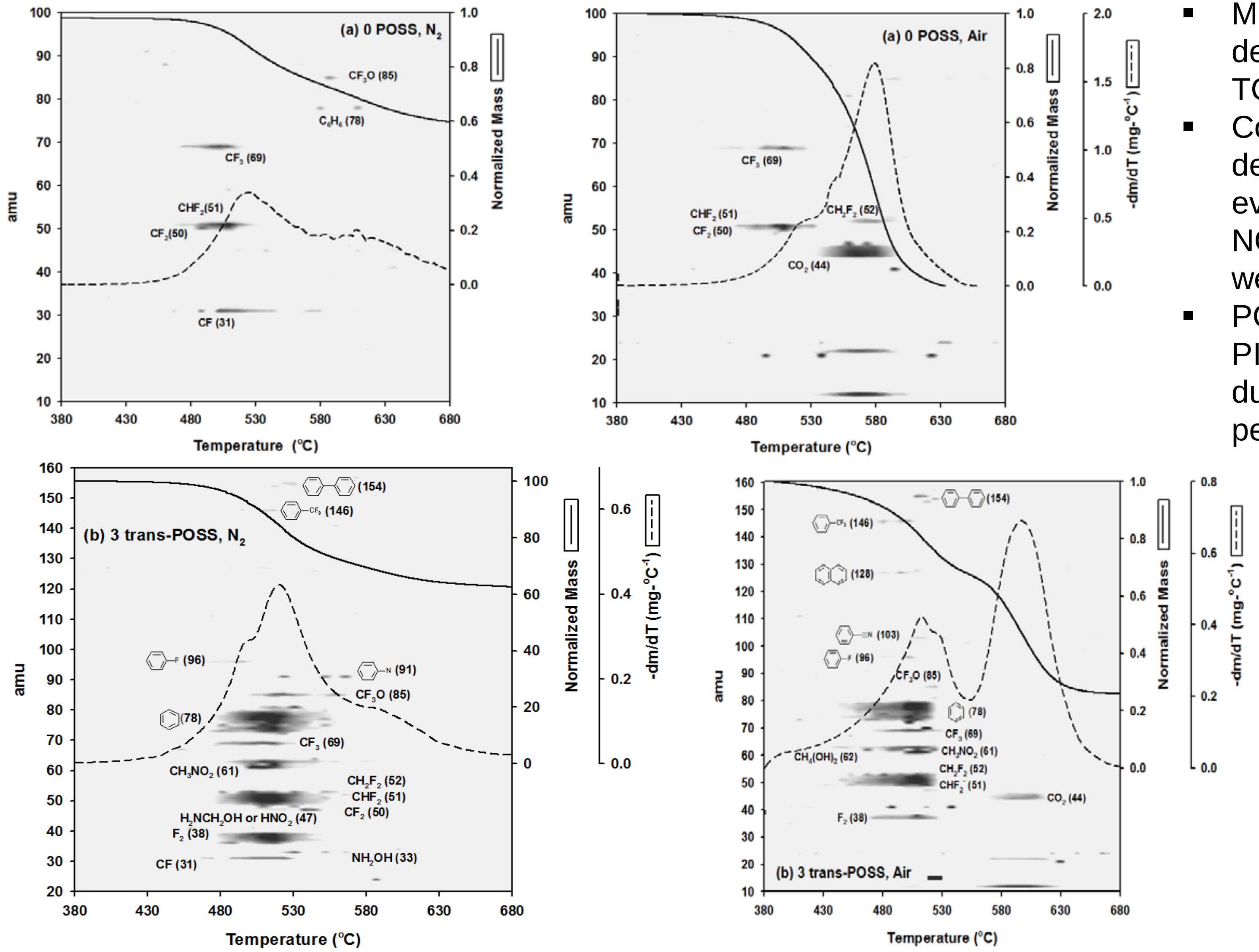
Cure Reaction Overview





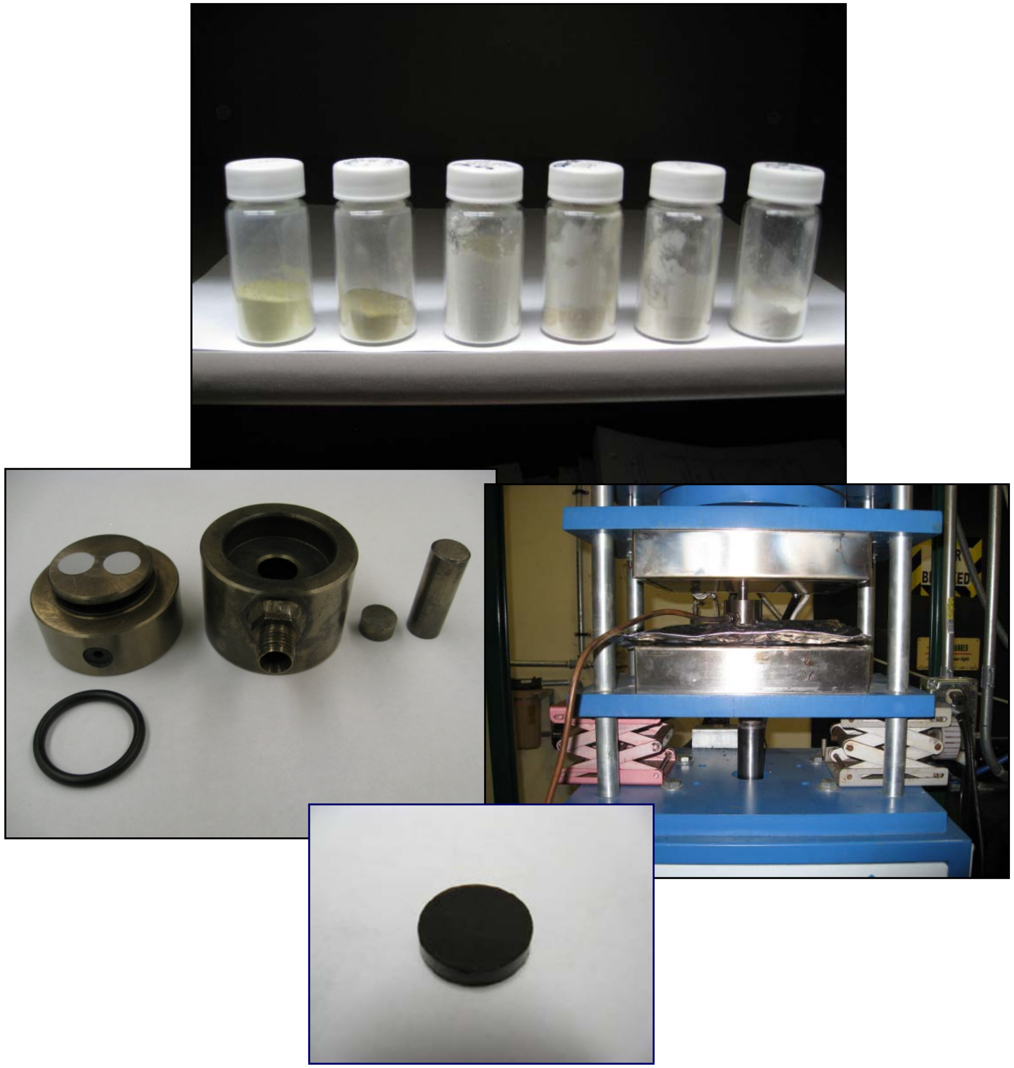
- DSC performed by cycling holding material at each isotherm for 60 minutes and measuring post-hold glass transition temperature
- Phenyl ethynyl cure reaction does not commence until ~300 °C
- Pre- and post-cure T_gs generally reduced by POSS copolymerization
- At lower cure temperatures, POSS accelerates cure reaction but more thermal energy is required for increased PE conversion as temperature is raised

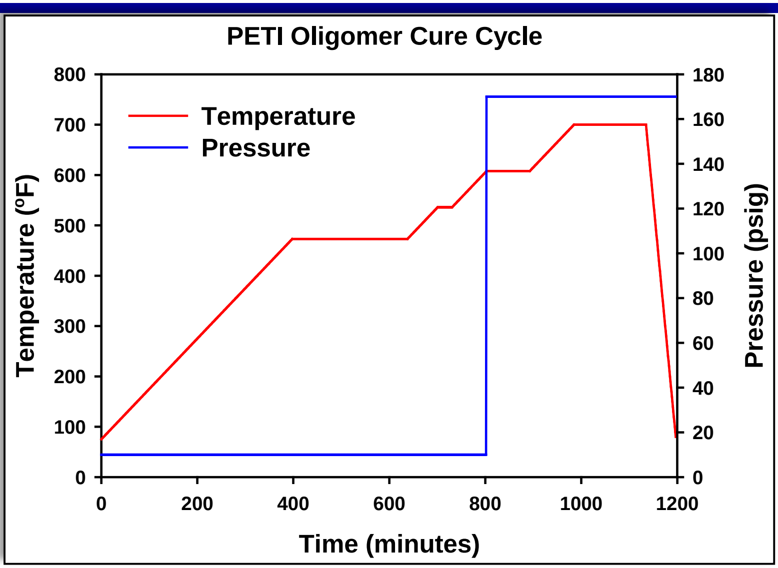
Thermal Decomposition



- Mass loss species determined through TGA-MS
- Control decomposition evolves CO_2, CO, NO, and H_2O as well understood
- POSS-containing PIs evolve benzene due to POSS periphery scission

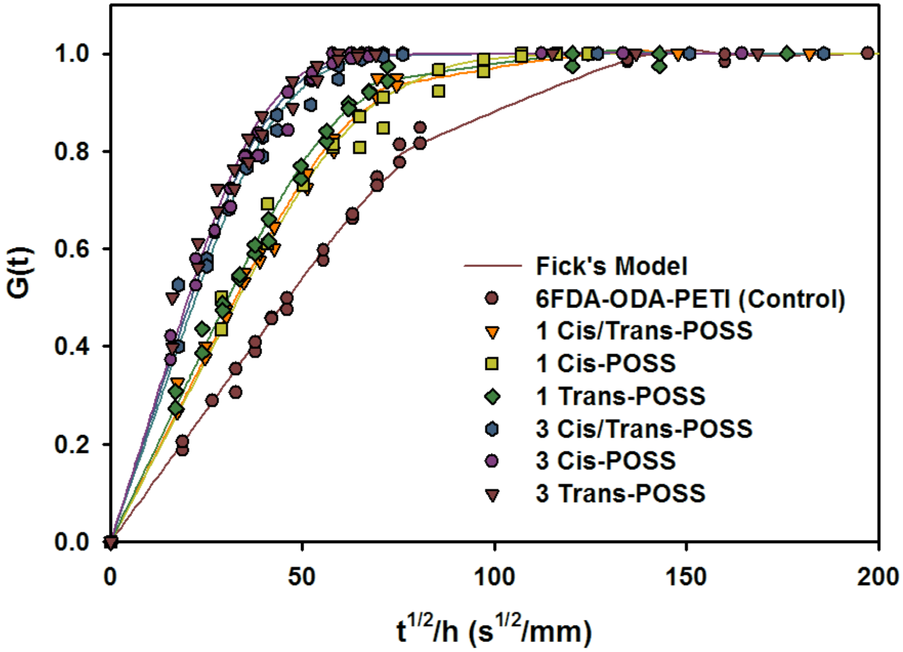
Neat Resin Processing



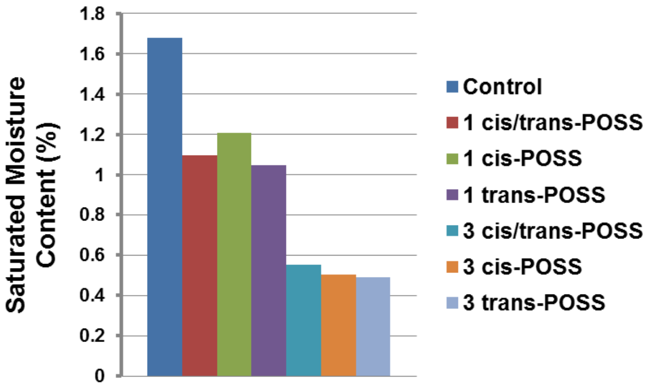


- Oligomer cure schedule typical of PETI composites
- Compression mold with vacuum used to consolidate cured disks of each PI
- Low pressure required in early stages of cycle controlled with jack stands due to lack of fidelity in molding machine

Moisture Diffusion



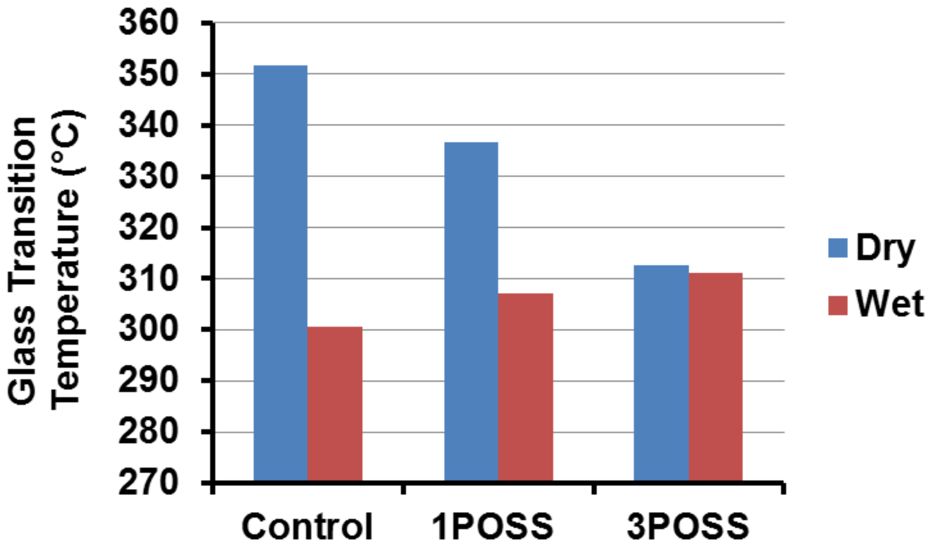
Cured Oligomer	Density (g/cc)	Thickness, h (mm)	D (mm²/s × 10^5)
6FDA-ODA-PETI	1.360	2.057	2.30
1 Cis/Trans-POSS	1.343	2.229	4.60
1 Cis-POSS	1.401	1.334	4.40
1 Trans-POSS	1.342	2.303	5.20
3 Cis/Trans-POSS	1.314	2.183	10.00
3 Cis-POSS	1.309	2.467	10.00
3 Trans-POSS	1.310	2.407	12.00

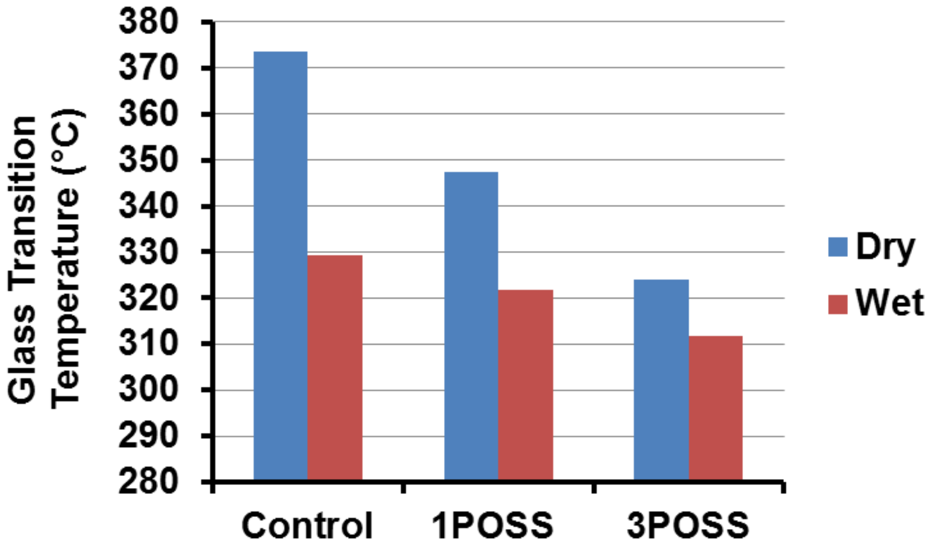


- In general, POSS reduces density
- Moisture diffusion experiments conducted in humidity chamber at 80 °C and 80% RH
- 1-D Fick's diffusion equation solved to determine diffusivity coefficients
- POSS increases diffusivity rate but lowers overall saturated water content

Effects of Water Uptake on Viscoelastic Properties

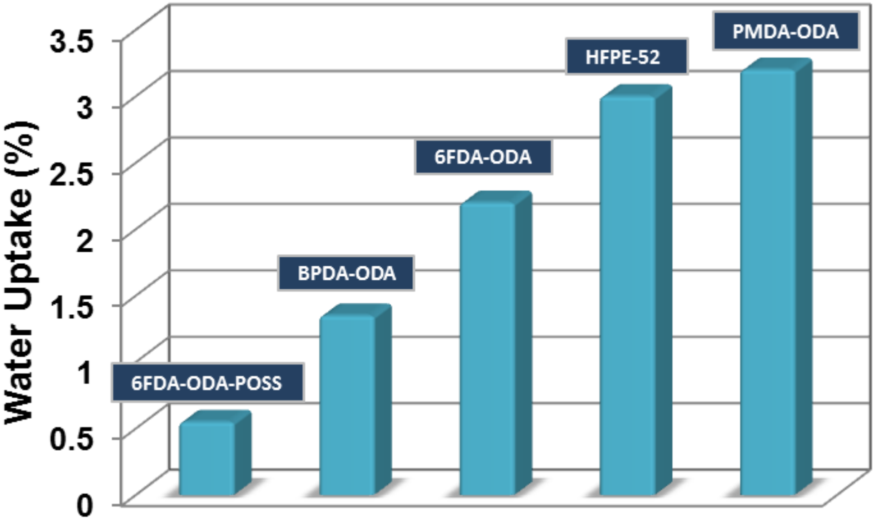
- Dynamic TMA experiments performed at high heating rate (50 °C/min) to ascertain the effects of moisture ingress on glass transition temperature
- Glass transition temperature measured by two methods: onset of storage modulus drop (G´) and peak in tan delta
- By both methods, the delta between dry and wet glass transition temperatures diminishes with increasing POSS content
- Storage modulus method more accurate since event occurs earlier giving water less of a chance to escape
- *Rapid water loss event witnessed for control, but not POSS-containing polyimides*





Conclusions

- POSS copolymerization generally improves processing viscosity
- POSS both increases and decreases phenyl ethynyl reaction rate as a function of cure temperature
- POSS improves thermal stability by TGA in both inert and oxidizing atmospheres, more so in the latter due to formation of protective SiO_2



- POSS decreases cured glass transition temperature in the dry state
- Although hybridization increases water diffusivity, it greatly reduces saturated moisture content
- POSS improves wet thermomechanical properties and could prevent shock associated with water release during rapid heating

Pinson, D. et al. “Thermosetting Poly(imide silisesquioxane)s Featuring Reduced Moisture Affinity and Improved Processability.” *Macromolecules.* 2013, 46: 7363-77.

