Material Characterization for Processing: Hexcel 8552

Material Model Development

Final Project Wrap-Up

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Approved: A.Poursartip

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Version: 1.0

Project Definition

Material: Hexcel 8552

Modeling performed for:

- Cure Kinetics
- Heat Capacity (Cp)
- Viscosity

Material Description

- **HEXPLY 8552**

- Material forms received from Hexcel are as follows:
  - Neat Resin: HS-AD-693 (received Oct 18, 2006)
  - Resin Film: 74#CCA1030/B430, 35G, 45.75 (received Oct 18, 2006)

Cure Kinetics Model

Nomenclature

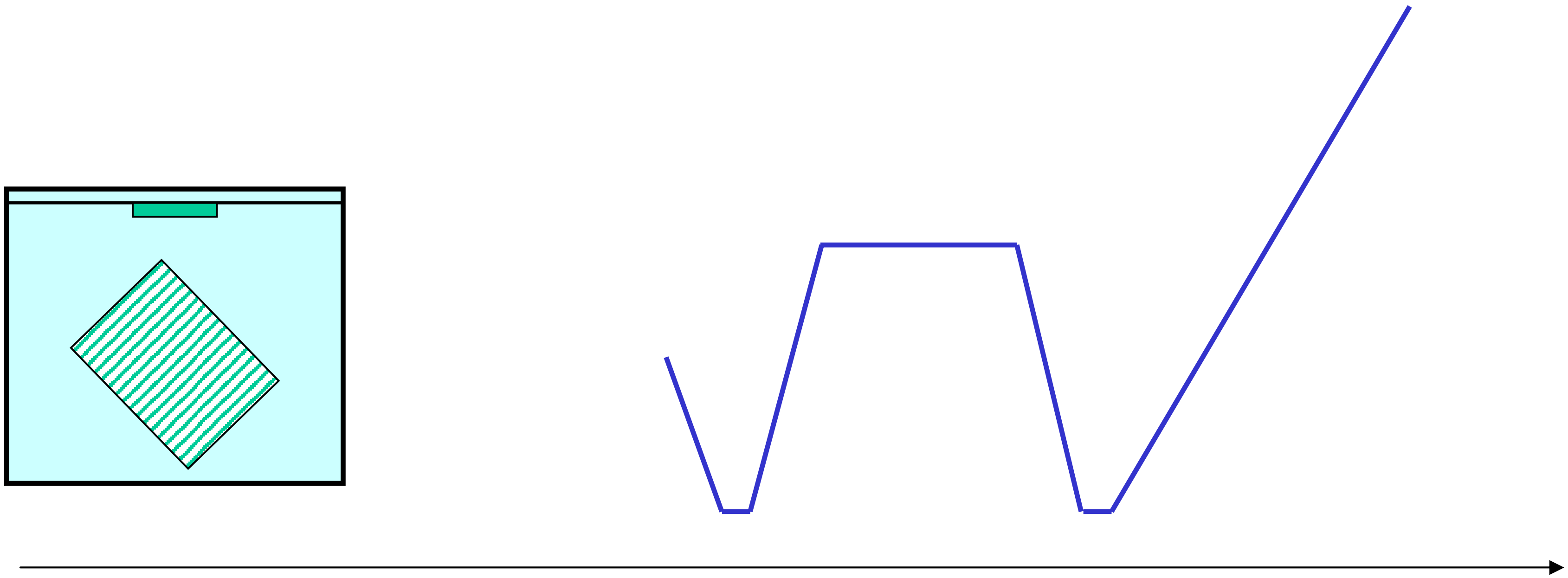
Iso

Int

Dyn

RES

HR Definition



DSC Test Procedure

Isothermal Tests

In isothermal tests, the sample is equilibrated at a very low temperature and then heated up to a predefined hold temperature at a very high rate. The sample is held at this temperature for a predetermined duration of time (i.e. until the reaction stops due to diffusion or full cure in the case of conditioned isothermal tests, or earlier as predefined for interrupted isothermal tests). The sample is then cooled down, followed by a residual ramp at a known rate (typically 1-4 cpm). The residual ramp ensures that the material is fully cured, and provides the material Tg at the end of hold, as well as the residual heat of reaction. A second ramp is also performed to determine the final (full cure) Tg.

Dynamic Tests

In dynamic tests, the sample is equili

DSC Tests Performed

The DSC tests were performed are summarized below:

- Dynamic tests: 18 tests @ 1 to 10ºCpm
- Isothermal tests: 11 tests 100ºC to 190ºC
- Interrupted Isothermal tests: 2 tests at 160ºC
- Other: 4 cure cycle tests, 4 dynamic on other forms

After careful examination of the results and investigation of the consistency of the data, the comp

DSC Tests Used in Model Fitting

Test	Mass (mg)	Rate (ºC/min)	$Temp_{max}$ (ºC)
8552-MDYN-01cpm-01	4.99	1	300
8552-MDYN-02cpm-01	4.19	2	300
8552-MDYN-02cpm-02	5.3	2	280
8552-MDYN-03cpm-01	5.59	3	300
8552-MDYN-04cpm-01	5.59	4	300
8552-MDYN-05cpm-01	4.79	5	300
8552-MDYN-06cpm-01	4.79	6	300
8552-MDYN-07cpm-01	5.89	7	300
8552-MDYN-08cpm-01	4.09	8	330
8552-MDYN-09cpm-01	5.19	9	330
8552-MDYN-10cpm-01	3.39	10	330

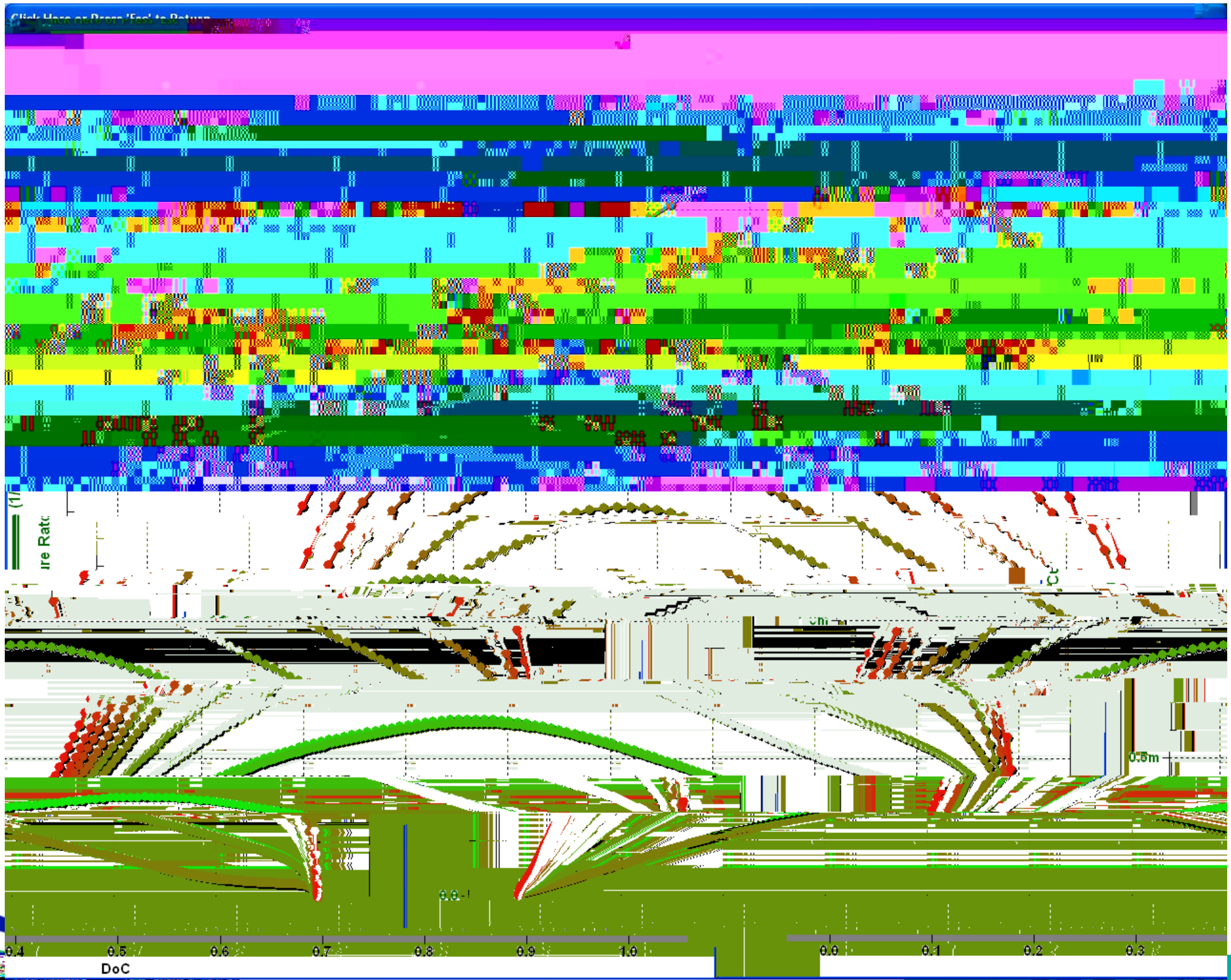
Mass
(mg)