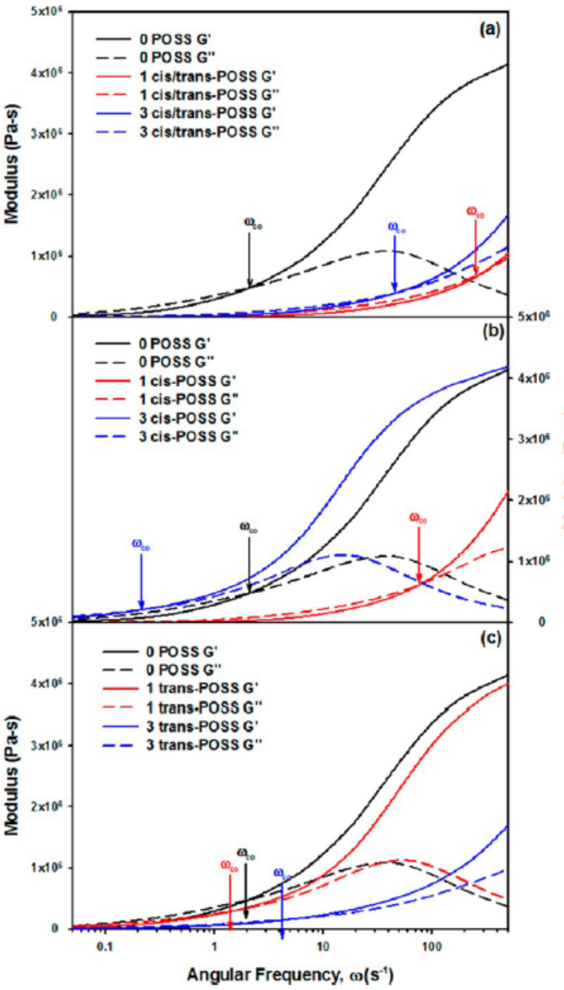


Figure 6. Storage and loss moduli measured from variable frequency oscillatory rheometry experiments at 250 °C for (a) cis/trans-, (b) cis-, and (c) trans-oligomers, in comparison with those of 0 POSS. The crossover frequencies are indicated by ω_{co}.

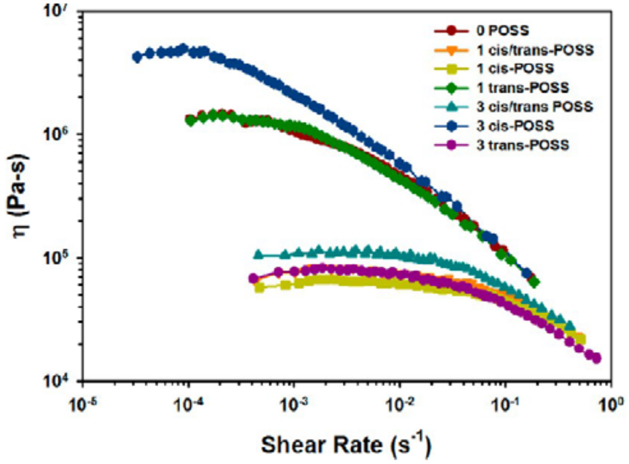
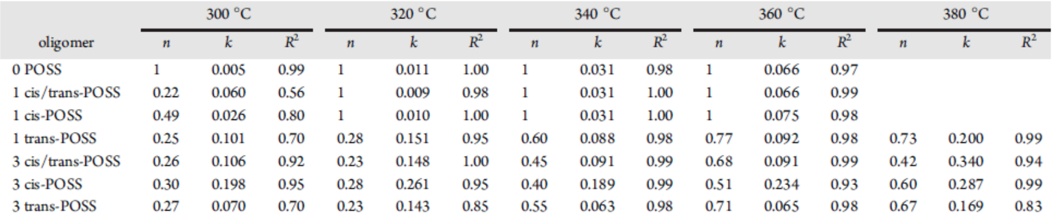


Figure 5. Steady shear viscosity dependence on shear rate for the uncured oligomides at 250 °C.

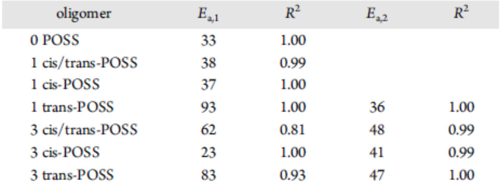
- 1-trans and 3-cis have significantly higher viscosities
- 1-cis has mugh higher crossover frequency while 3-cis has less
- Cis/Trans blends exhibit higher crossover frequencies
- Trans isomer has little effect on crossover frequency

Table 4. Reaction Exponents and Rate Constants with Associated Coefficients of Determination (R^2) Resulting from Fitting Isothermal Conversion Data to First-Order Reaction Rate and Avrami Conversion Models (k in min^{-n})



	300 °C			320 °C			340 °C			360 °C			380 °C		
oligomer	n	k	R^2	n	k	R^2	n	k	R^2	n	k	R^2	n	k	R^2
0 POSS	1	0.005	0.99	1	0.011	1.00	1	0.031	0.98	1	0.066	0.97			
1 cis/trans-POSS	0.22	0.060	0.56	1	0.009	0.98	1	0.031	1.00	1	0.066	0.99			
1 cis-POSS	0.49	0.026	0.80	1	0.010	1.00	1	0.031	1.00	1	0.075	0.98			
1 trans-POSS	0.25	0.101	0.70	0.28	0.151	0.95	0.60	0.088	0.98	0.77	0.092	0.98	0.73	0.200	0.99
3 cis/trans-POSS	0.26	0.106	0.92	0.23	0.148	1.00	0.45	0.091	0.99	0.68	0.091	0.99	0.42	0.340	0.94
3 cis-POSS	0.30	0.198	0.95	0.28	0.261	0.95	0.40	0.189	0.99	0.51	0.234	0.93	0.60	0.287	0.99
3 trans-POSS	0.27	0.070	0.70	0.23	0.143	0.85	0.55	0.063	0.98	0.71	0.065	0.98	0.67	0.169	0.83

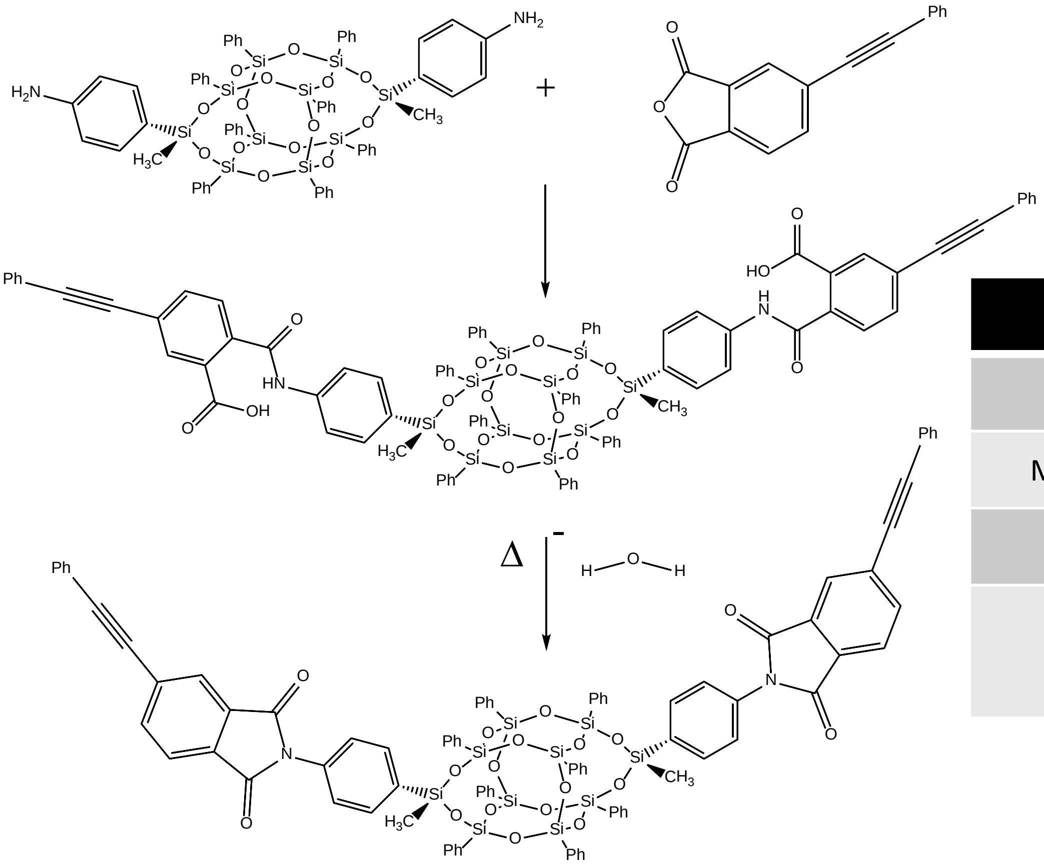
Table 5. Calculated Arrhenius Parameters Pertaining to the Thermal Cure of the Oligoimides Synthesized in This Study



oligomer	$E_{a,1}$	R^2	$E_{a,2}$	R^2
0 POSS	33	1.00		
1 cis/trans-POSS	38	0.99		
1 cis-POSS	37	1.00		
1 trans-POSS	93	1.00	36	1.00
3 cis/trans-POSS	62	0.81	48	0.99
3 cis-POSS	23	1.00	41	0.99
3 trans-POSS	83	0.93	47	1.00

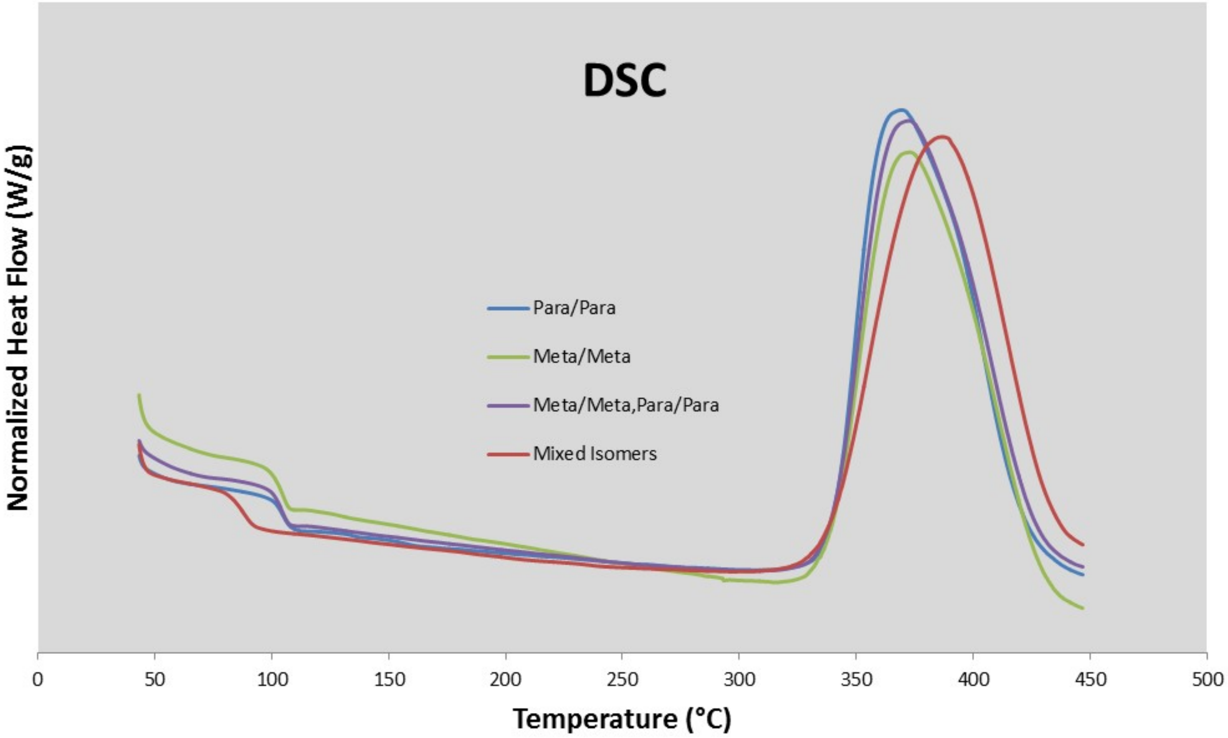
- 3-cis shows significantly higher rate constants and lower activation energy
- 1-trans shows higher activation energy

BisPEPI-POSS Synthesis



	Para/Para	Meta/Para	Meta/Meta
Para/Para	100%	0%	0%
Mixed Isomers	25%	50%	25%
Meta/Meta	0%	0%	100%
Meta/Meta, Para/Para	50%	0	50%