Degree of Cure Calculation

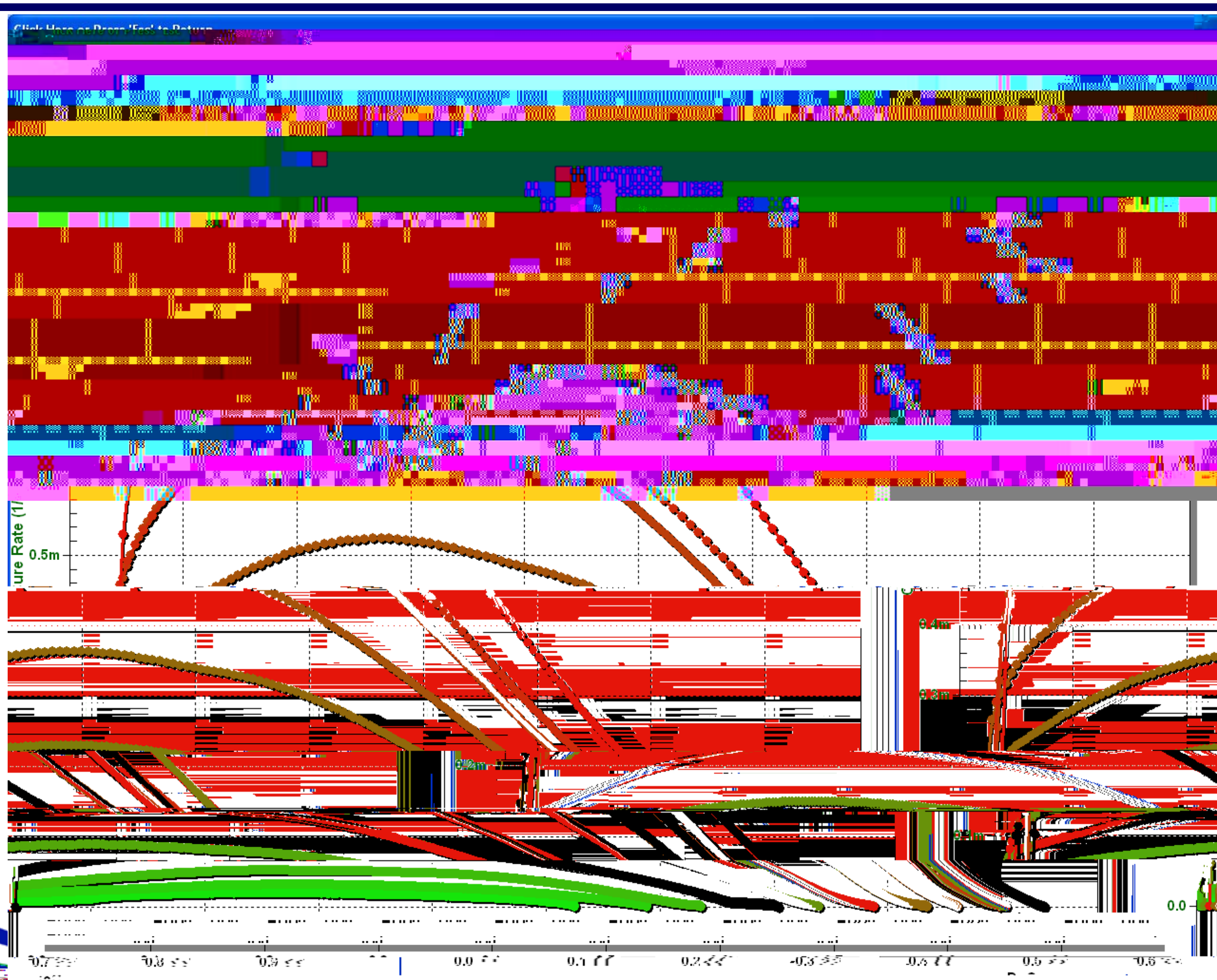
Raw DSC data was linearly sparsed and smoothed.

A bi-linear baseline was considered for dynamic tests in order to calculate the total heat of reaction and degree of cure.
Baselines fitted to

Cure Rate vs. DoC – Dyns



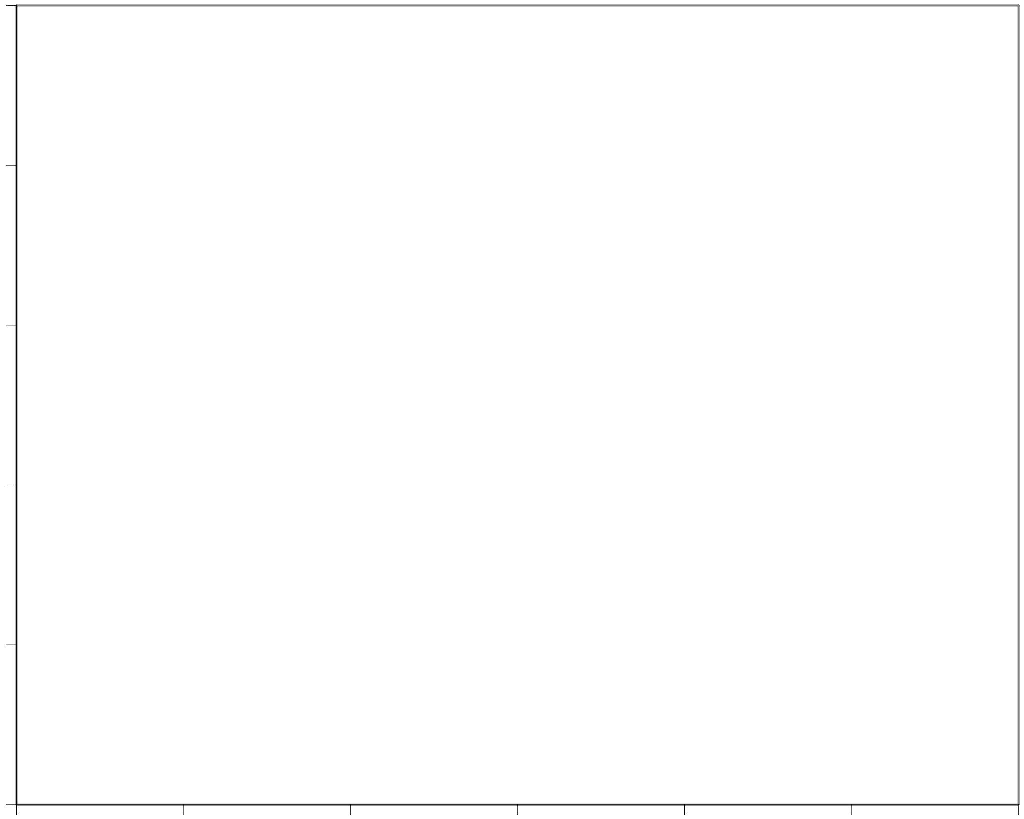
Cure Rate vs. DoC – Isos



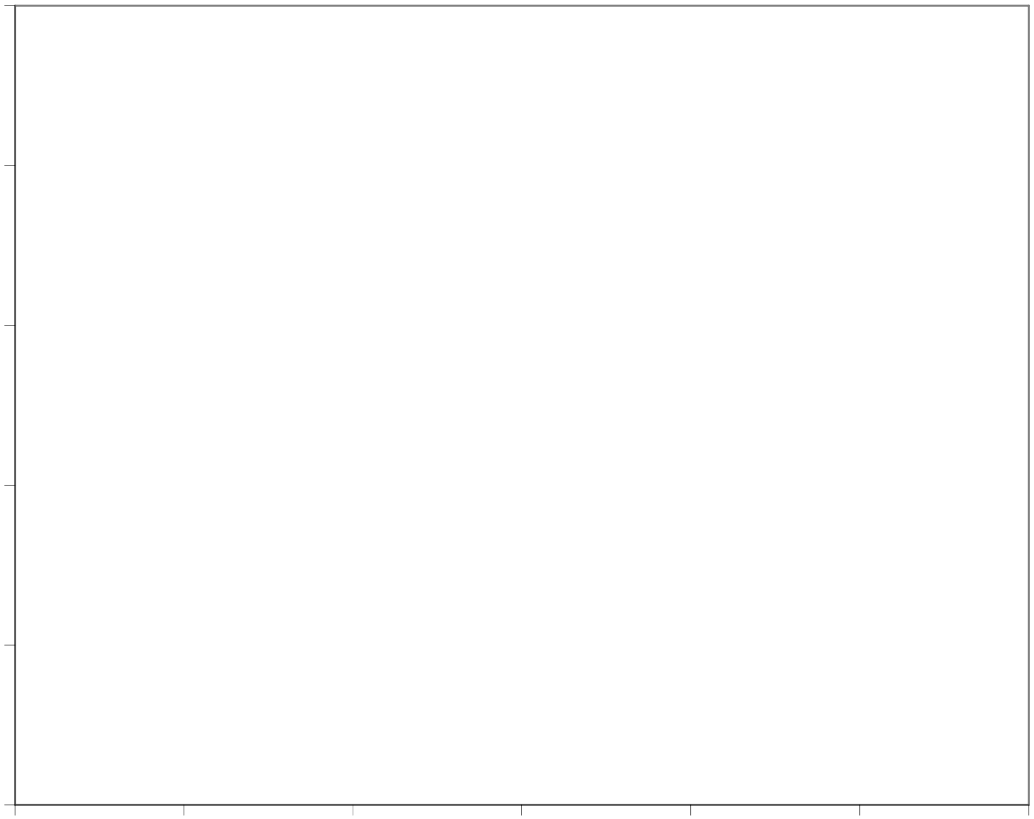
Heat Flow Response – 1cpm

DoC and Cure Rate – 1cpm

Heat Flow Response – 2cpm-1



DoC and Cure Rate – 2cpm-1



Heat Flow Response – 3cpm

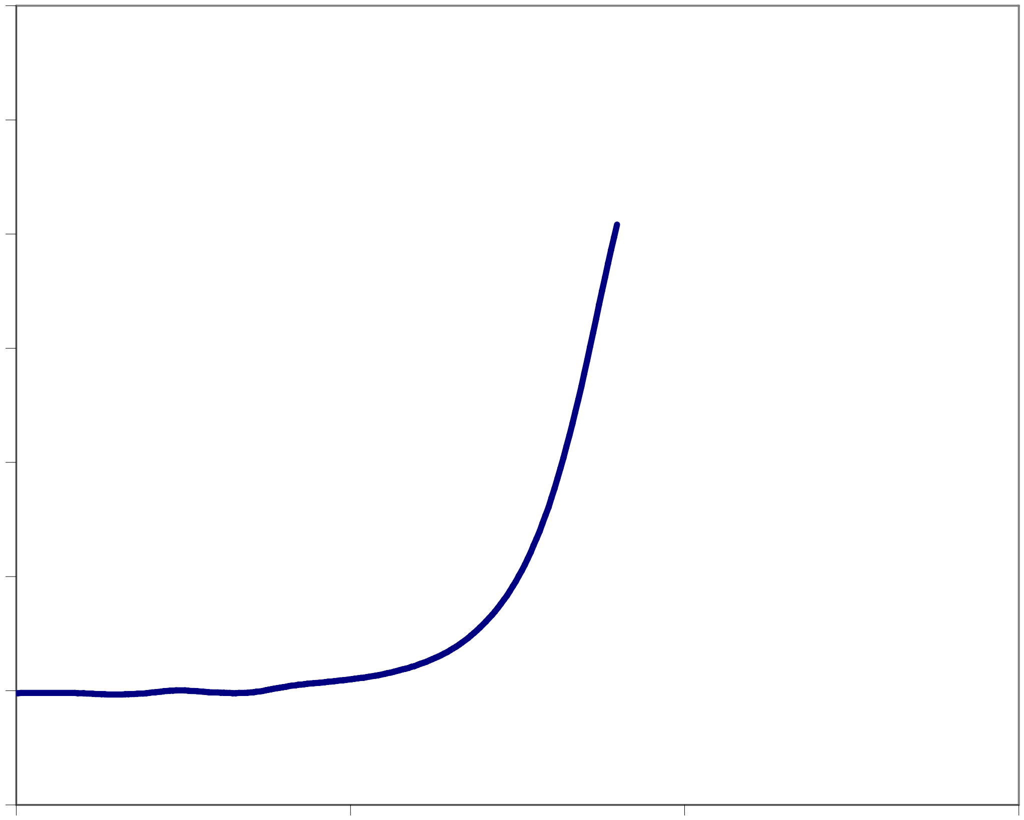
DoC and Cure Rate – 3cpm

DoC and Cure Rate – 4cpm

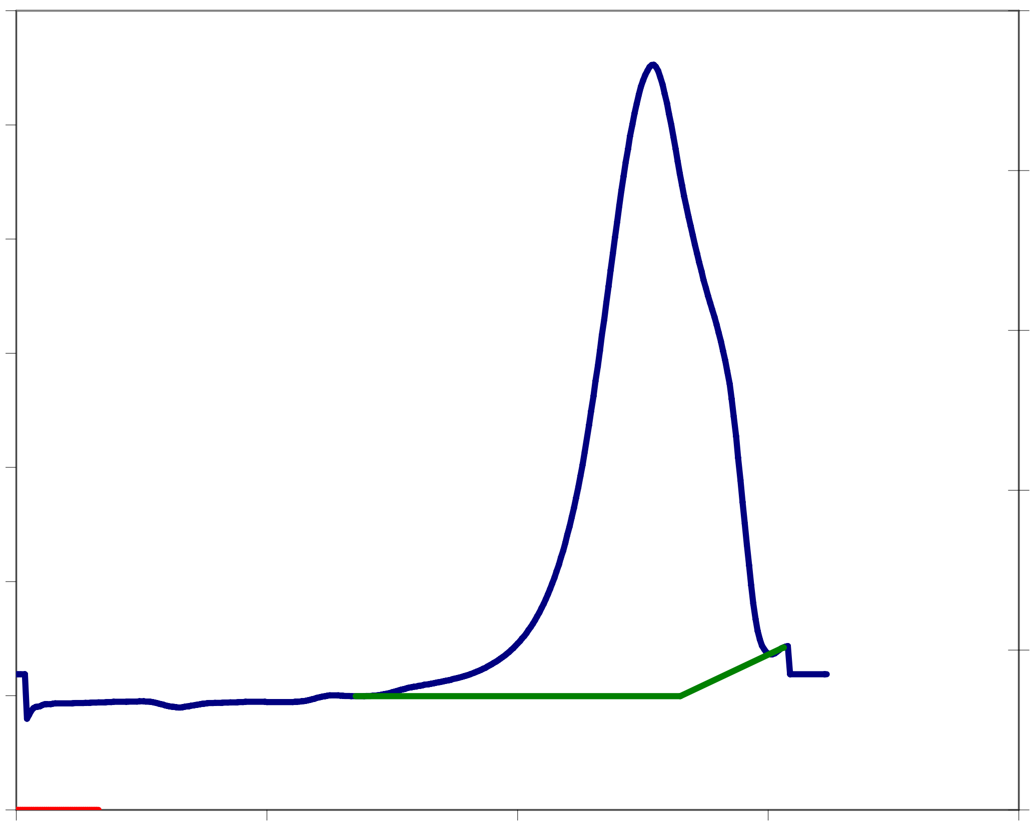
Heat Flow Response – 5cpm

DoC and Cure Rate – 5cpm

Heat Flow Response – 6cpm



Heat Flow Response – 7cpm



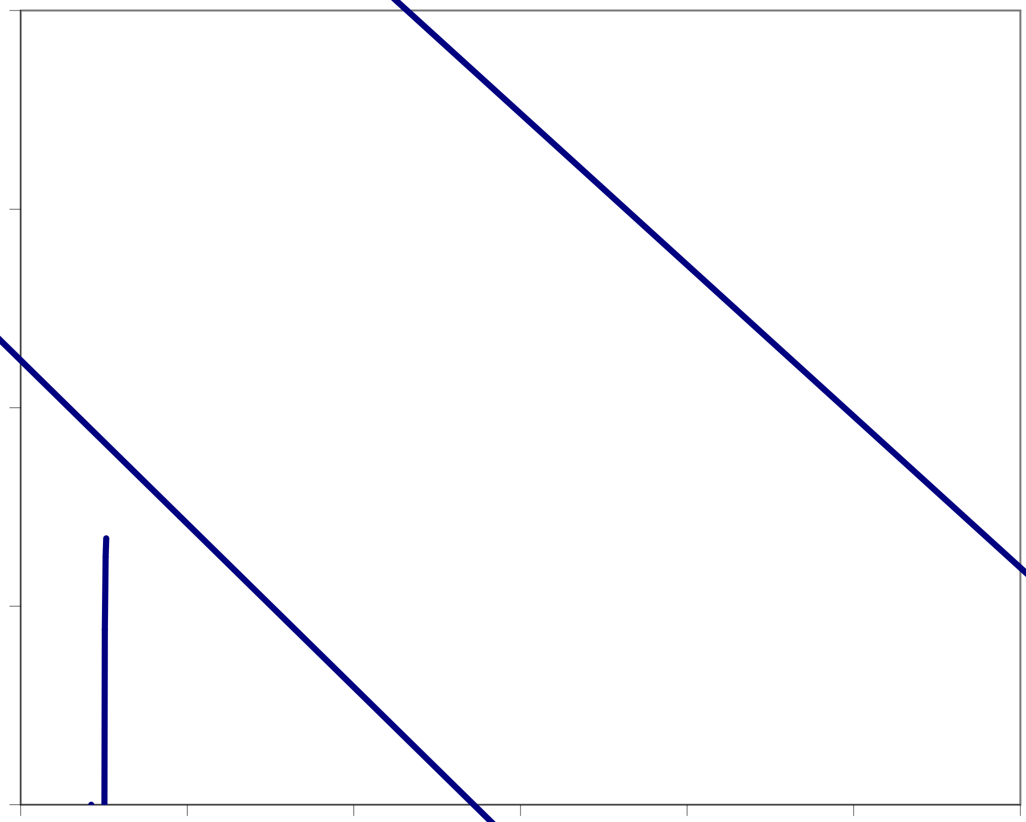
Heat Flow Response – 9cpm

DoC and Cure Rate – 9cpm

DoC and Cure Rate – 10cpm