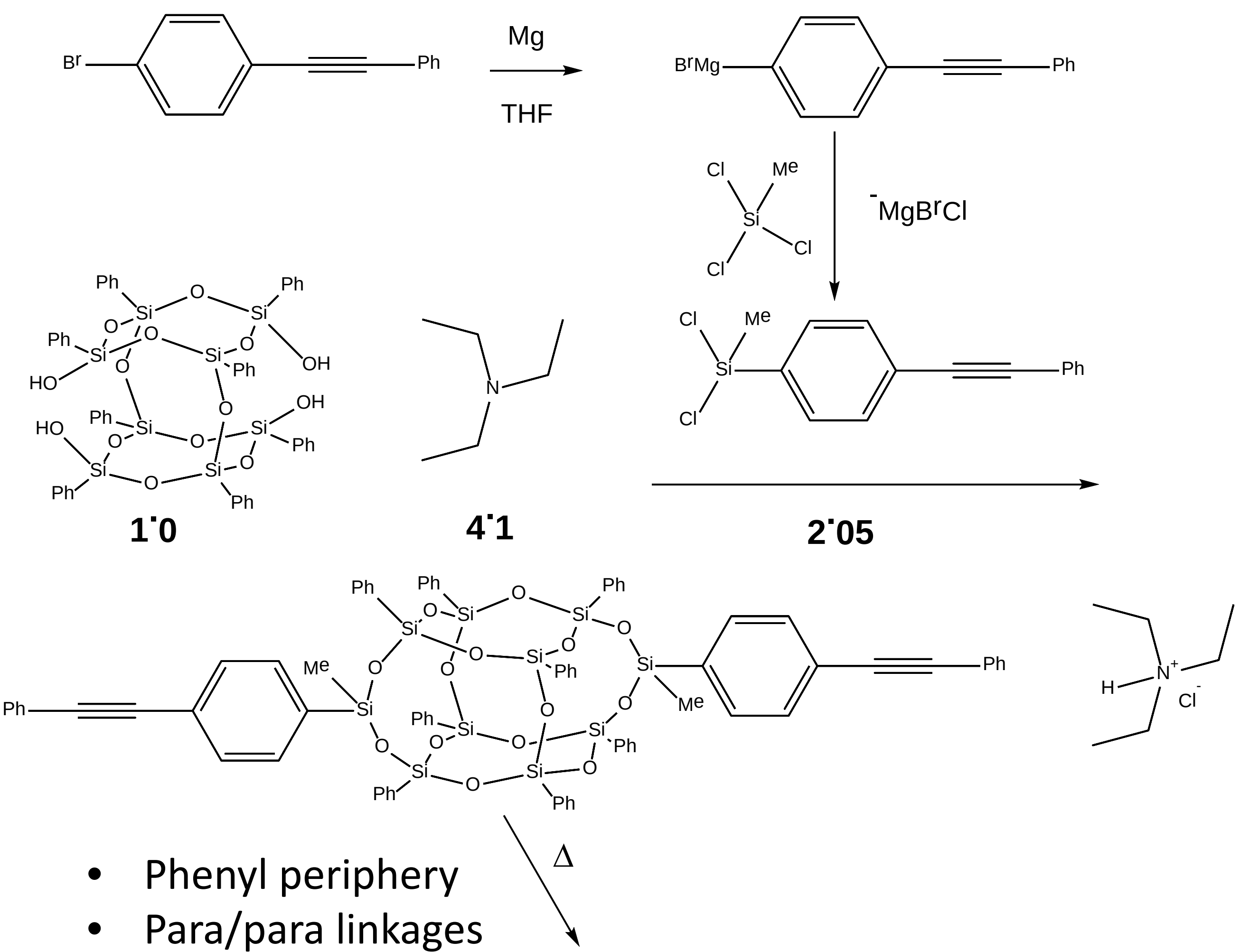
BisPEPI-POSS DSC



Sample	Onset Cure Temp (°C)	Peak Cure Temp (°C)	Cure Energy (J/g)
Para/Para	340.21	369.51	162.2
Meta/Meta	338.75	373.25	160.9
Meta/Meta,Para/Para	339.51	372.28	160.9
Mixed Isomers	338.83	386.20	160.4

BisPE-POSS Synthesis

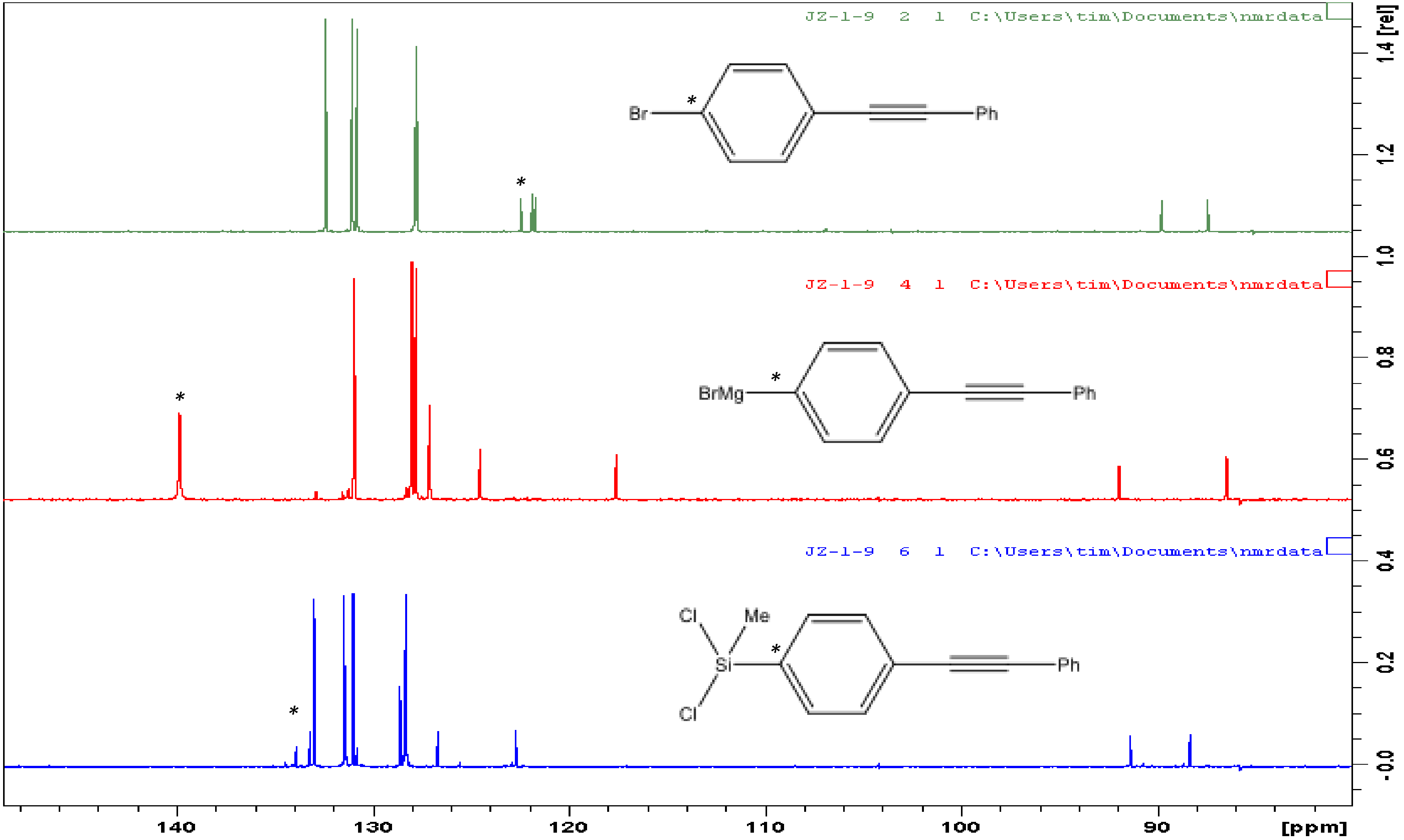
Bis-phenylethynyl-POSS



Thermally robust crosslinked material

- Phenyl periphery
- Para/para linkages

C13 NMR Spectra of starting material, Grignard reagent, and dichlorosilane



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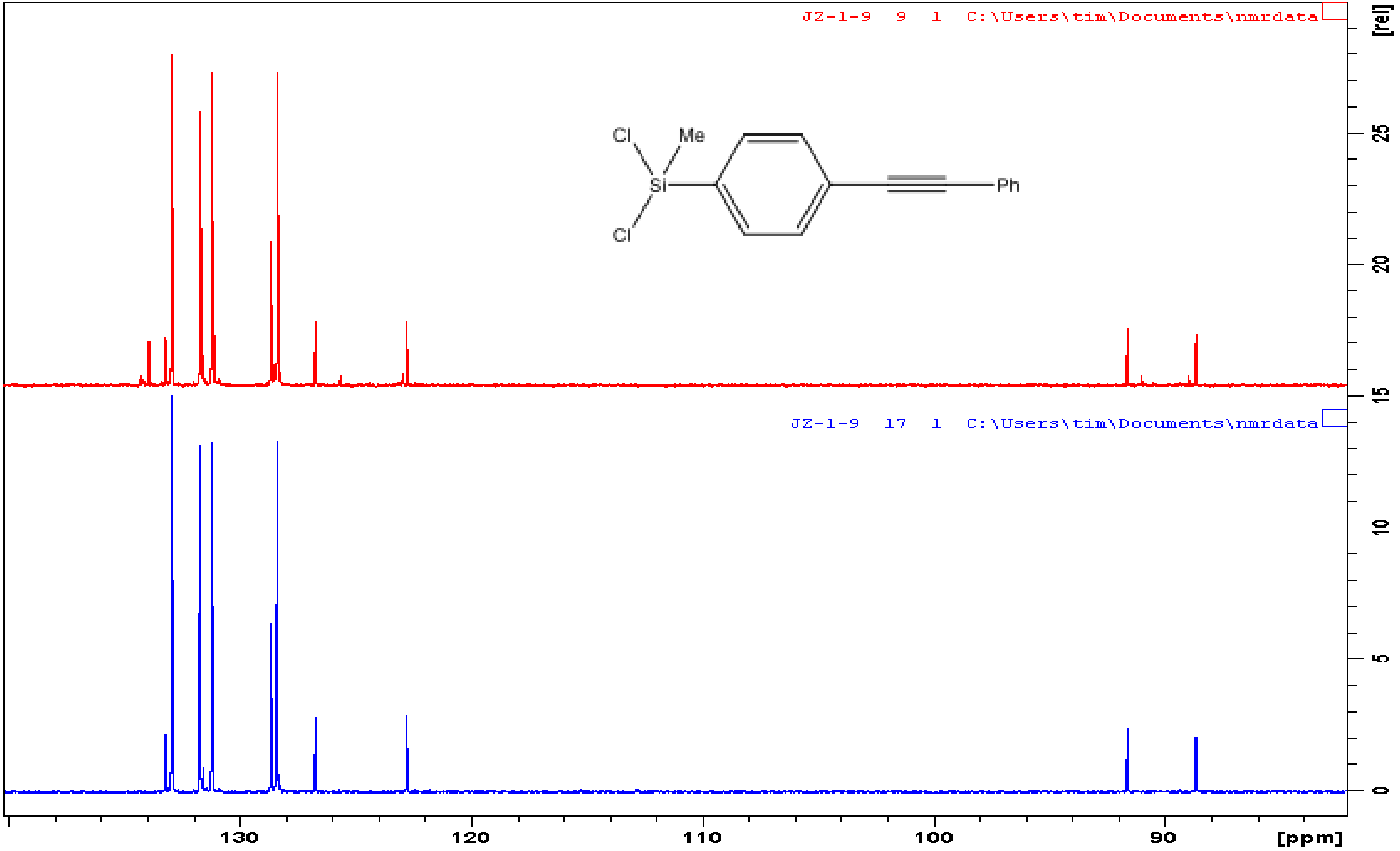
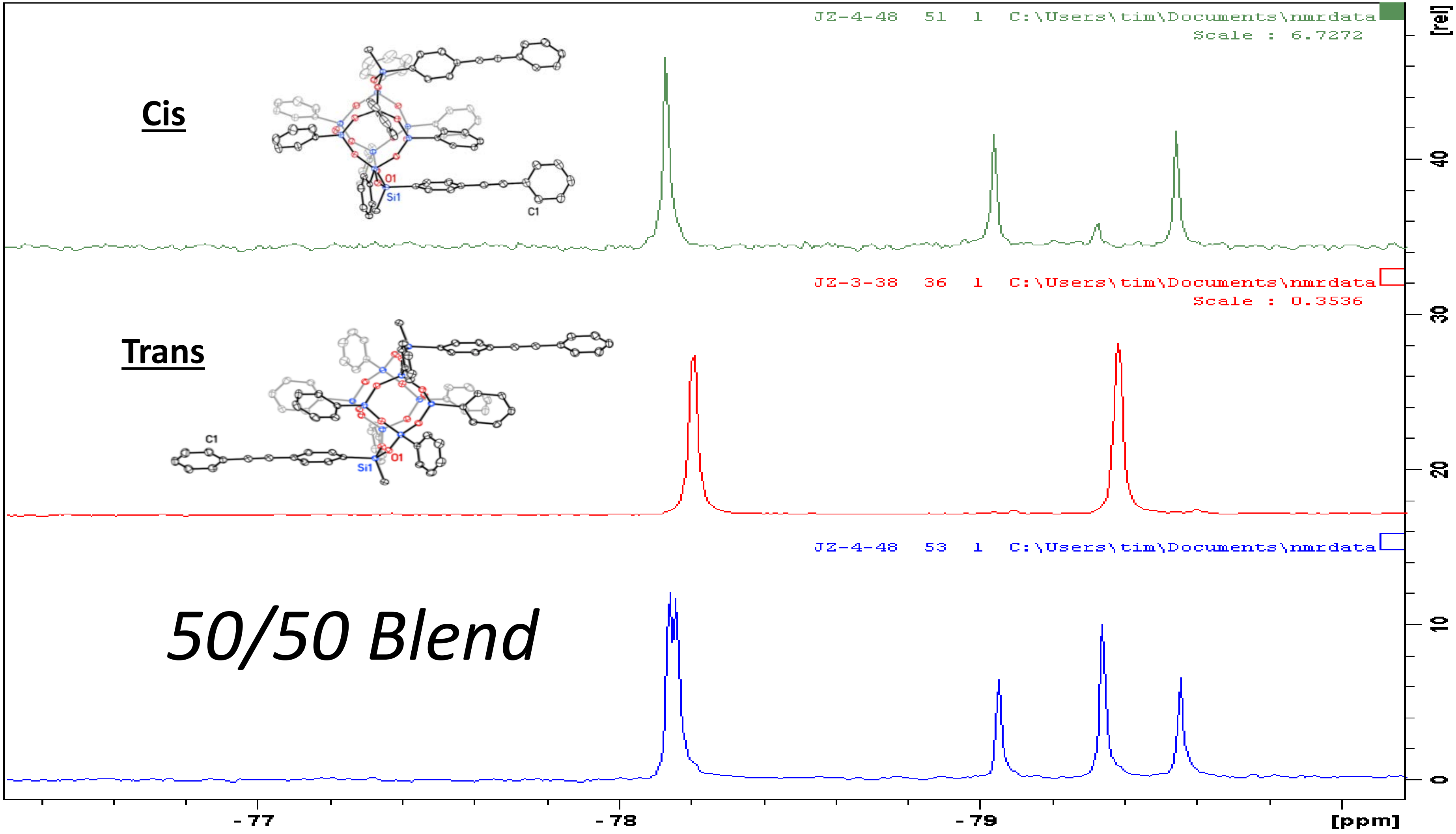
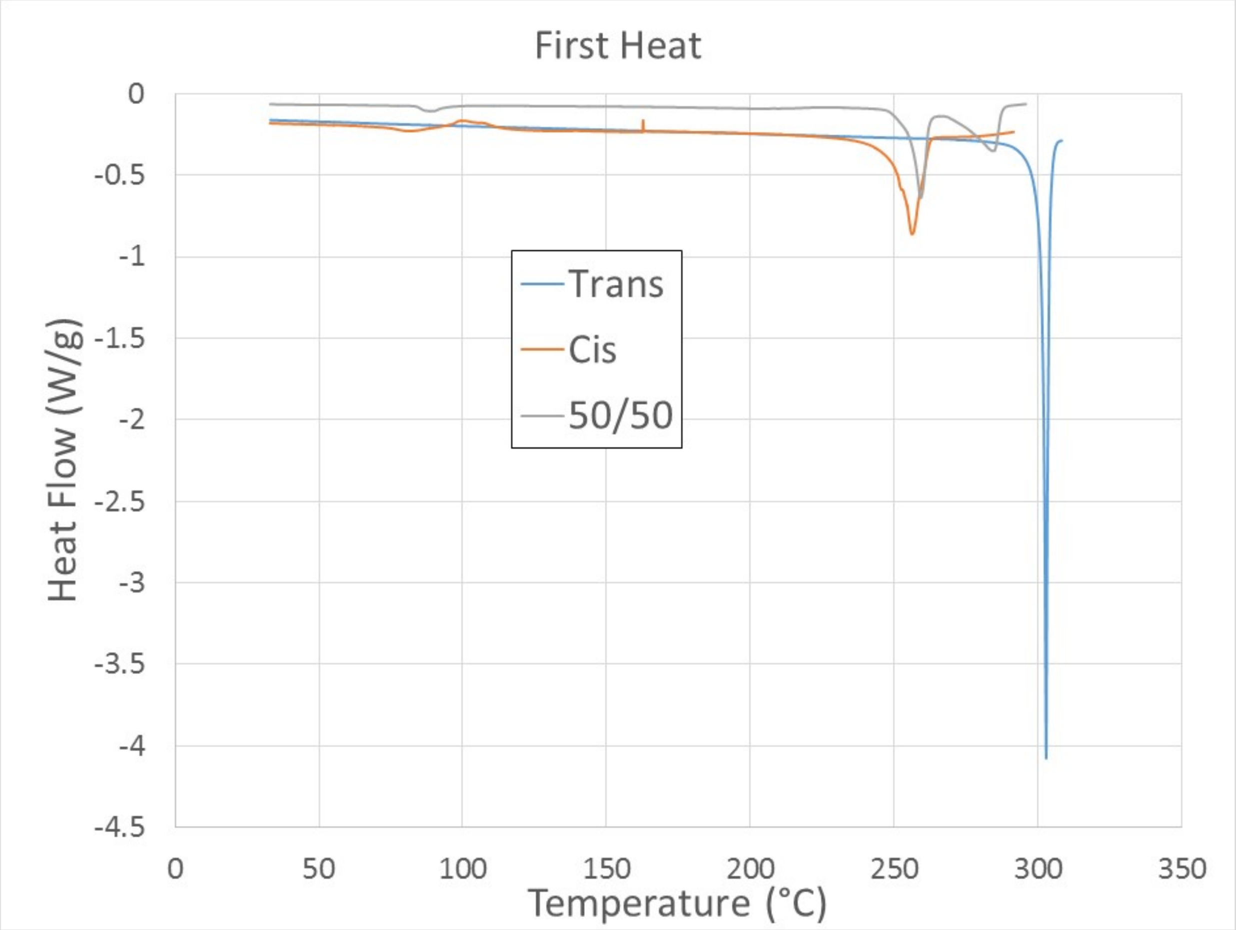
crude dichlorosilane vs. purified dichlorosilane