Heat Flow Response – 120ºC

Heat Flow Response – 130ºC



Heat Flow Response – 140ºC

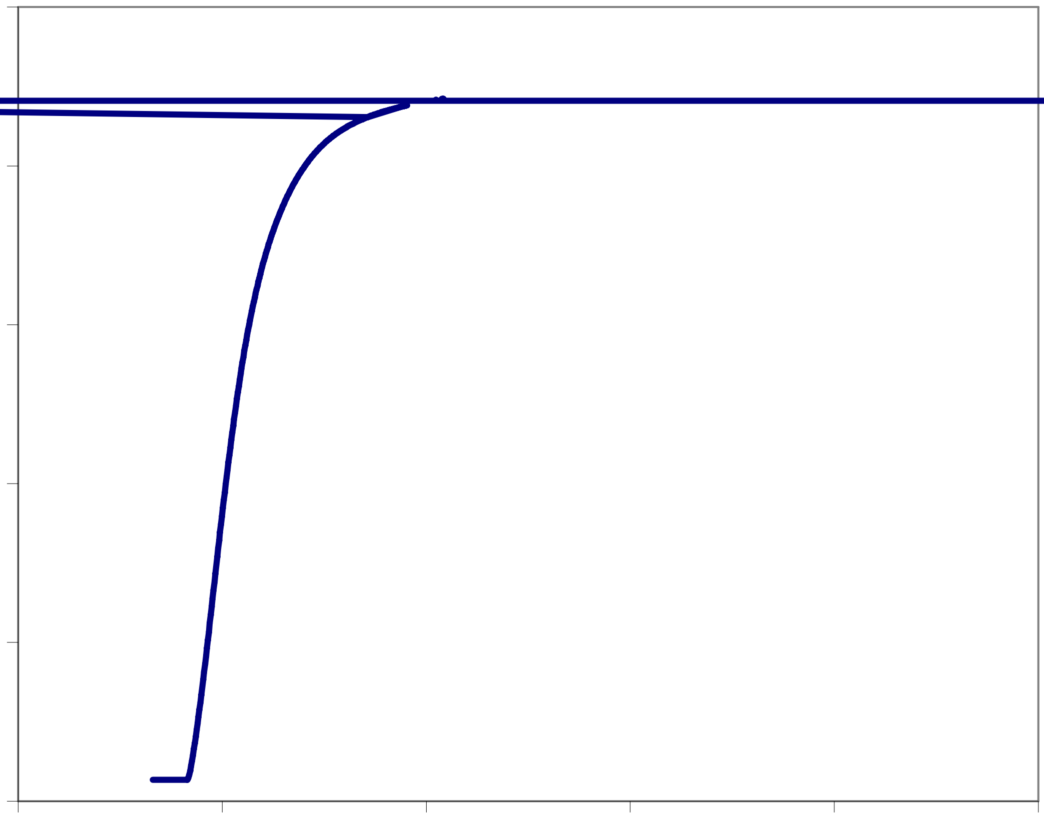
DoC and Cure Rate – 140ºC

Heat Flow Response – 150ºC

Heat Flow Response – 160ºC

DoC and Cure Rate – 160ºC

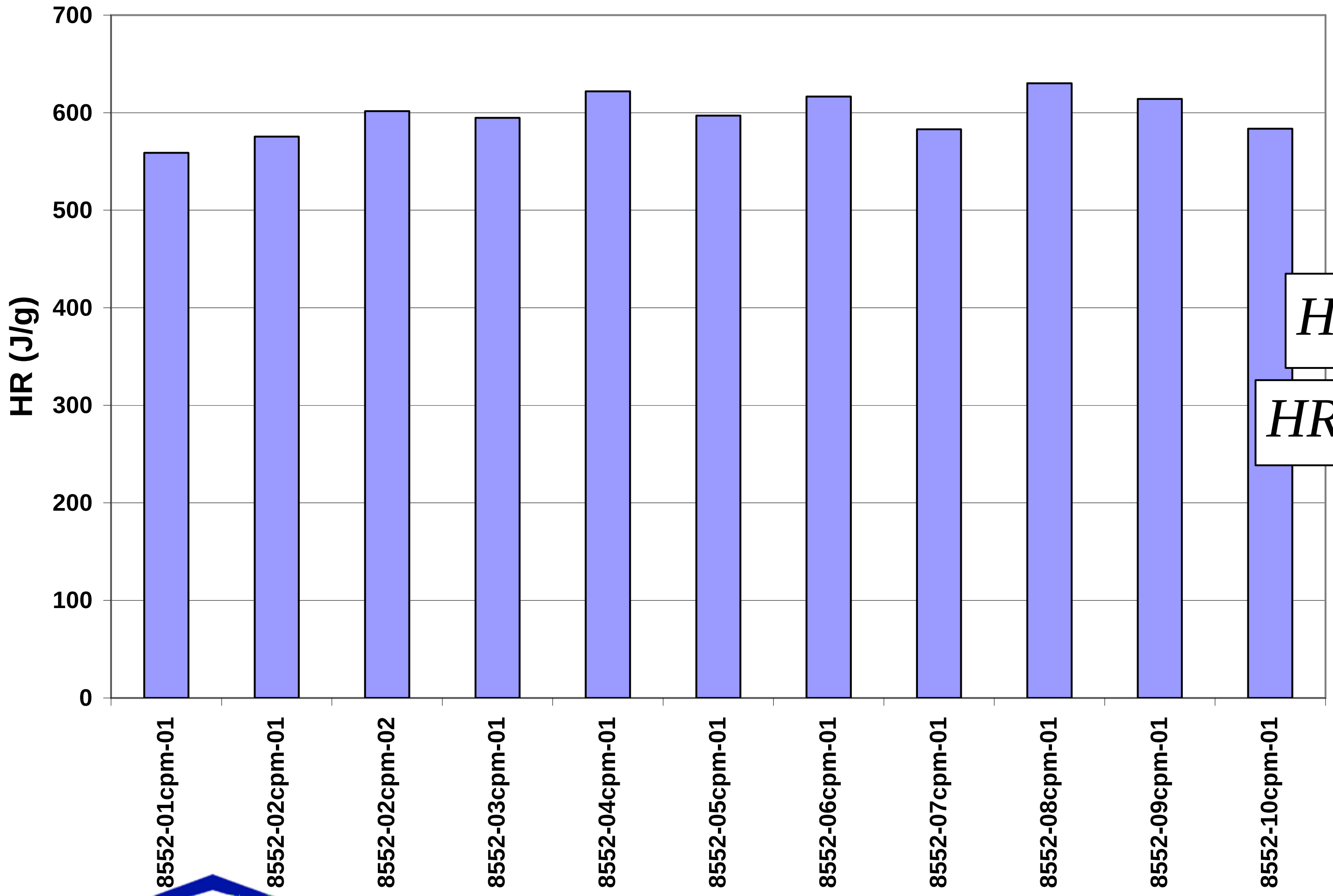
DoC and Cure Rate – 180ºC-2



DoC and Cure Rate – 190ºC

[redacted]

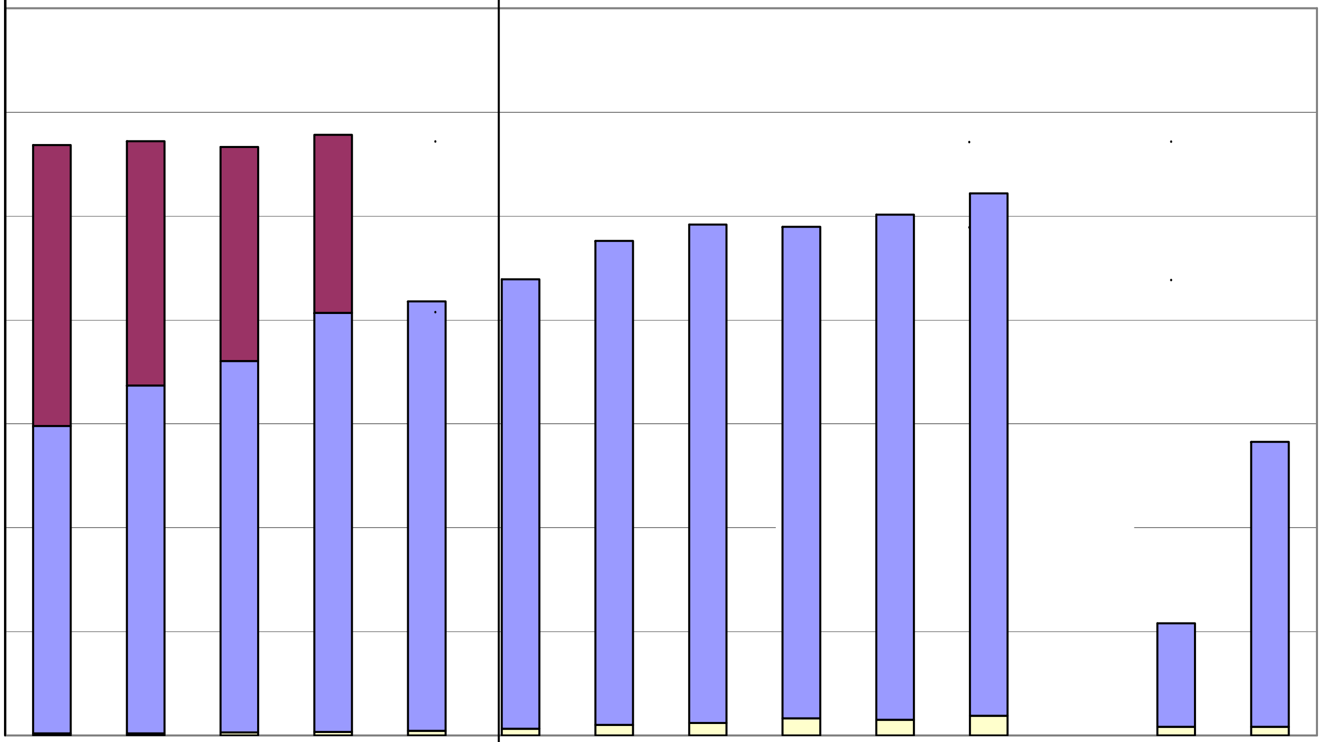
Total Heat of Reaction - Dyns

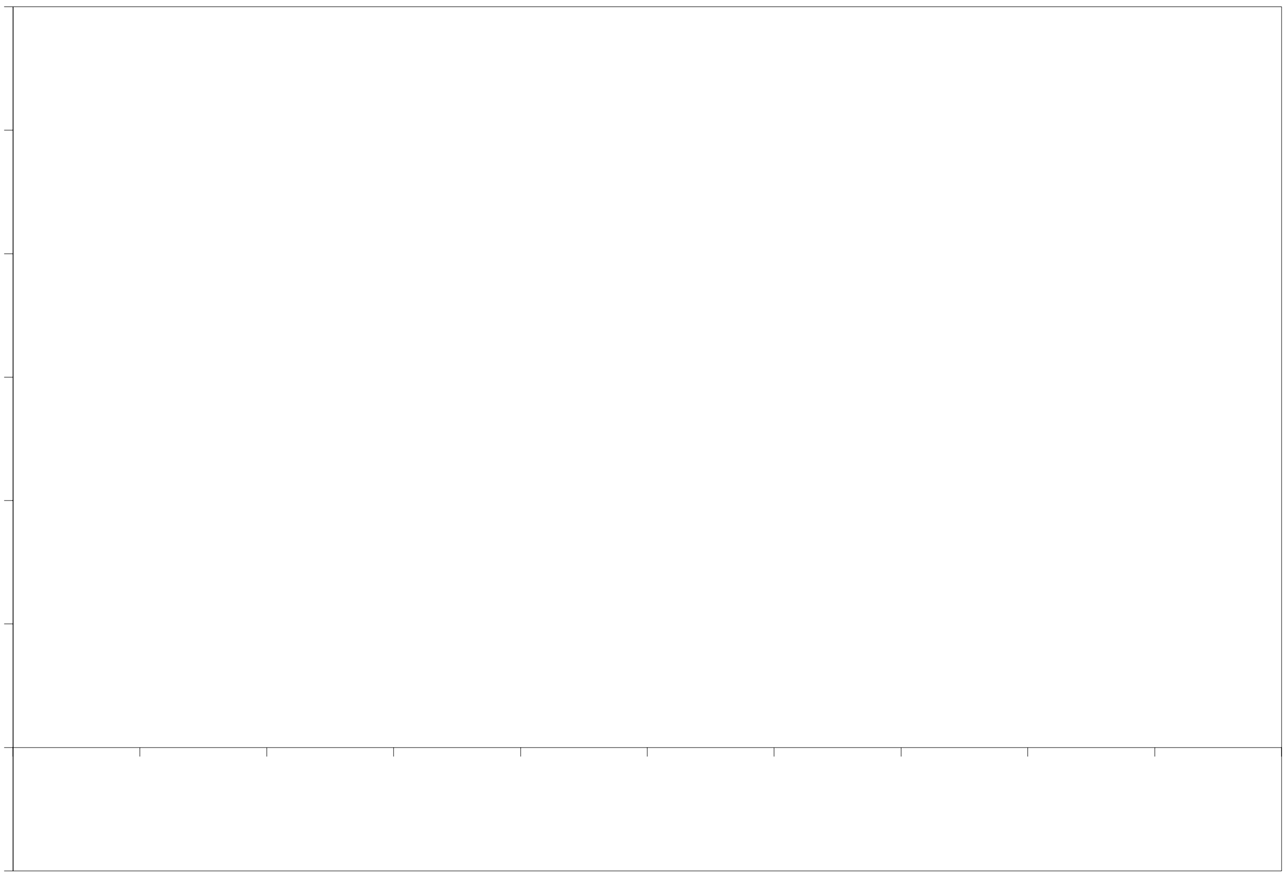


$$HR_{Dyn}^{avg} = 598 \quad \text{J/g}$$

$$HR_{Model} = 600 \quad \text{J/g}$$

Total Heat of Reaction - Isos





Cure Kinetics Model

Chemical Reaction:

1 k 2

where

Cure Kinetics Model

Diffusion Component:

$$\dot{x}_d = K_{d0} e^{\frac{-B}{f}} F_d(x) \longrightarrow \dot{x}_d = 4.0 \times e^{\frac{-0.21}{f}}$$

where[1] $f = a(T - T_g) + b$

with $a = 4.8 \times 10^{-4}$

and

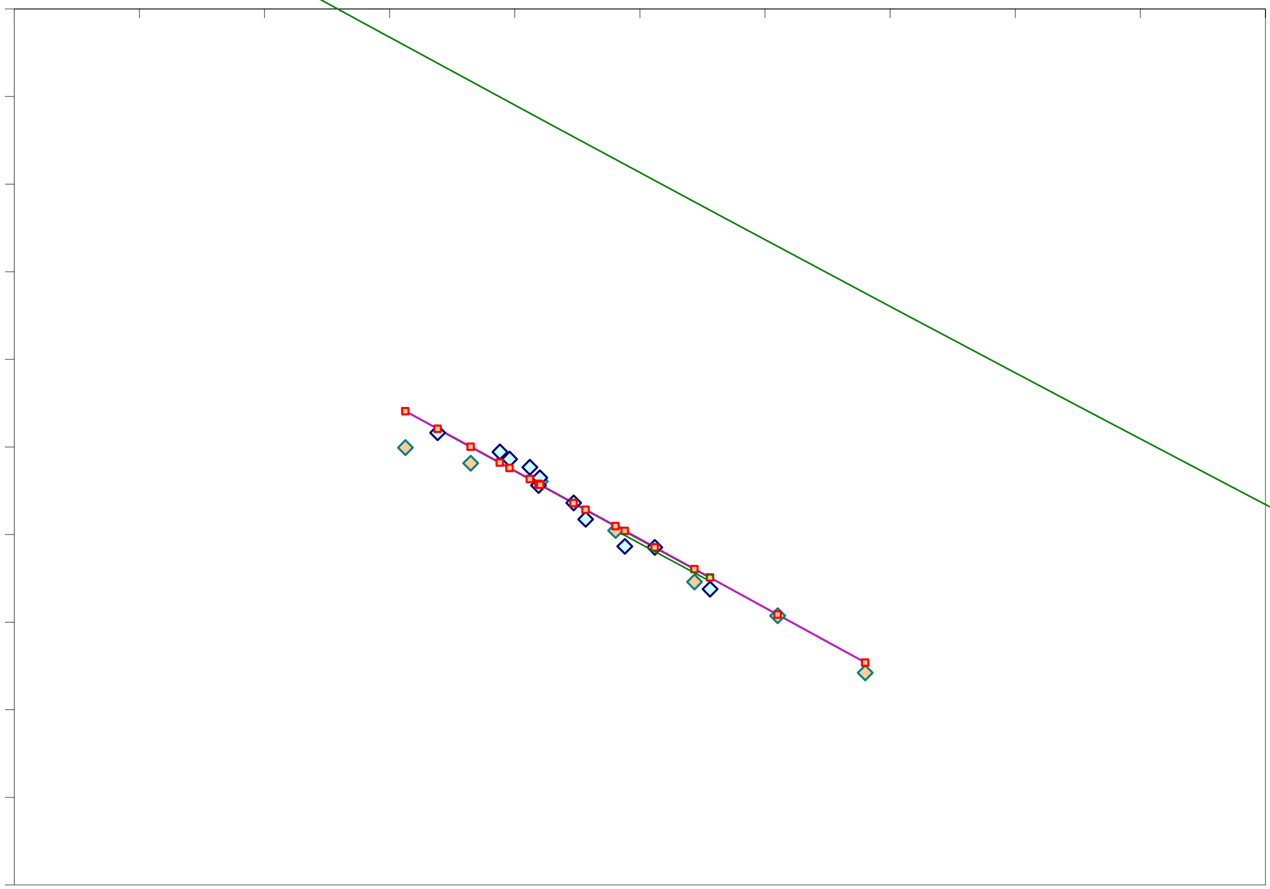
$$b = \begin{cases} 0.021 & T_g < 120°C \\ \text{Linear bw } 0.021 \text{ and } 0.031 & 120°C < T_g < 195°C \\ 0.031 & T_g > 195°C \end{cases}$$

[1] [redacted]ars2.f90)