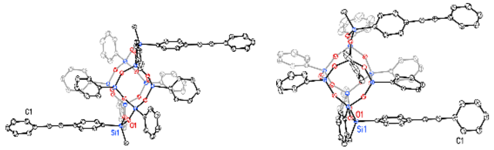


Si29 NMR spectral comparison



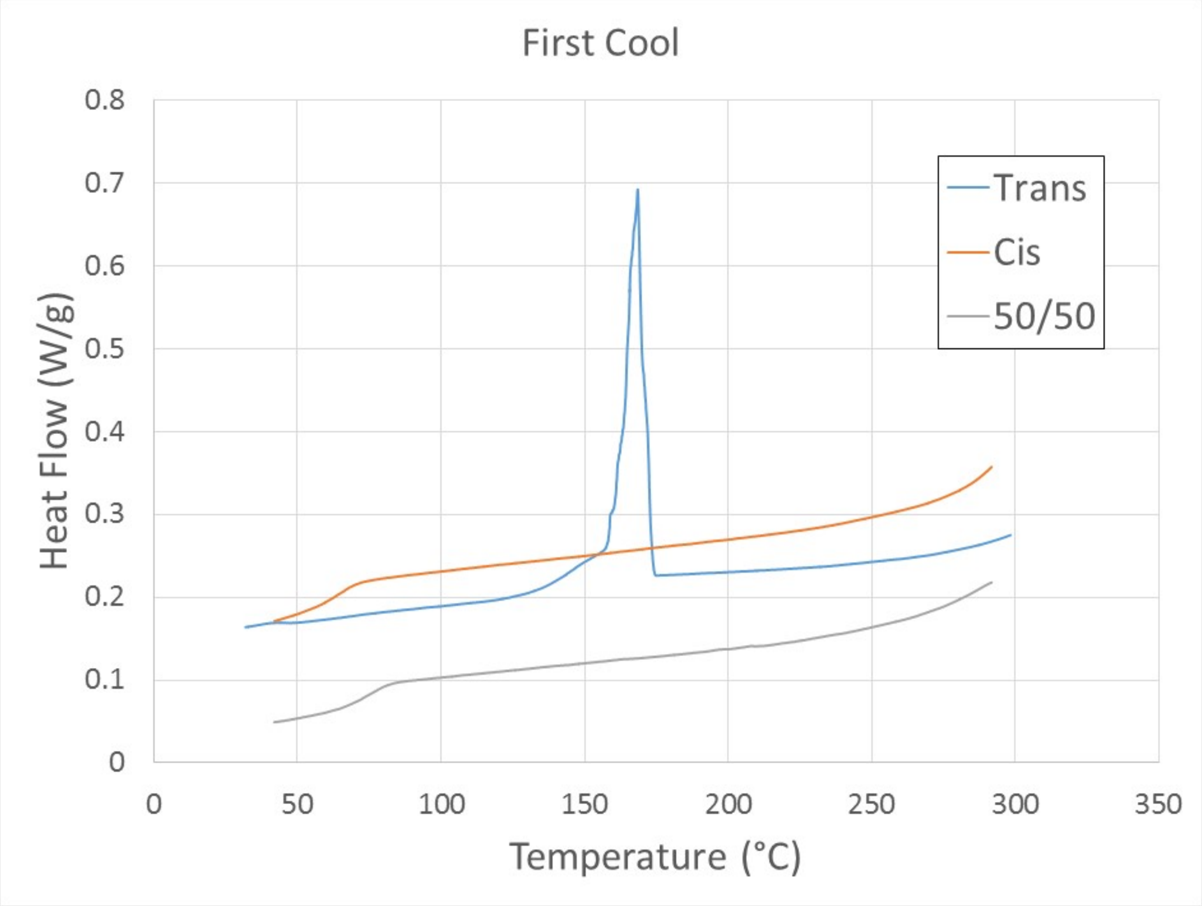
DSC

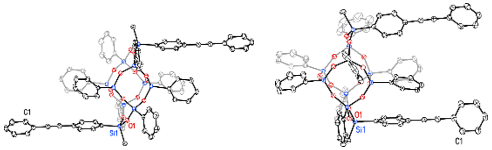




	Trans	Cis	50/50
Tg (°C)	-	77.5	85.3
Peak Tm (°C)	302.8	256.3	259.2, 284.5
Melt Enthalpy (J/g)	55.0	35.3	43.3

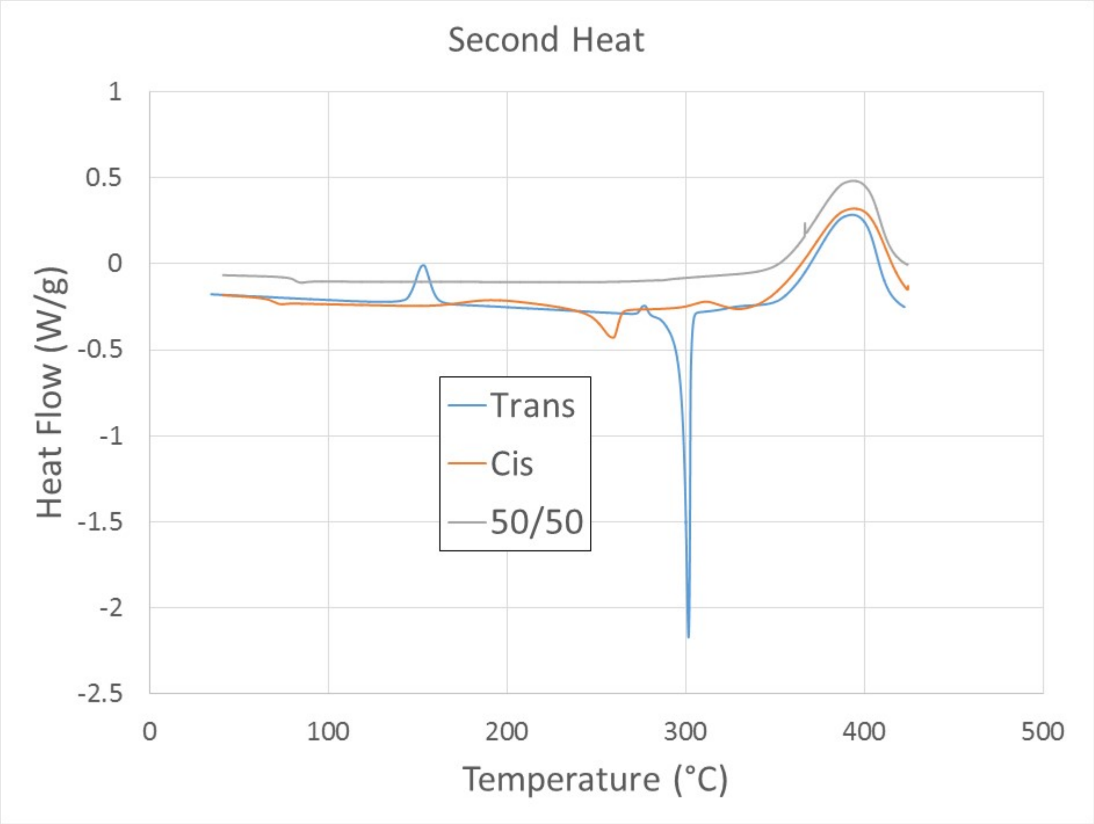
DSC

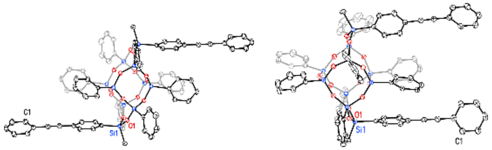




	Trans	Cis	50/50
Tg (°C)	-	66.4	73.1
Peak Tc (°C)	168.5	-	-
Crystallization Enthalpy (J/g)	24.8 (45.1%)	-	-

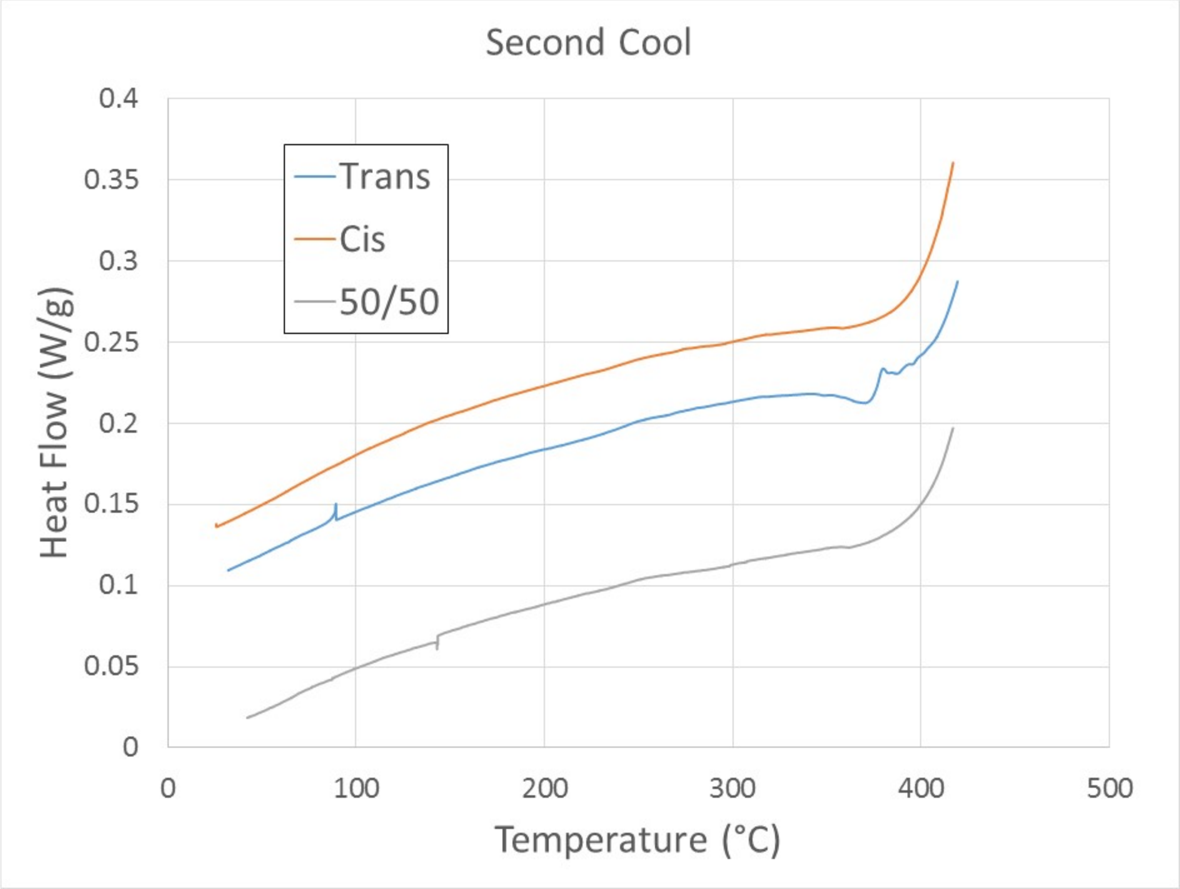
DSC

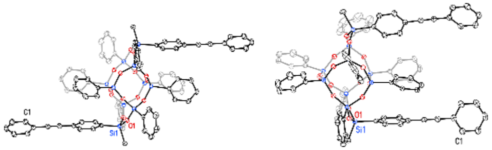




	Trans	Cis	50/50
Tg (°C)	-	71.0	81.5
Peak Tc (°C)	301.6	259.5	-
Melt Enthalpy (J/g)	50.94	16.9	-
Cure Peak Temperature (°C)	393.4	392.9	393.2
Cure Enthalpy (J/g)	124.5	137.8	123.6