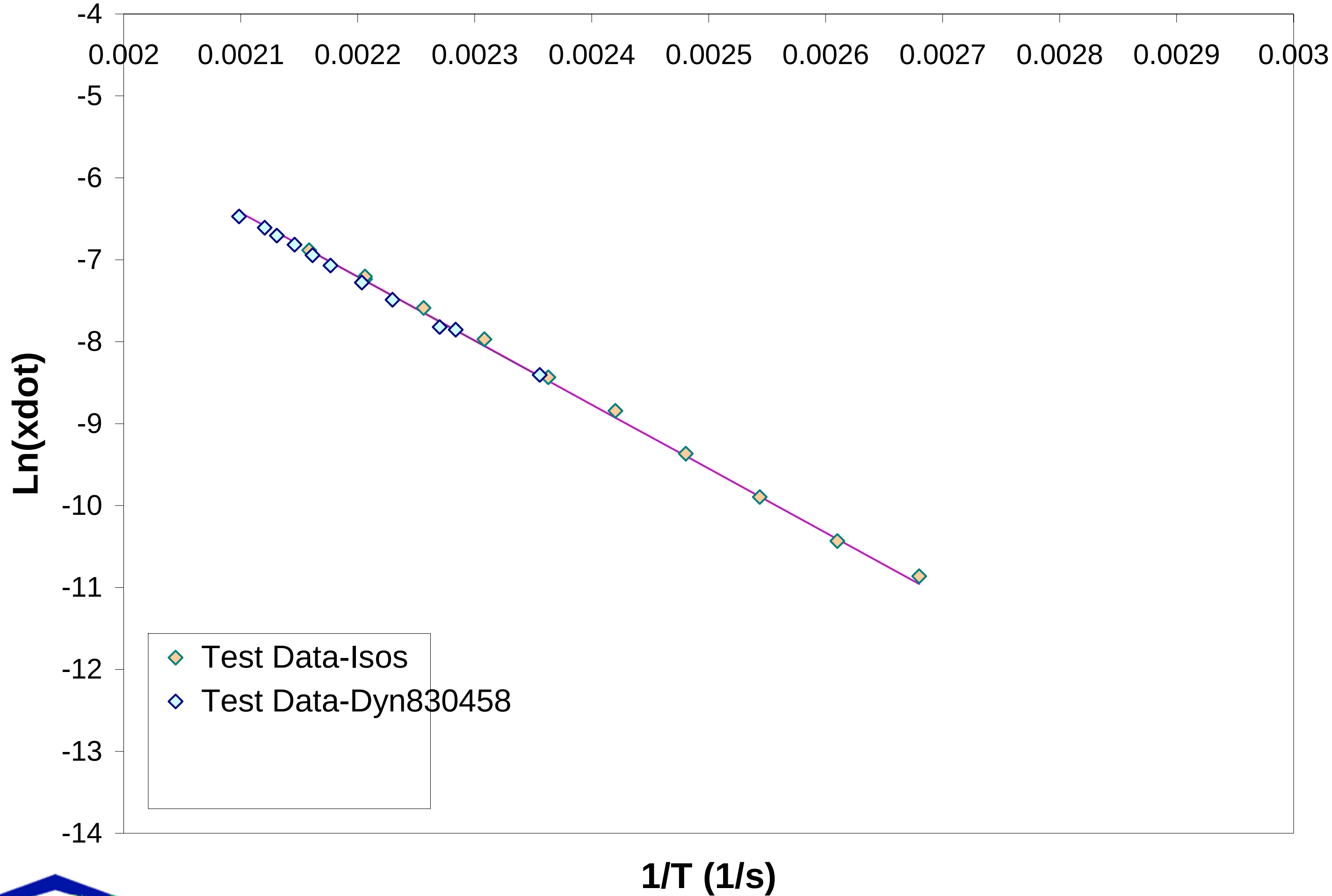
Cure Kinetics Model

$$\dot{x} = \left(\frac{1}{\dot{x}_k} + \frac{1}{\dot{x}_d} \right)^{-1}$$

Ln(xdot)-1/T

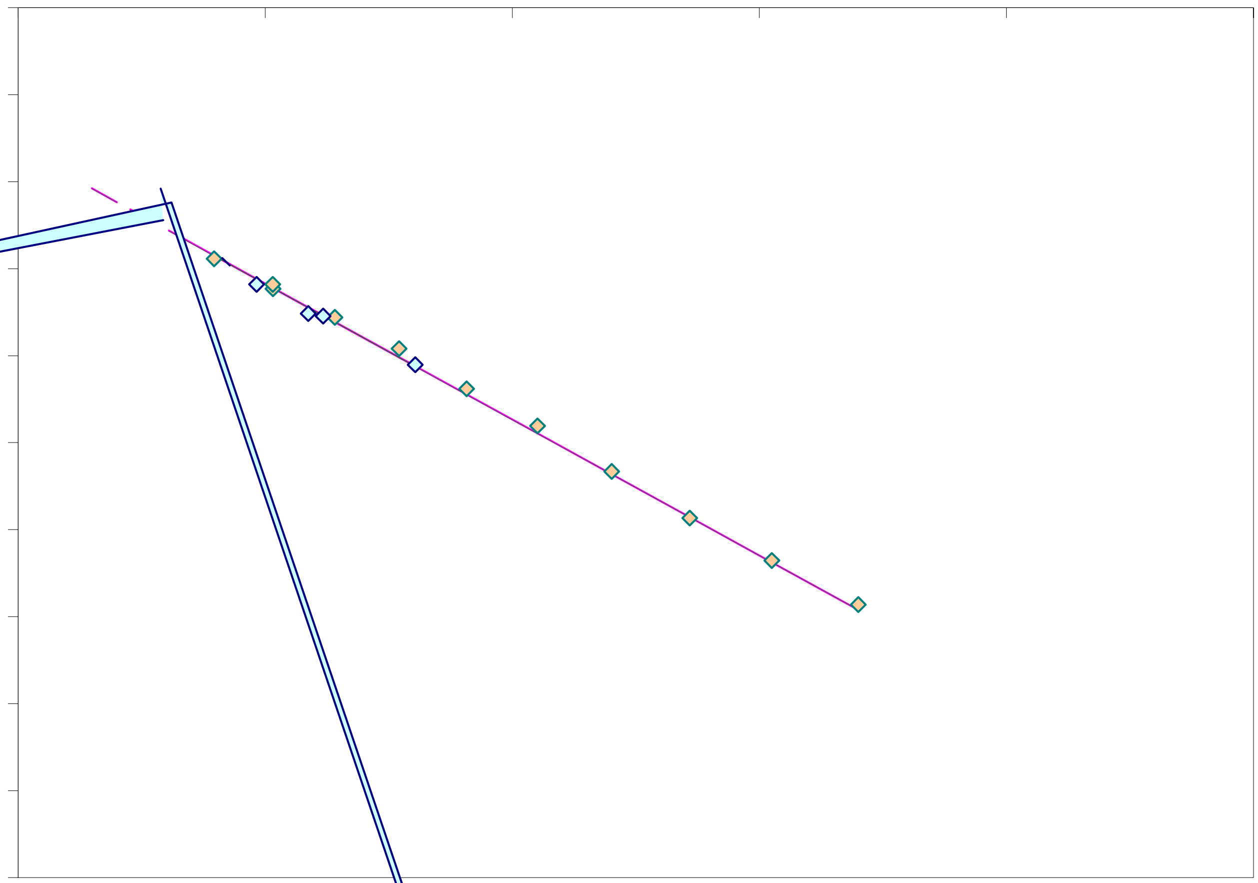


Ln(xdot)-1/T

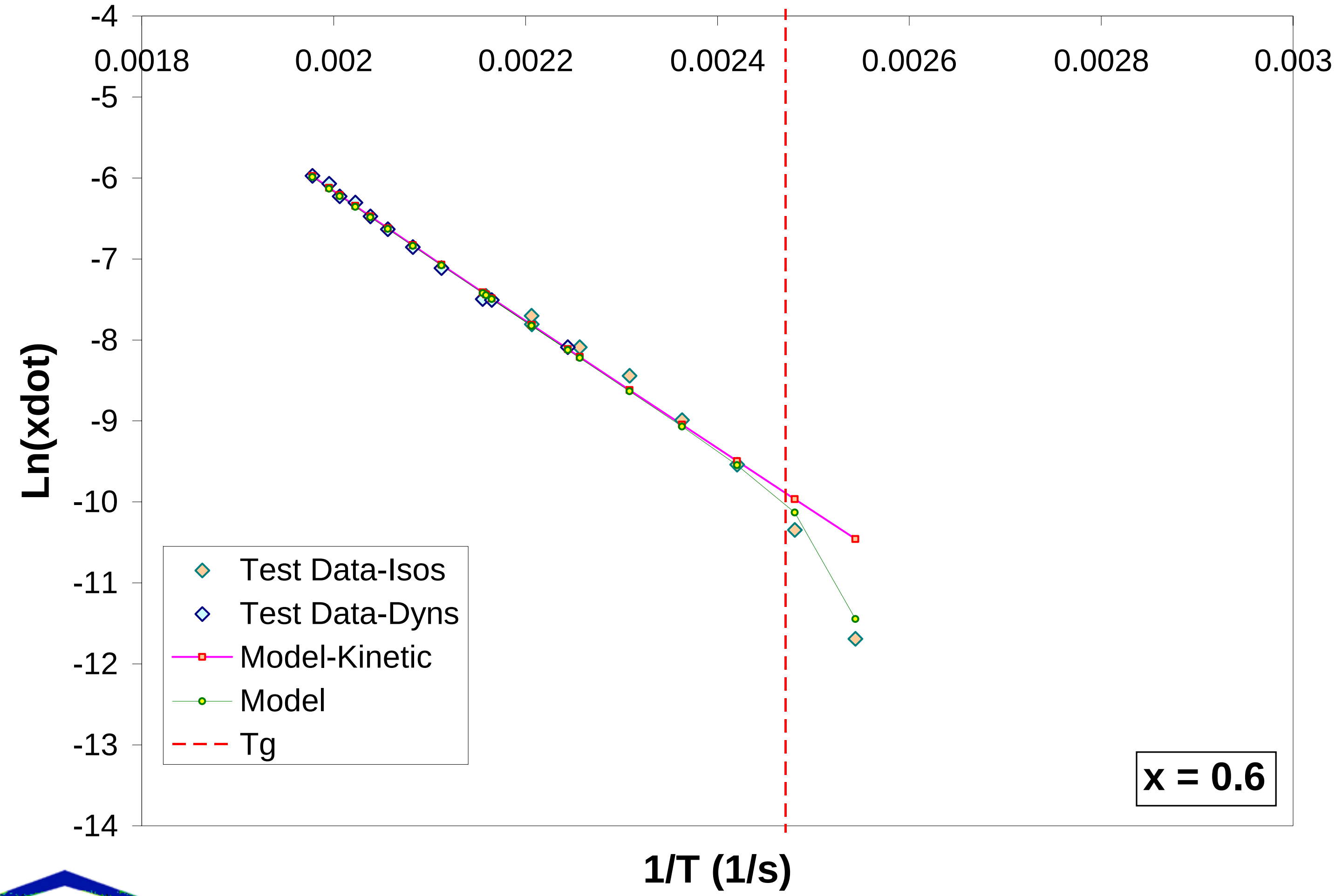


-4
-5
-6
-7
-8
-9
-10
-11
-12
-13
-14
0.002
0.0021
0.0022
0.0023
0.0024
0.0025
0.0026
0.0027
0.0028
0.0029
0.003
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyn830458

Ln(xdot)-1/T



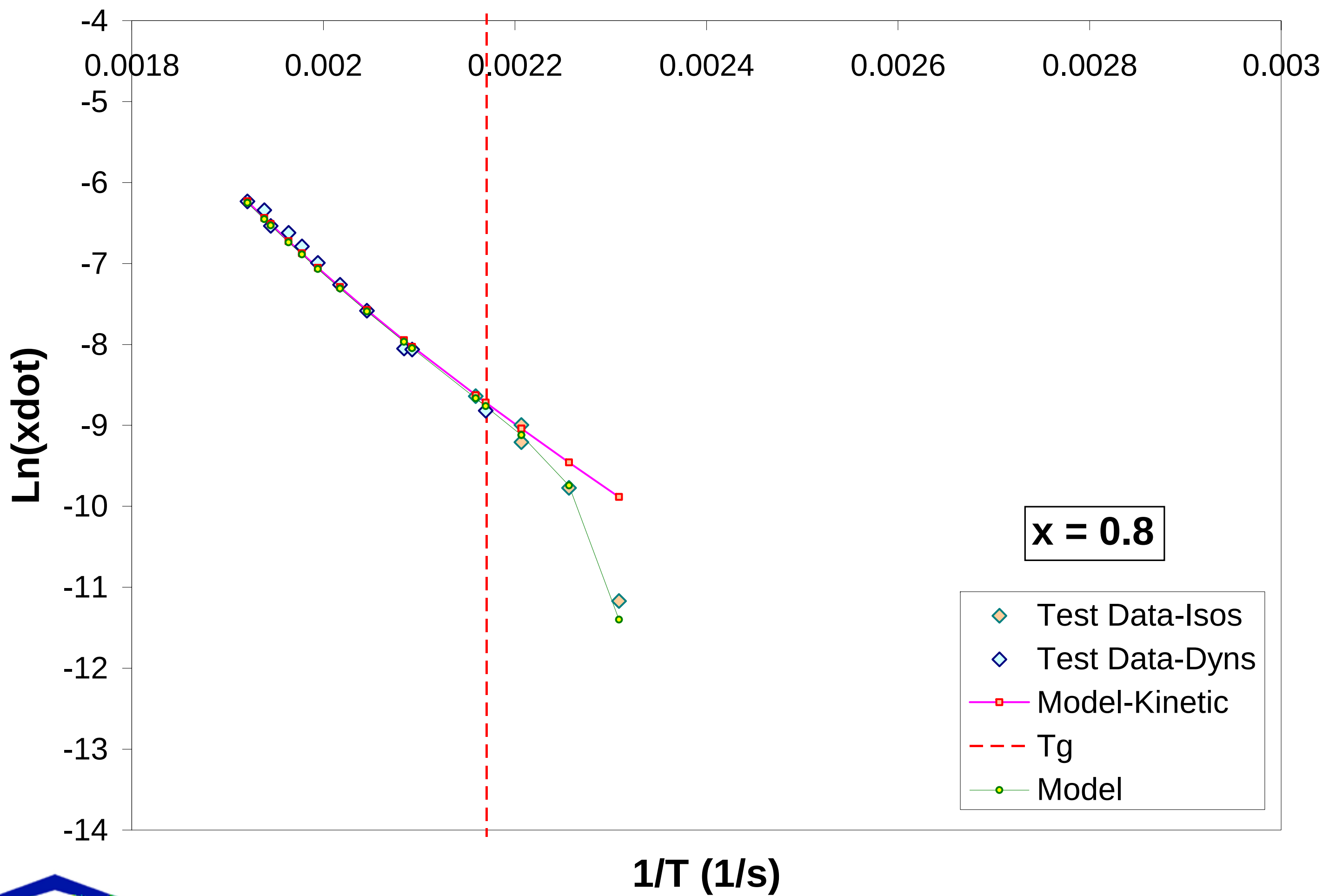
Ln(xdot)-1/T



-4
-5
-6
-7
-8
-9
-10
-11
-12
-13
-14
0.0018
0.002
0.0022
0.0024
0.0026
0.0028
0.003
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.6