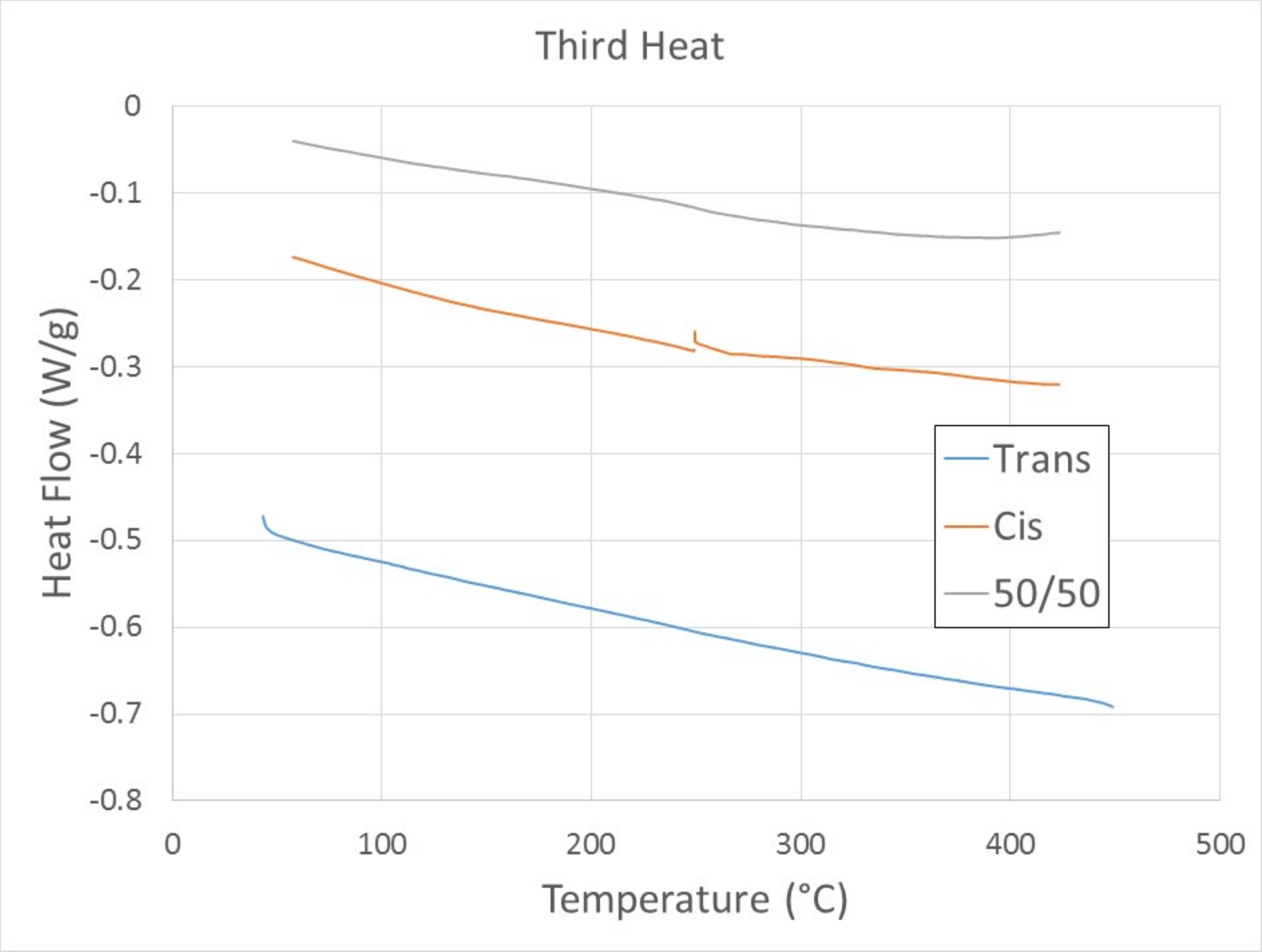
DSC





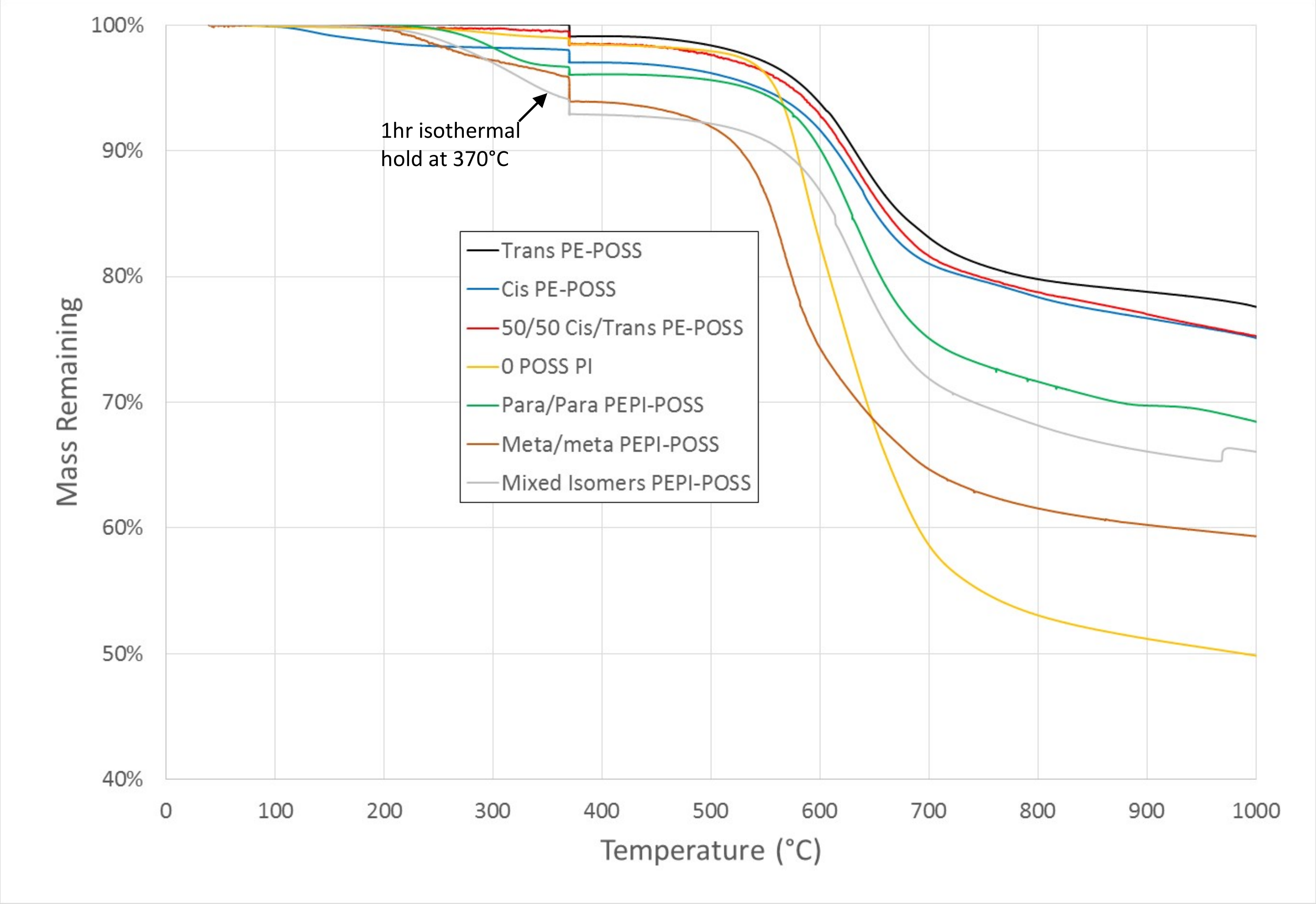
	Trans	Cis	50/50
Residual Cure Enthalpy (J/g)	2.4	13.0	5.7
Total Cure Enthalpy (J/g)	126.9	150.8	129.3

DSC


Third Heat
Heat Flow (W/g)
0
-0.1
-0.2
-0.3
-0.4
-0.5
-0.6
-0.7
-0.8
0
100
200
300
400
500
Temperature (°C)
Trans
Cis
50/50

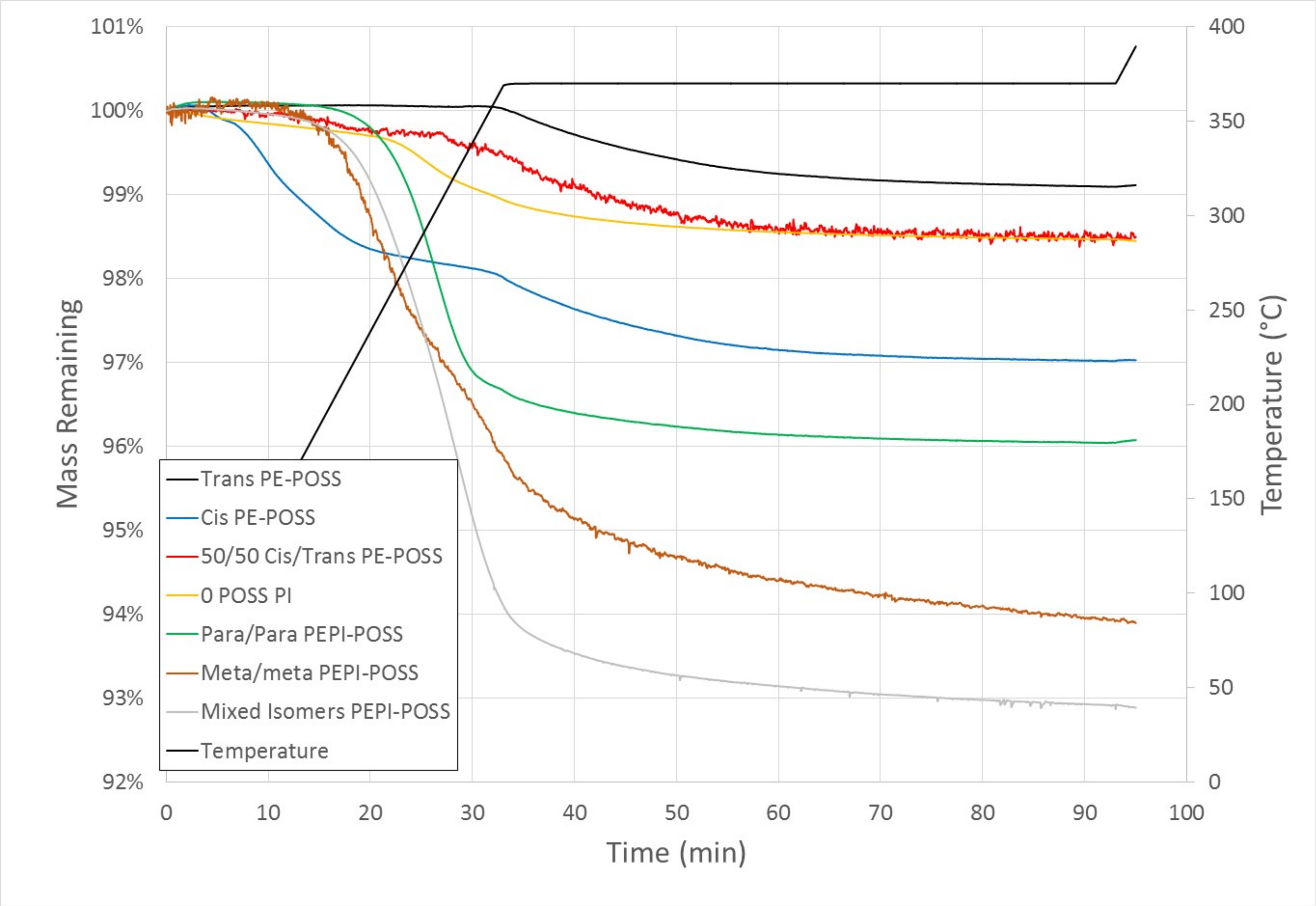
TGA

(10°C/min, N2)



TGA

(10°C/min, N2)



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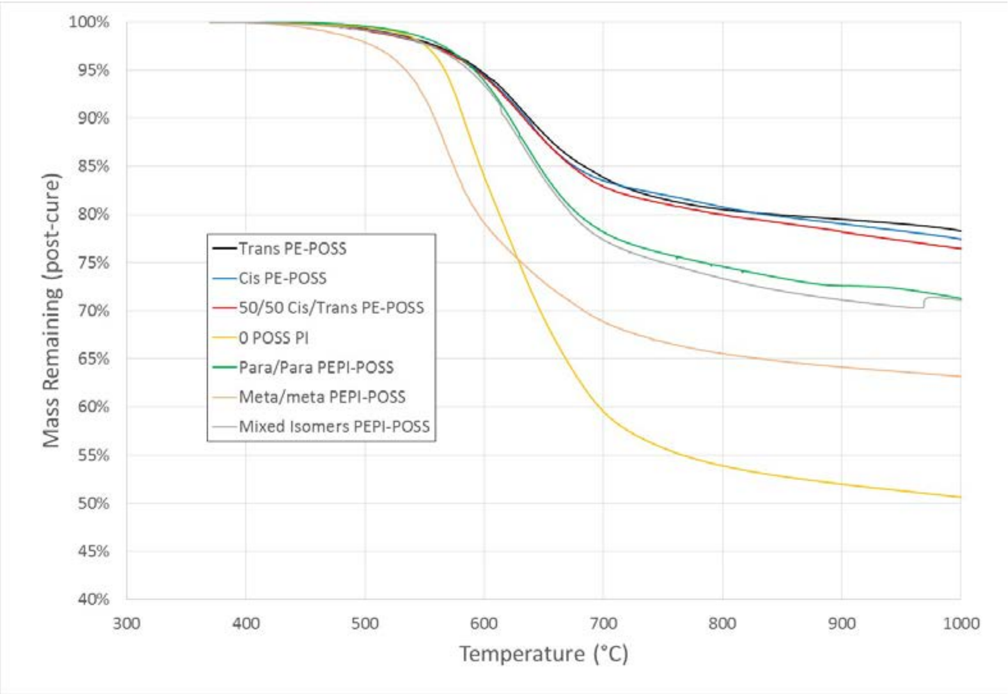
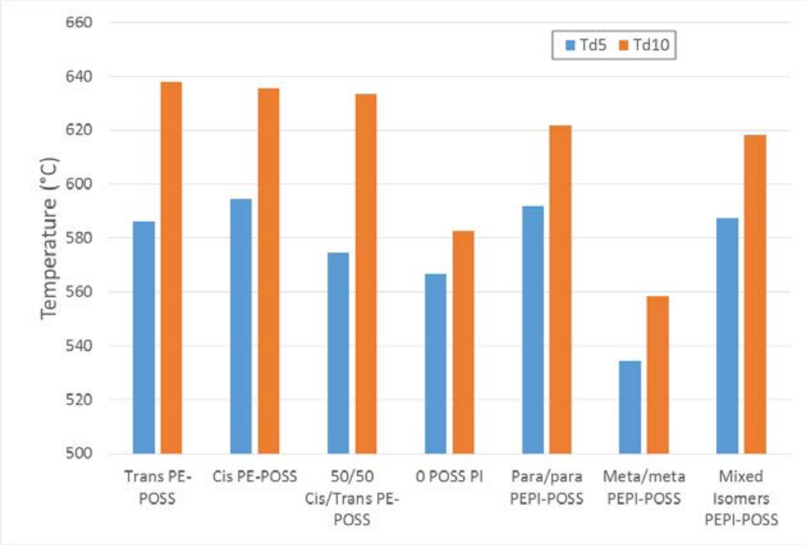
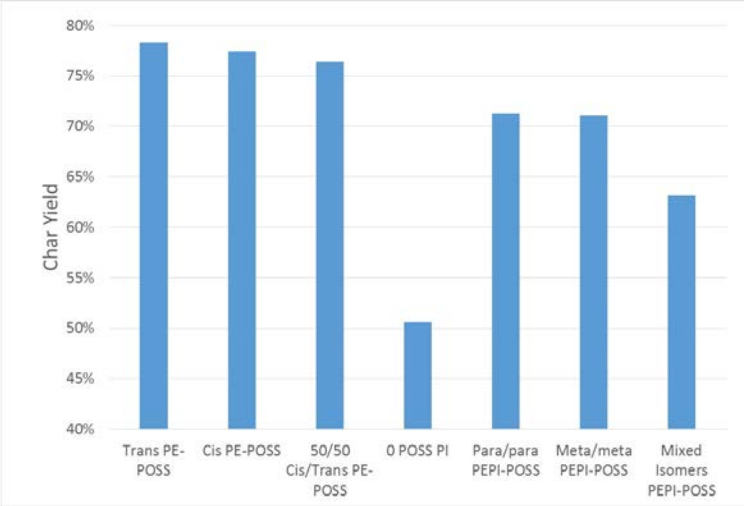
TGA

(10°C/min, N2)