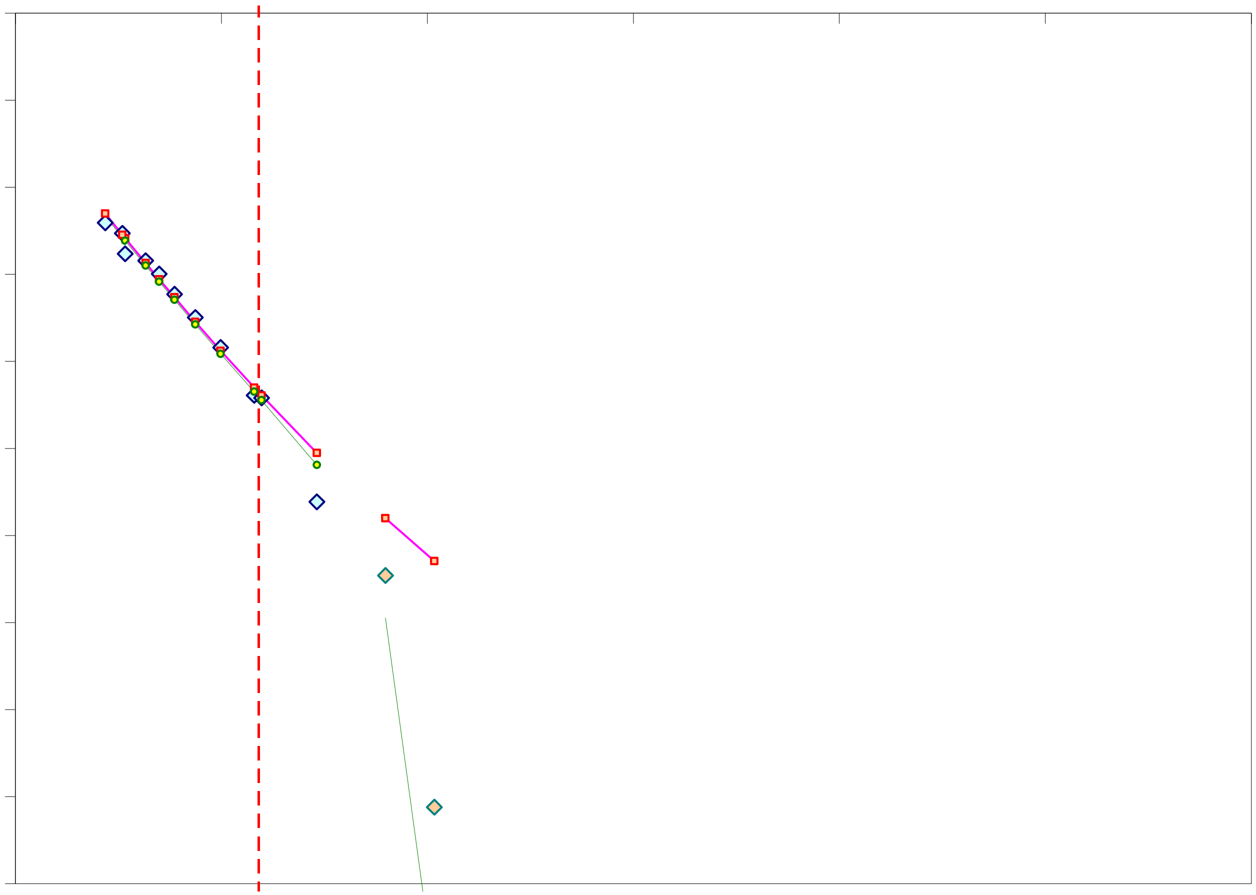
Ln(xdot)-1/T

Ln(xdot)-1/T

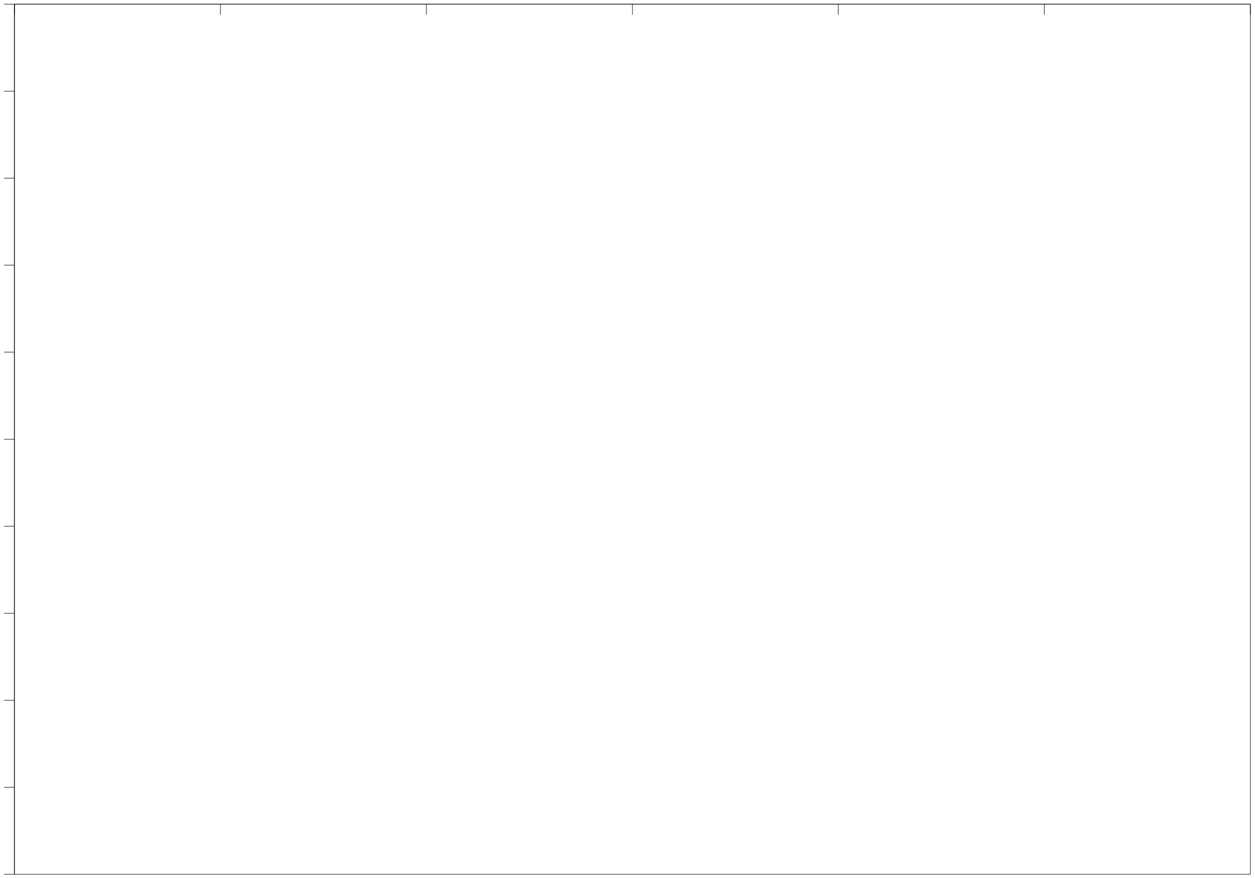


x = 0.8
Ln(xdot)
1/T (1/s)
-4
-5
-6
-7
-8
-9
-10
-11
-12
-13
-14
0.0018
0.002
0.0022
0.0024
0.0026
0.0028
0.003
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Tg
Model

Ln(xdot)-1/T



Ln(xdot)-1/T



Model Parameters

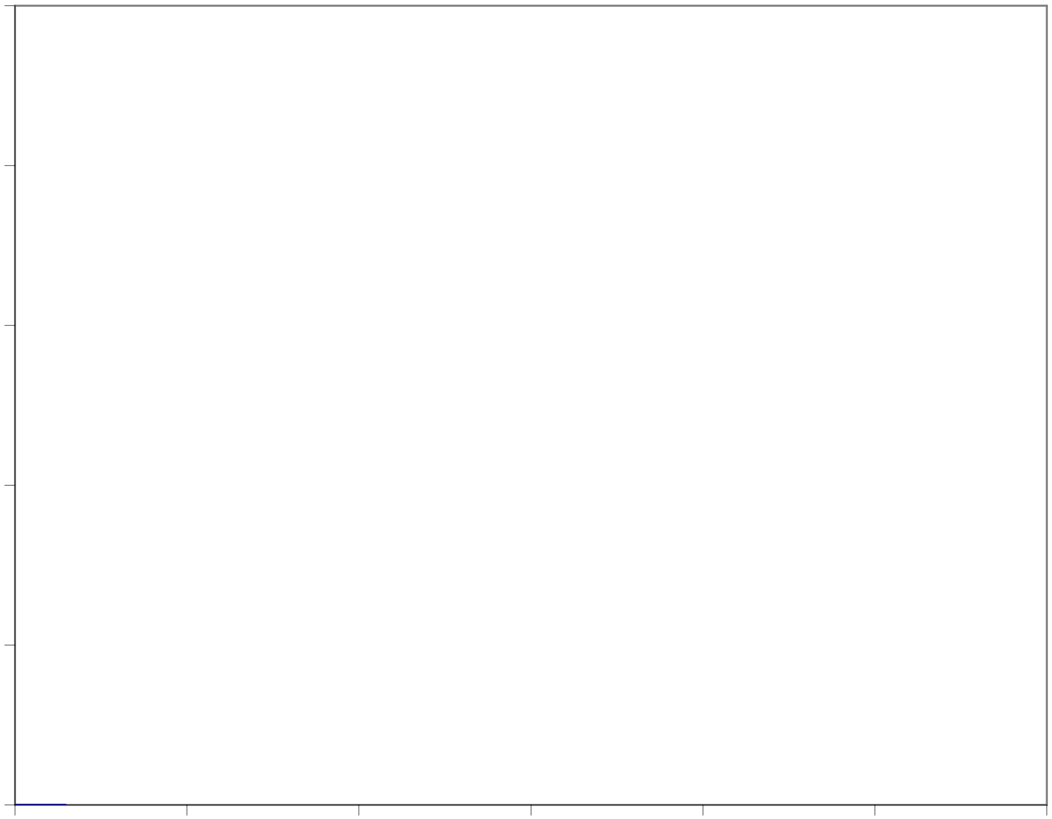
Parameter	Reaction 1	Reaction 2
(1/s)	153,900.5	3.963E+11
(J/mol)	64,929.5	133,168.3
	2.347	1.029
	1.00	1.00
	0.00	0.00
	1.00	1.00
	0.1594	0.00
	1.413	5.586