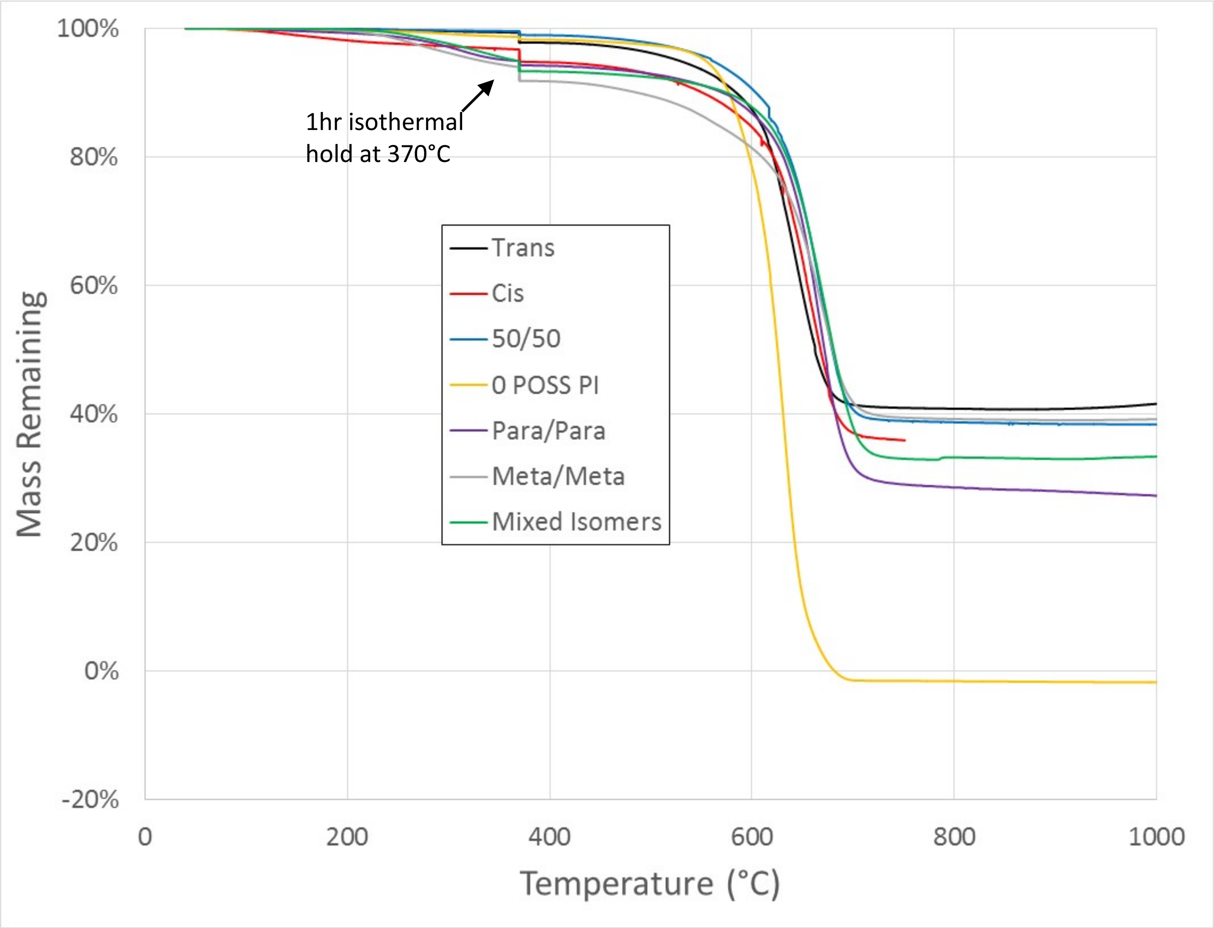






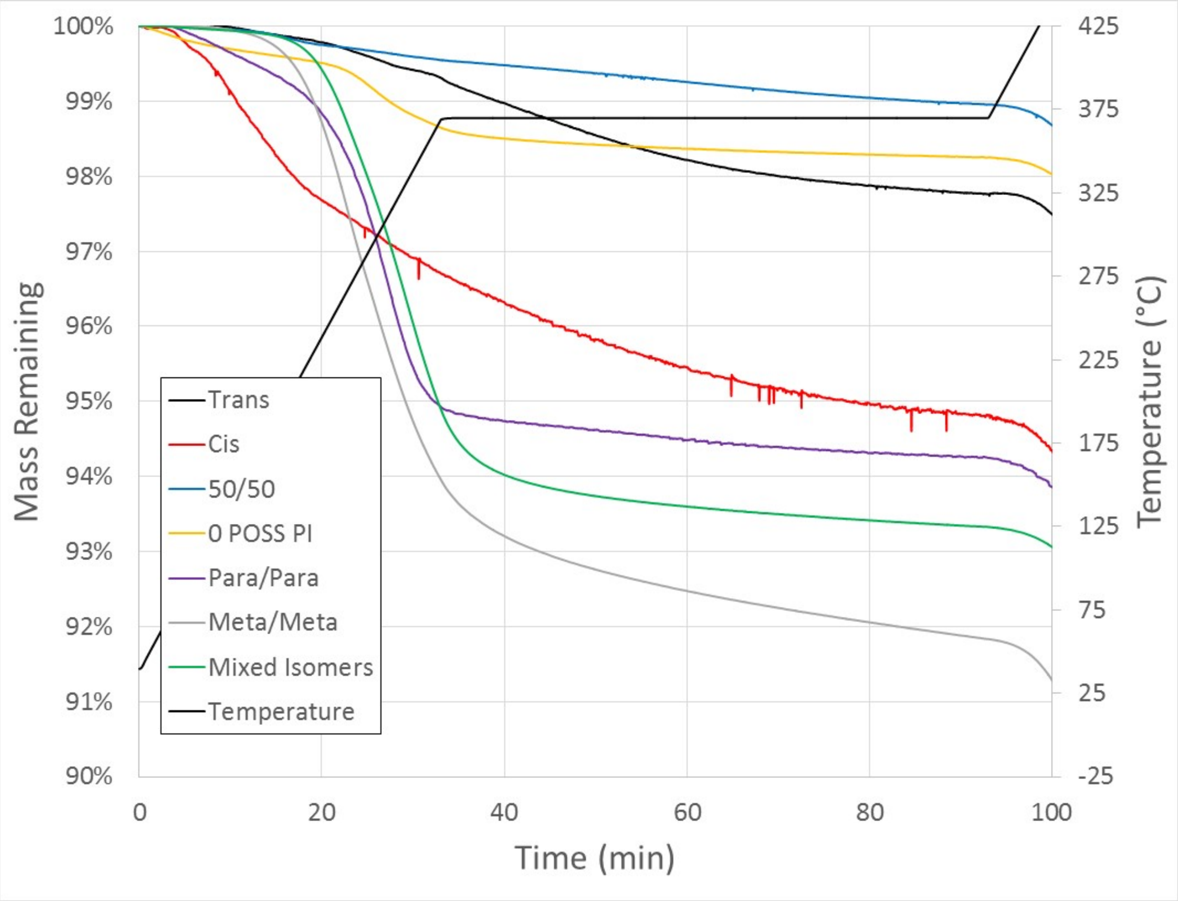
TGA

(10°C/min, AIR)



TGA

(10°C/min, AIR)



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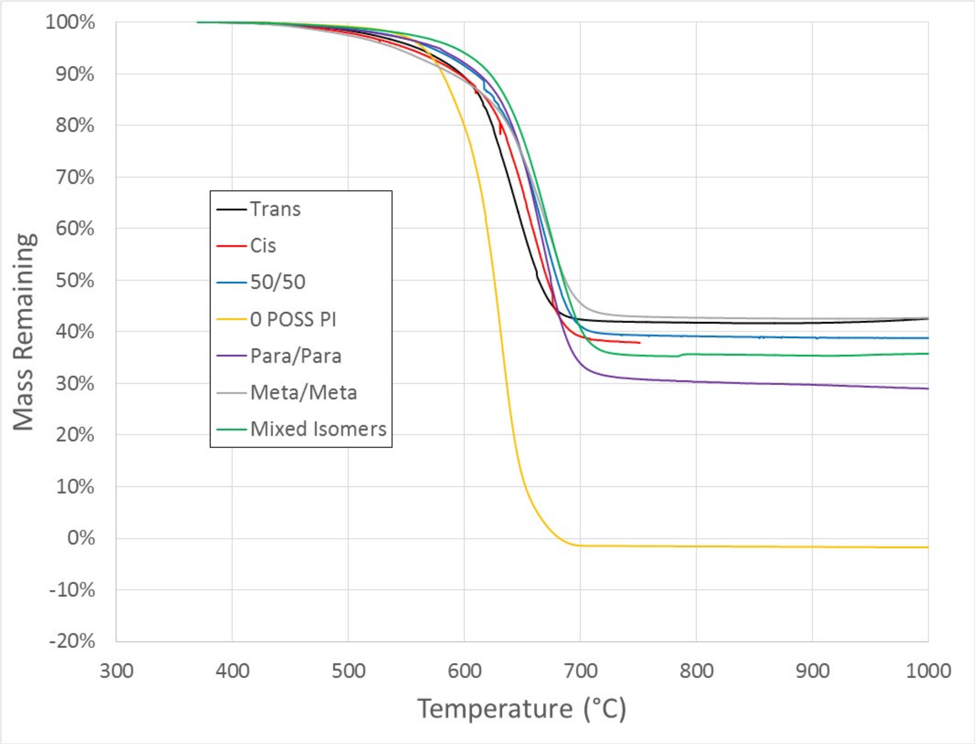
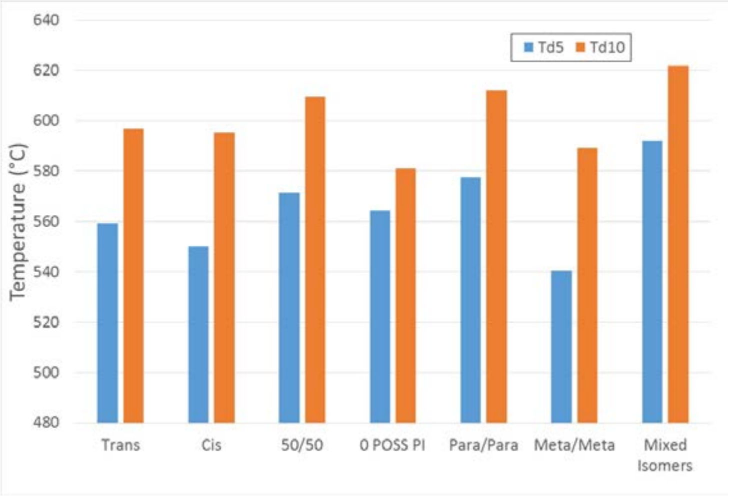
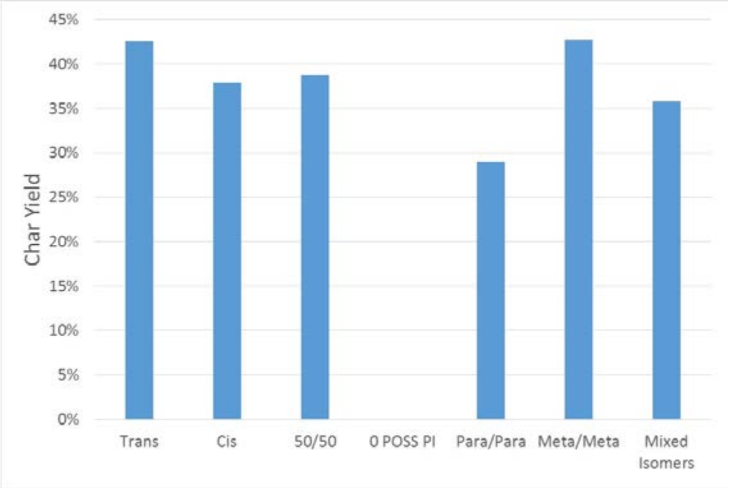
TGA

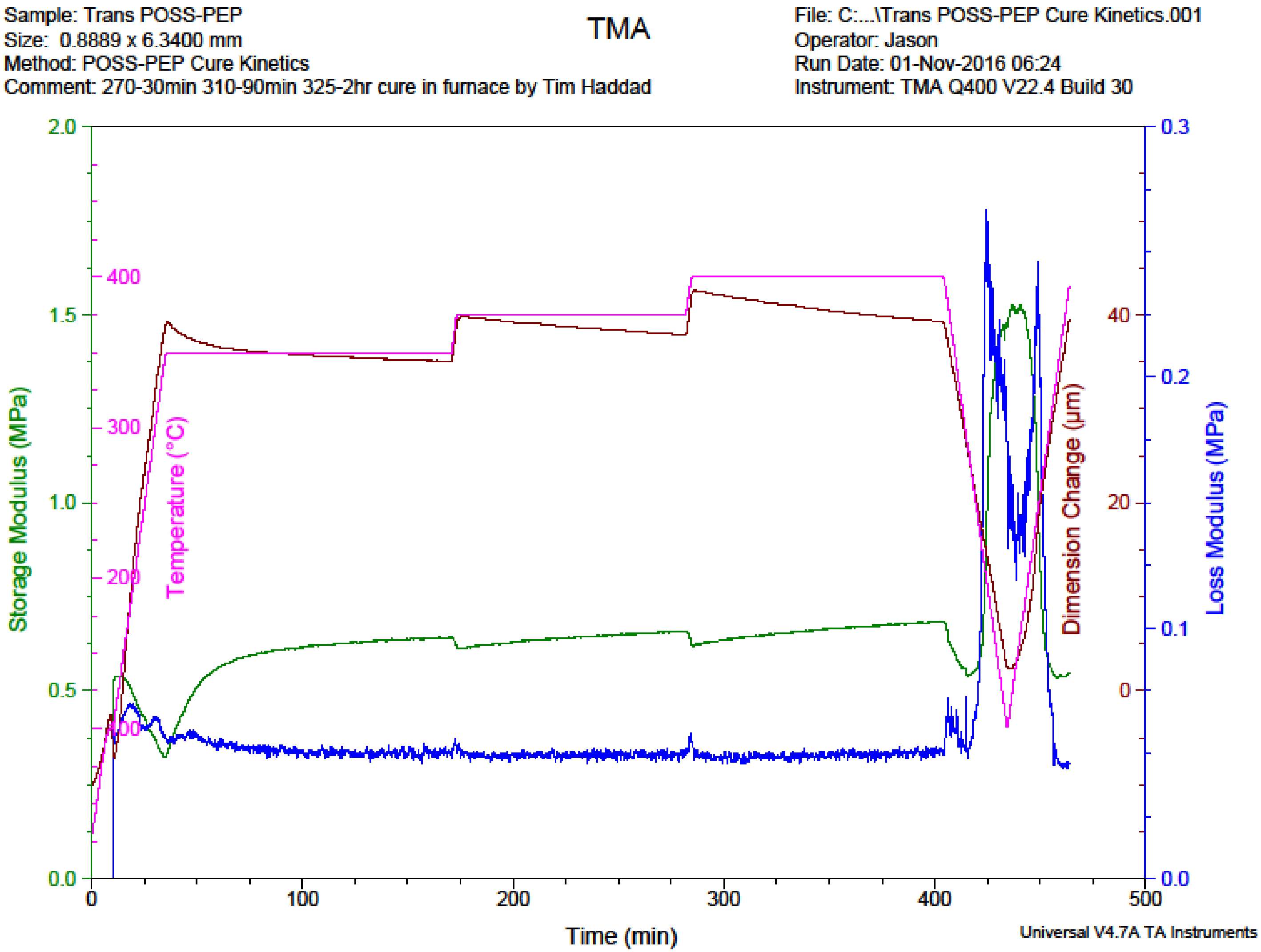
(10°C/min, AIR)







Trans BisPE-POSS TMA



Sample: Trans POSS-PEP
Size: 0.8889 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 270-30min 310-90min 325-2hr cure in furnace by Tim Haddad

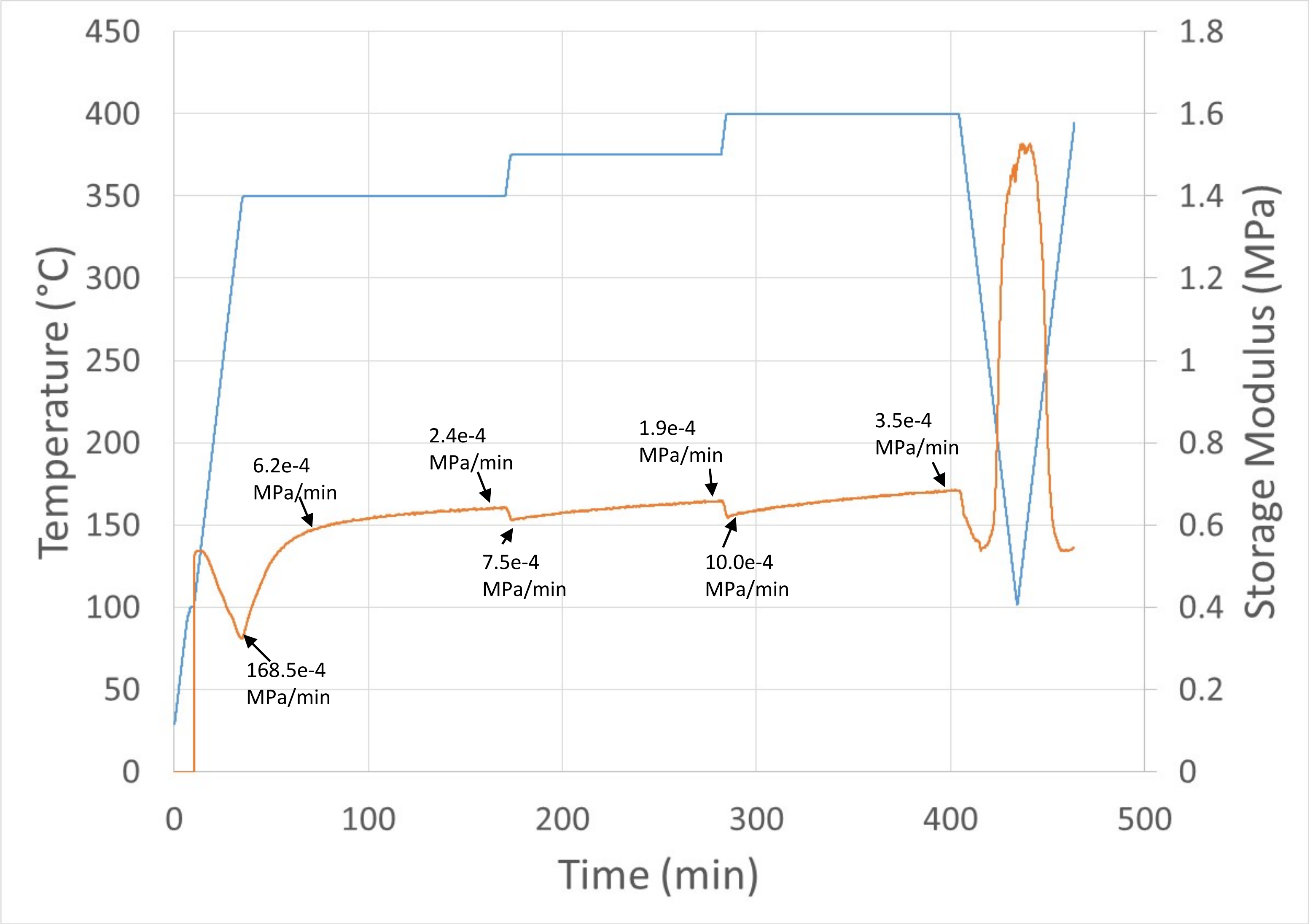
TMA

File: C:...\Trans POSS-PEP Cure Kinetics.001
Operator: Jason
Run Date: 01-Nov-2016 06:24
Instrument: TMA Q400 V22.4 Build 30

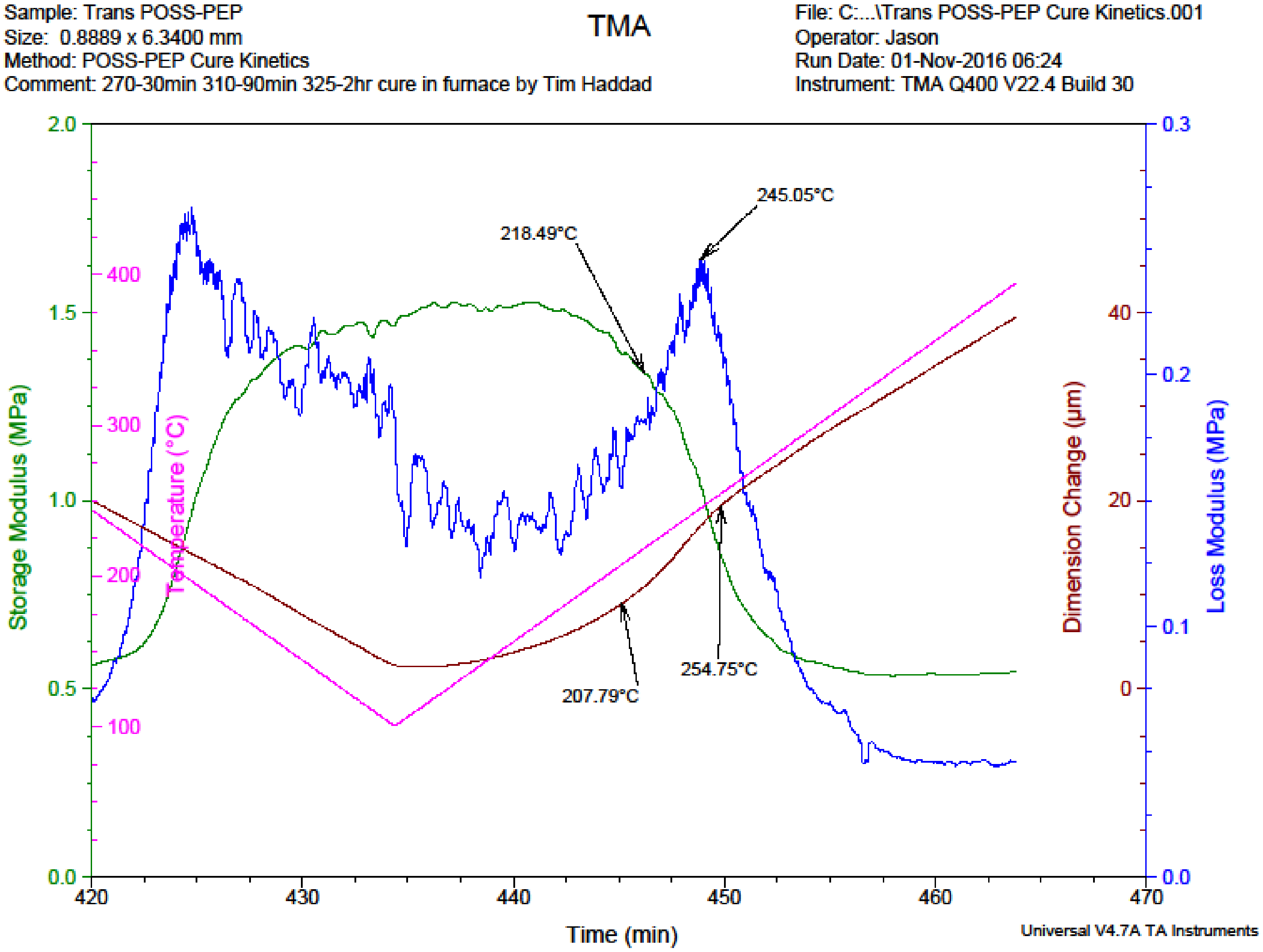
Universal V4.7A TA Instruments

Trans BisPE POSS
TMA Post-Cure

Cured in Furnace
- 270C, 30min
- 310C, 90min
- 325, 2hr



Trans BisPE-POSS
10°C/min Tg (TMA)

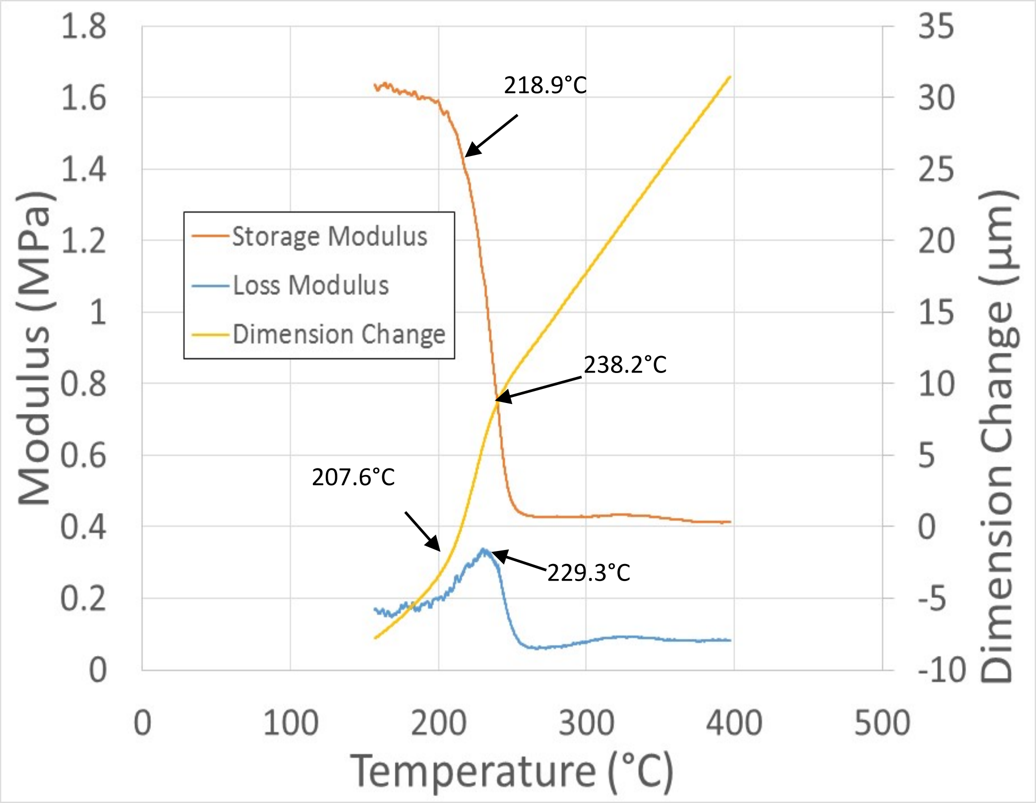


Sample: Trans POSS-PEP
Size: 0.8889 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 270-30min 310-90min 325-2hr cure in furnace by Tim Haddad

TMA

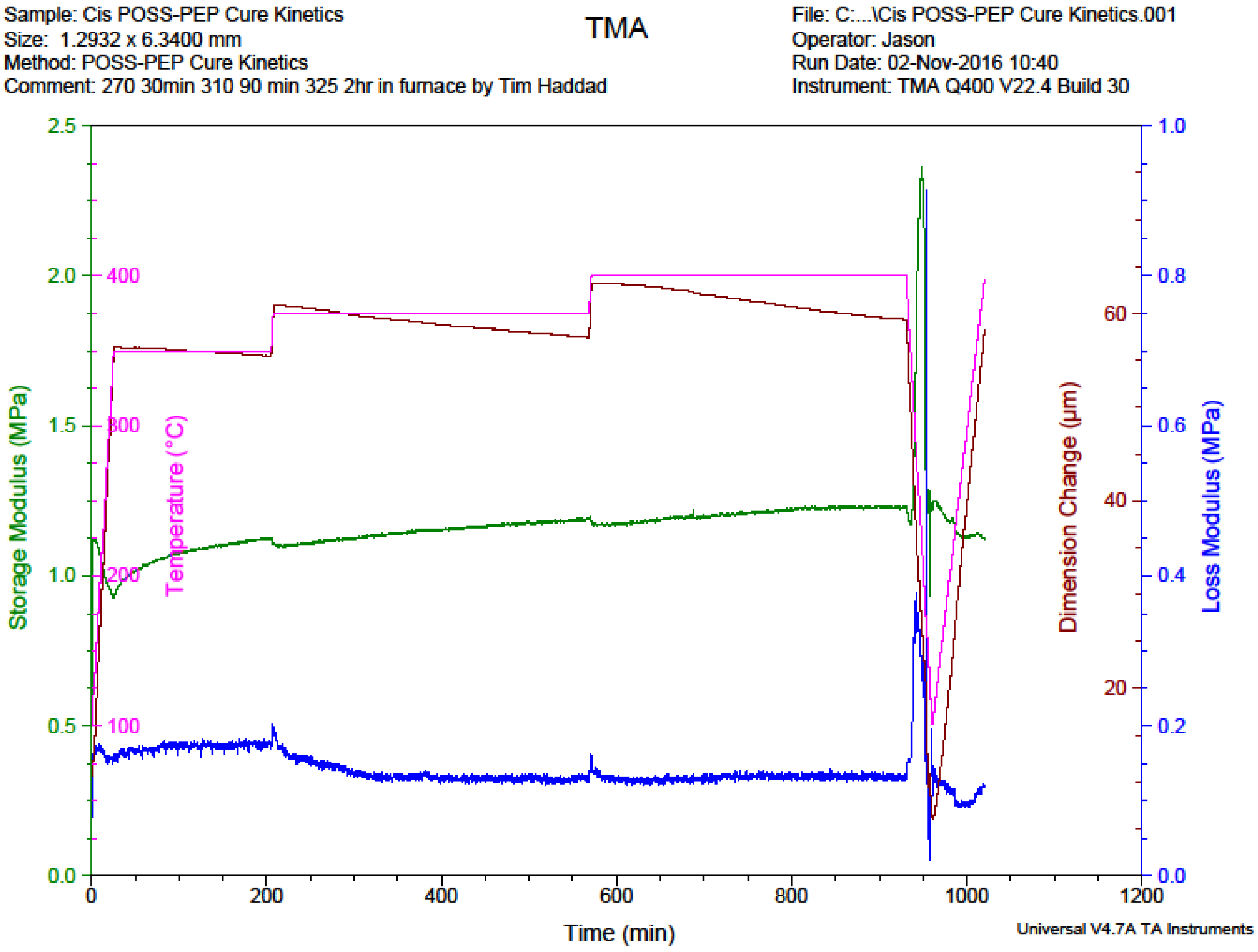
File: C:...\Trans POSS-PEP Cure Kinetics.001
Operator: Jason
Run Date: 01-Nov-2016 06:24
Instrument: TMA Q400 V22.4 Build 30

Trans BisPE-POSS
5°C/min Tg (TMA)



Method	Tg (°C) 5°C/min	Tg (°C) 10°C/min
Storage Modulus	218.9	218.5
Loss Modulus	229.3	245.1
Tan Delta	240.9	254.9
Dimension Change (Low)	207.6	207.8
Dimension Change (High)	238.2	254.8

Cis BisPE-POSS TMA



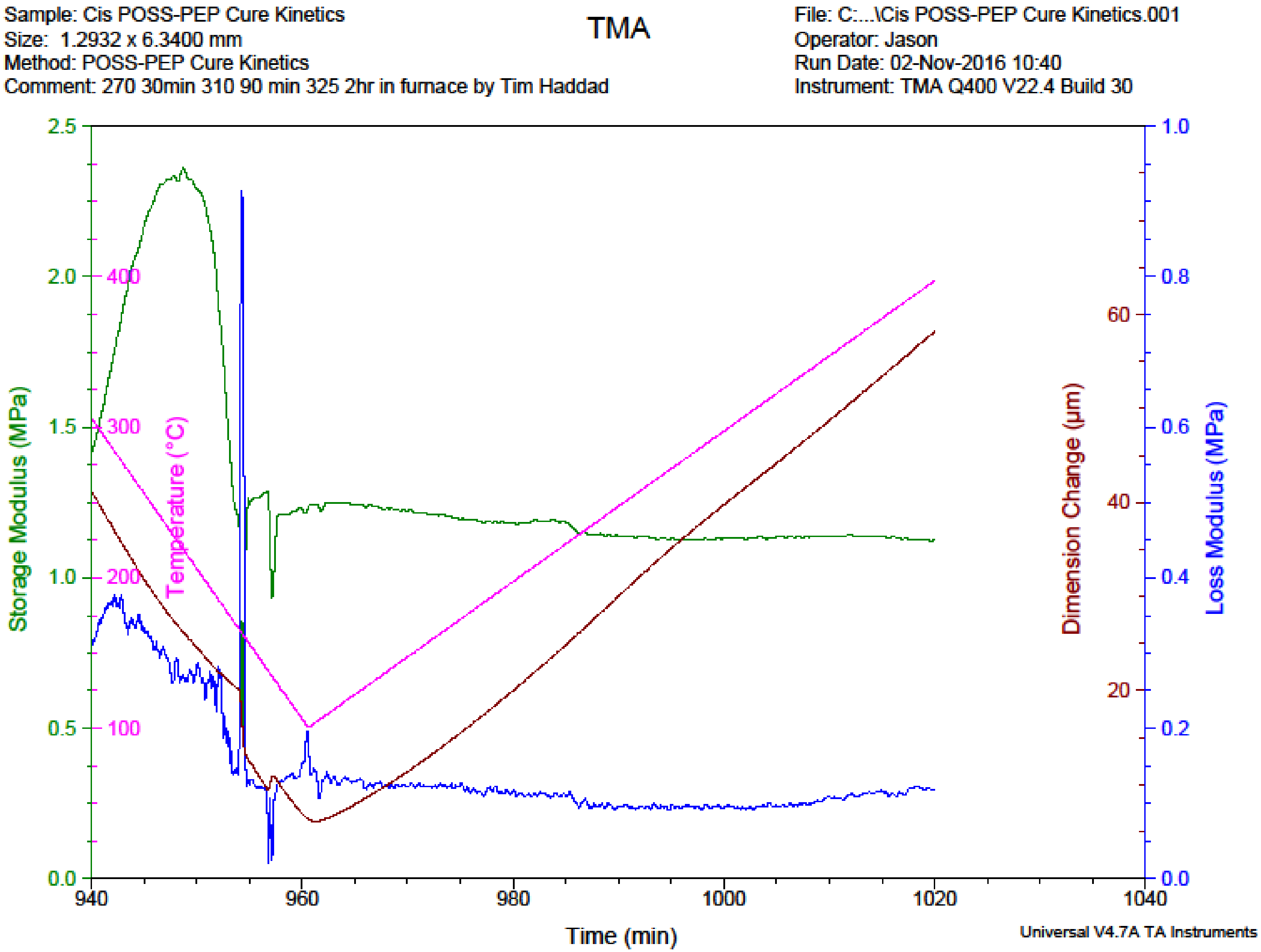
Sample: Cis POSS-PEP Cure Kinetics
Size: 1.2932 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 270 30min 310 90 min 325 2hr in furnace by Tim Haddad

TMA

File: C:...\Cis POSS-PEP Cure Kinetics.001
Operator: Jason
Run Date: 02-Nov-2016 10:40
Instrument: TMA Q400 V22.4 Build 30

Universal V4.7A TA Instruments

Cis BisPE-POSS TMA



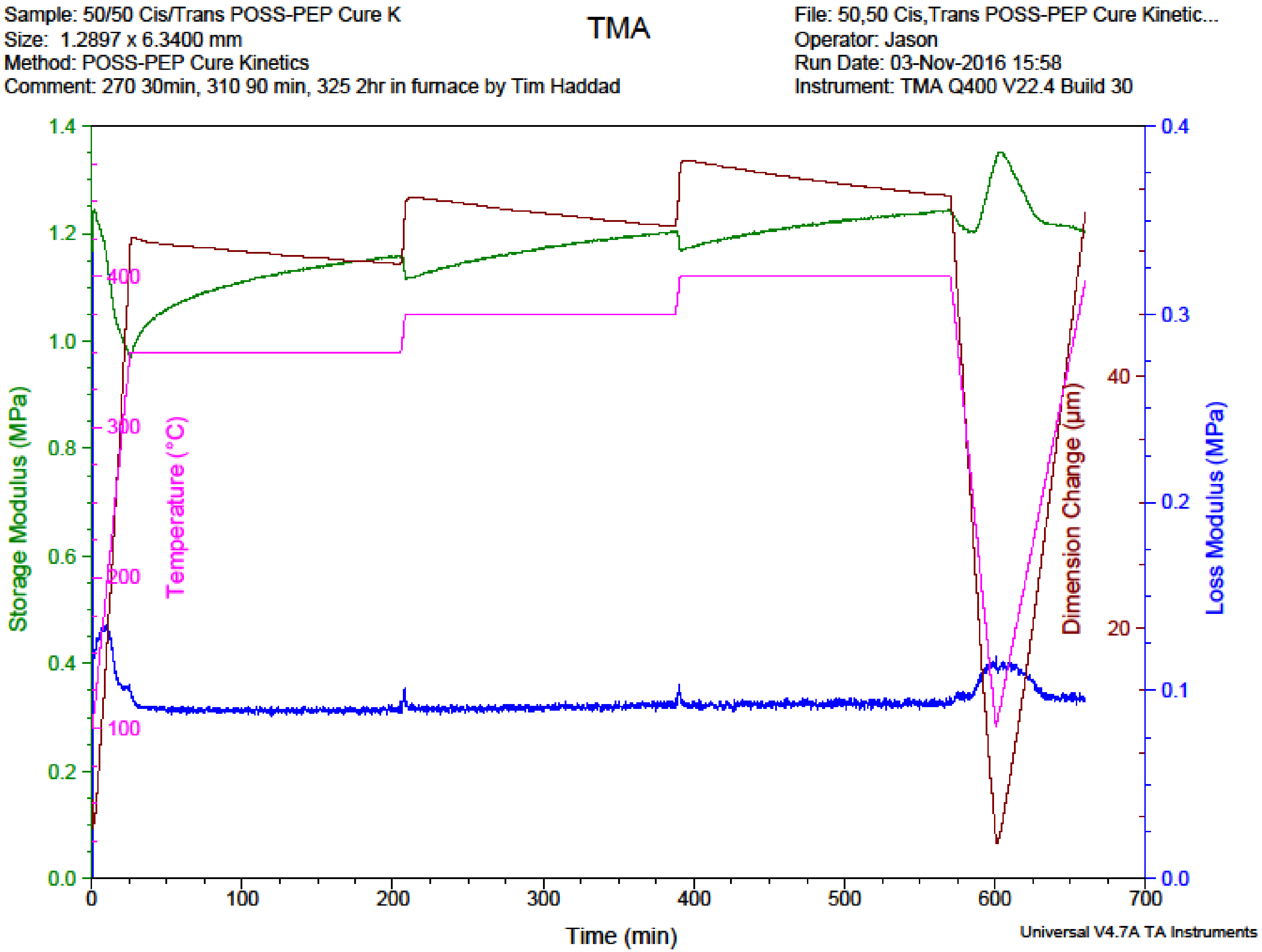
Sample: Cis POSS-PEP Cure Kinetics
Size: 1.2932 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 270 30min 310 90 min 325 2hr in furnace by Tim Haddad

TMA

File: C:...\Cis POSS-PEP Cure Kinetics.001
Operator: Jason
Run Date: 02-Nov-2016 10:40
Instrument: TMA Q400 V22.4 Build 30

Universal V4.7A TA Instruments

50/50 Cis/Trans BisPE-POSS TMA



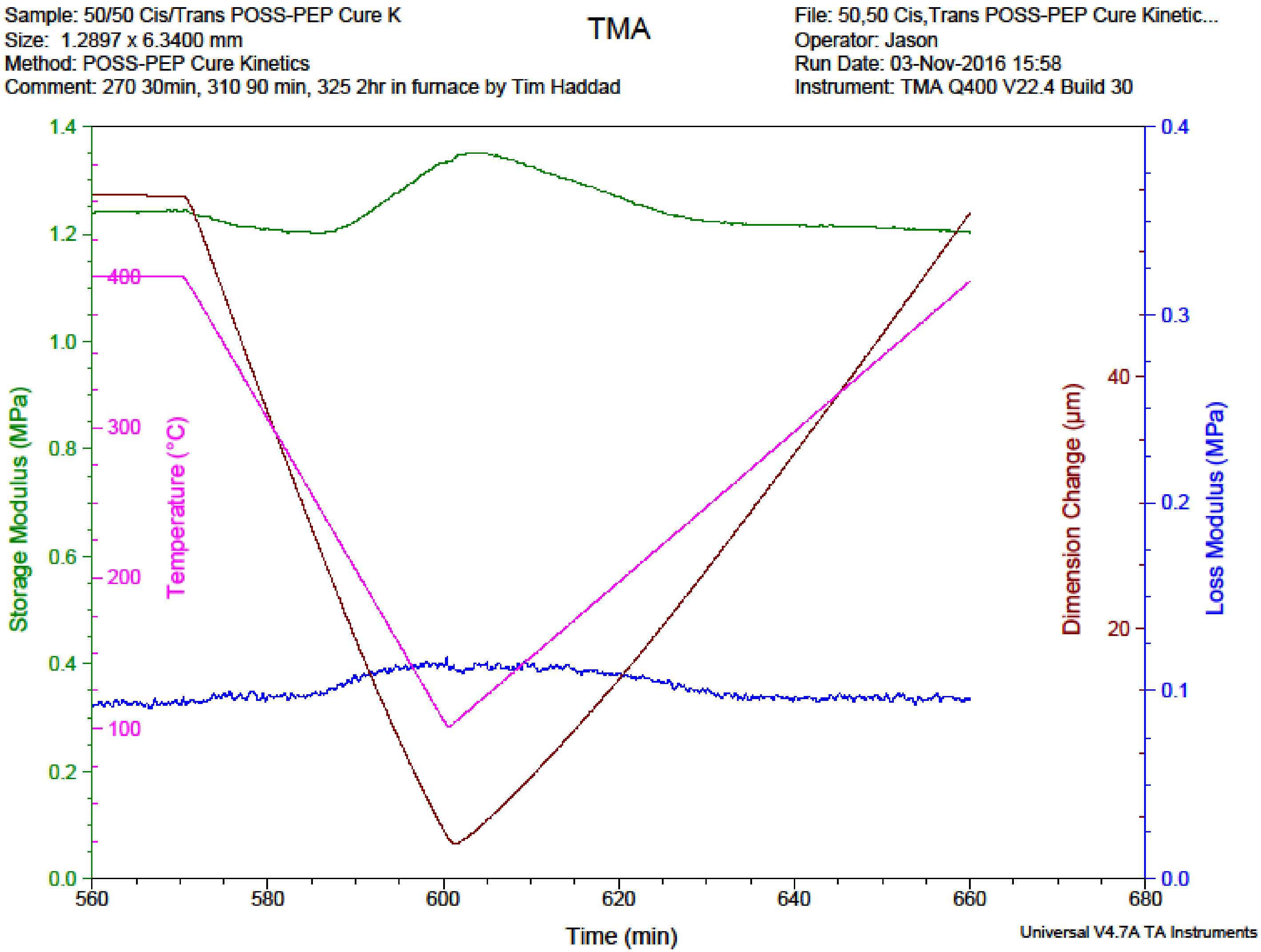
Sample: 50/50 Cis/Trans POSS-PEP Cure K
Size: 1.2897 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 270 30min, 310 90 min, 325 2hr in furnace by Tim Haddad

TMA

File: 50,50 Cis,Trans POSS-PEP Cure Kinetic...
Operator: Jason
Run Date: 03-Nov-2016 15:58
Instrument: TMA Q400 V22.4 Build 30

Universal V4.7A TA Instruments

50/50 Cis/Trans BisPE-POSS TMA



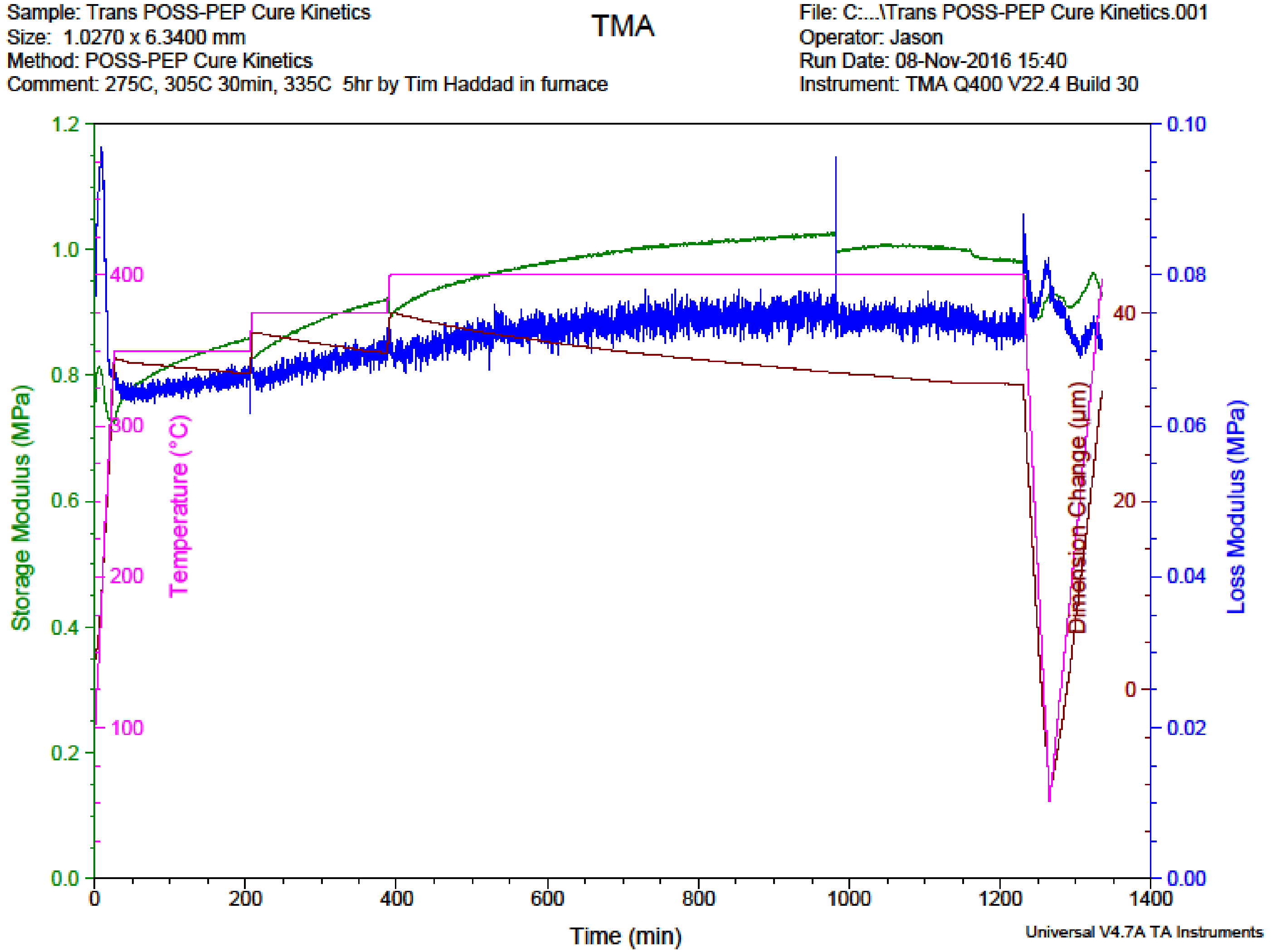
Sample: 50/50 Cis/Trans POSS-PEP Cure K
Size: 1.2897 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 270 30min, 310 90 min, 325 2hr in furnace by Tim Haddad

TMA

File: 50,50 Cis,Trans POSS-PEP Cure Kinetic...
Operator: Jason
Run Date: 03-Nov-2016 15:58
Instrument: TMA Q400 V22.4 Build 30

Trans BisPE-POSS TMA

400°C for 12 hrs



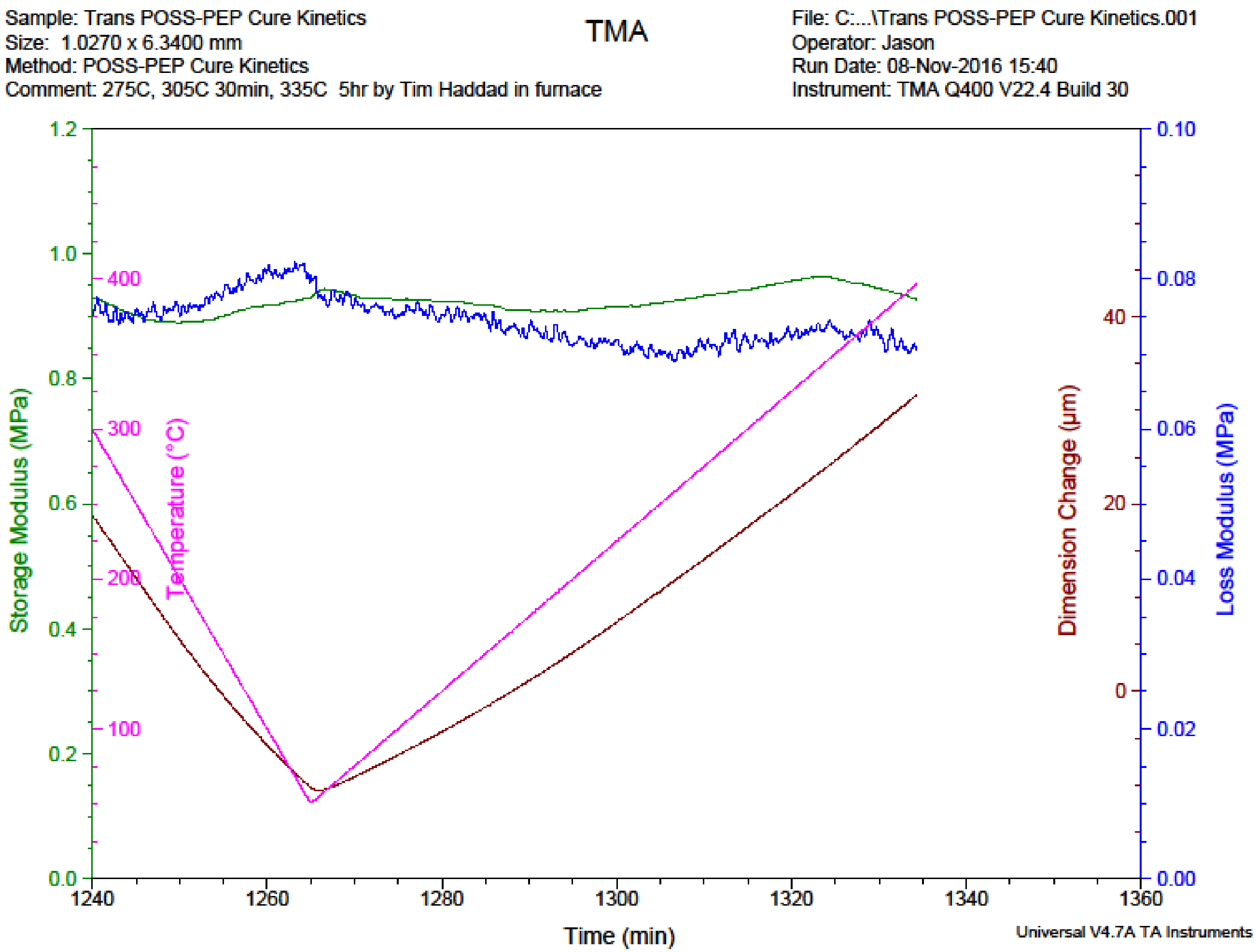
Sample: Trans POSS-PEP Cure Kinetics
Size: 1.0270 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 275C, 305C 30min, 335C 5hr by Tim Haddad in furnace

TMA

File: C:...\Trans POSS-PEP Cure Kinetics.001
Operator: Jason
Run Date: 08-Nov-2016 15:40
Instrument: TMA Q400 V22.4 Build 30

Trans BisPE-POSS TMA

400°C for 12 hrs



Sample: Trans POSS-PEP Cure Kinetics
Size: 1.0270 x 6.3400 mm
Method: POSS-PEP Cure Kinetics
Comment: 275C, 305C 30min, 335C 5hr by Tim Haddad in furnace

TMA

File: C:...\Trans POSS-PEP Cure Kinetics.001
Operator: Jason
Run Date: 08-Nov-2016 15:40
Instrument: TMA Q400 V22.4 Build 30

Sample	Density (g/cc)
Cis bisPE 3hr	1.203
50/50 bisPE 3hr	1.207
Trans bisPE 3hr	1.224
Trans bisPE 12hr	1.203
Para/Para bisPEPI	1.240
Mixed Isomers bisPEPI	1.245

Summary & Future Work

- High Performance Polyimides successfully made using silsesquioxanes
  - POSS helps with moisture uptake
  - POSS helps with thermal stabilty
- Long Polyimide Oligomers and very short POSS-dimides can be used
  - Tgs' a little too low in long oligomers
- Strategy to produce Imide-Free BisphenylethynylPOSS sucessful
  - Fully characterization demonstrates and excellent moist
  - Improved thermal stability
  - Excellent moisture resistance
- Next Steps:
  - Synthesize mixed isomers to reduce melting point
  - Make composites and measure mechanical and thermal properties

QUESTIONS?