Parameter	Value (unit)
	-7 (ºC)
	250 (ºC)
	0.78

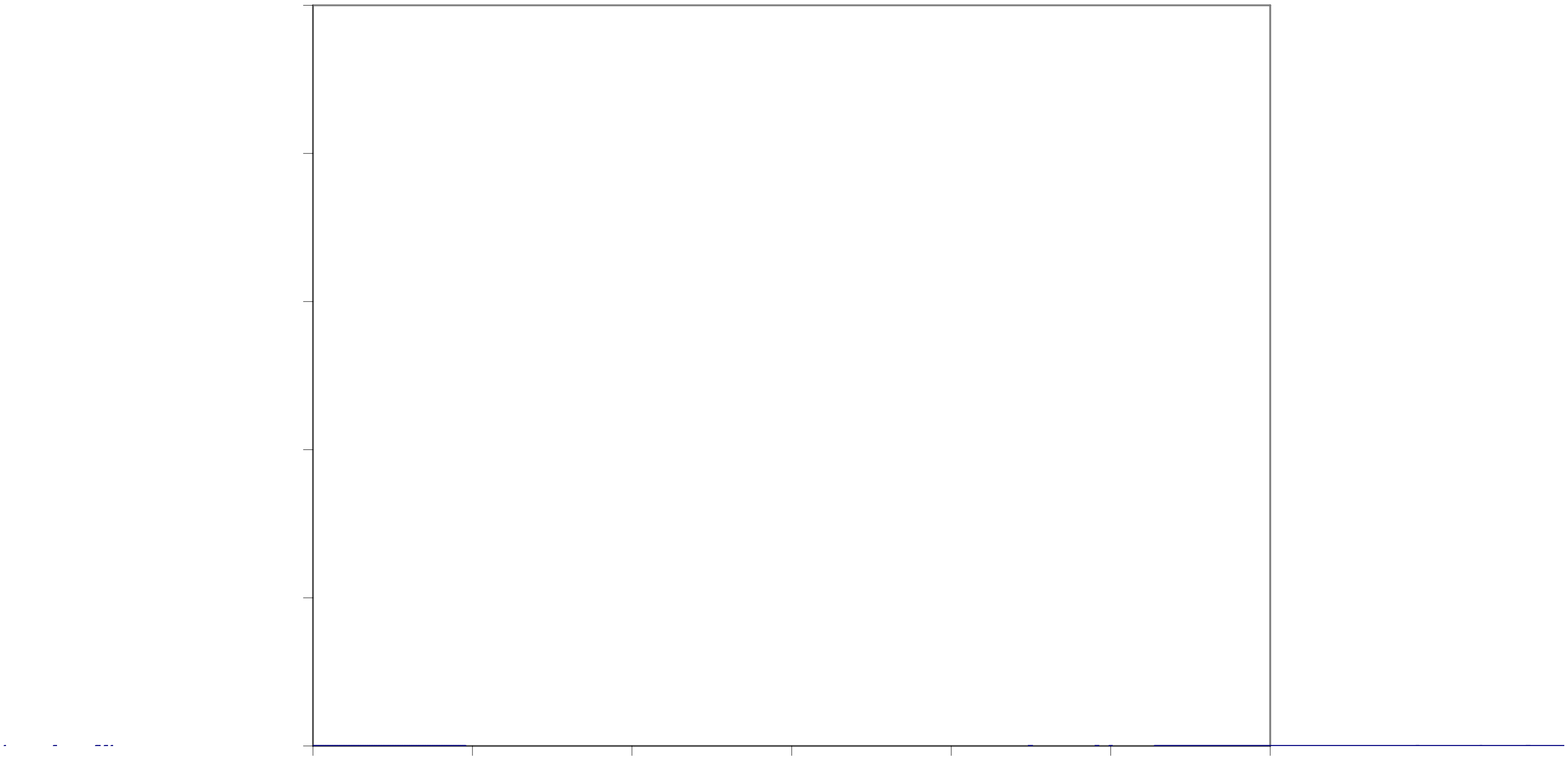
Parameter	Value (unit)
	4.0 (1/s)
	0.21
	4.8E-04 (1/ºC)
	4.8E-04 (1/ºC)
	0 (ºC)
	100 (ºC)
	0.021
	0.031
	120 (ºC)
	195 (ºC)

Dynamic Tests – 1cpm

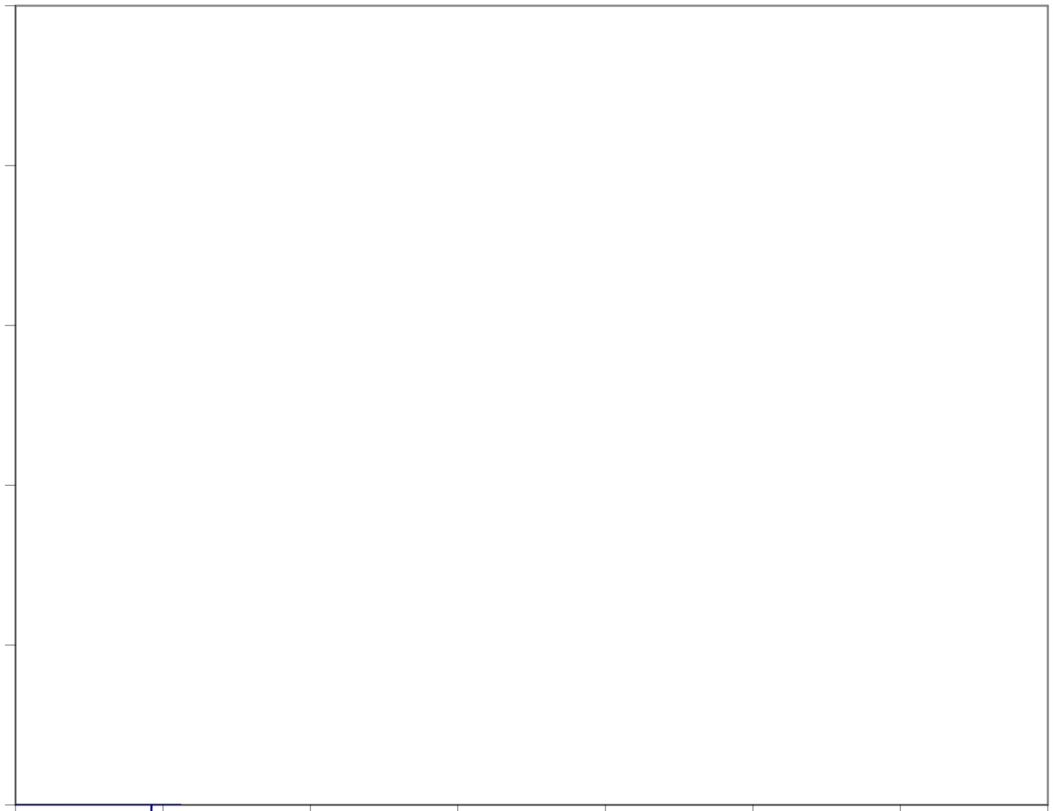
Dynamic Tests – 2cpm-2



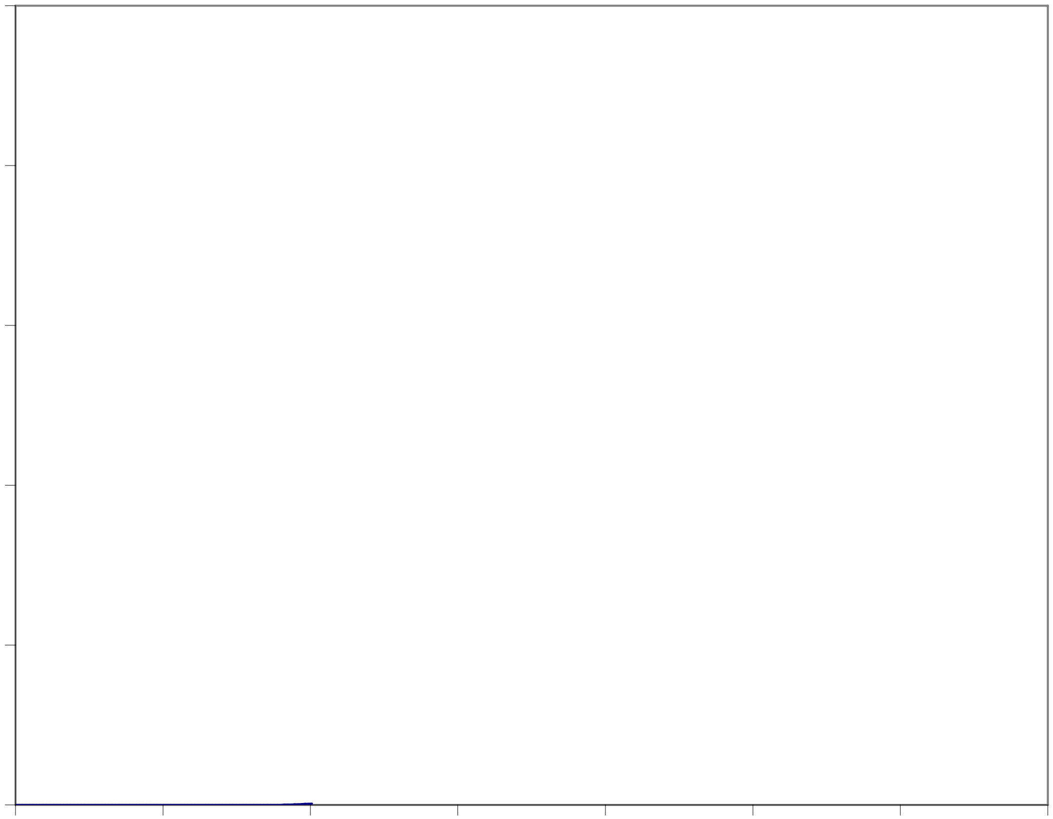
Dynamic Tests – 3cpm



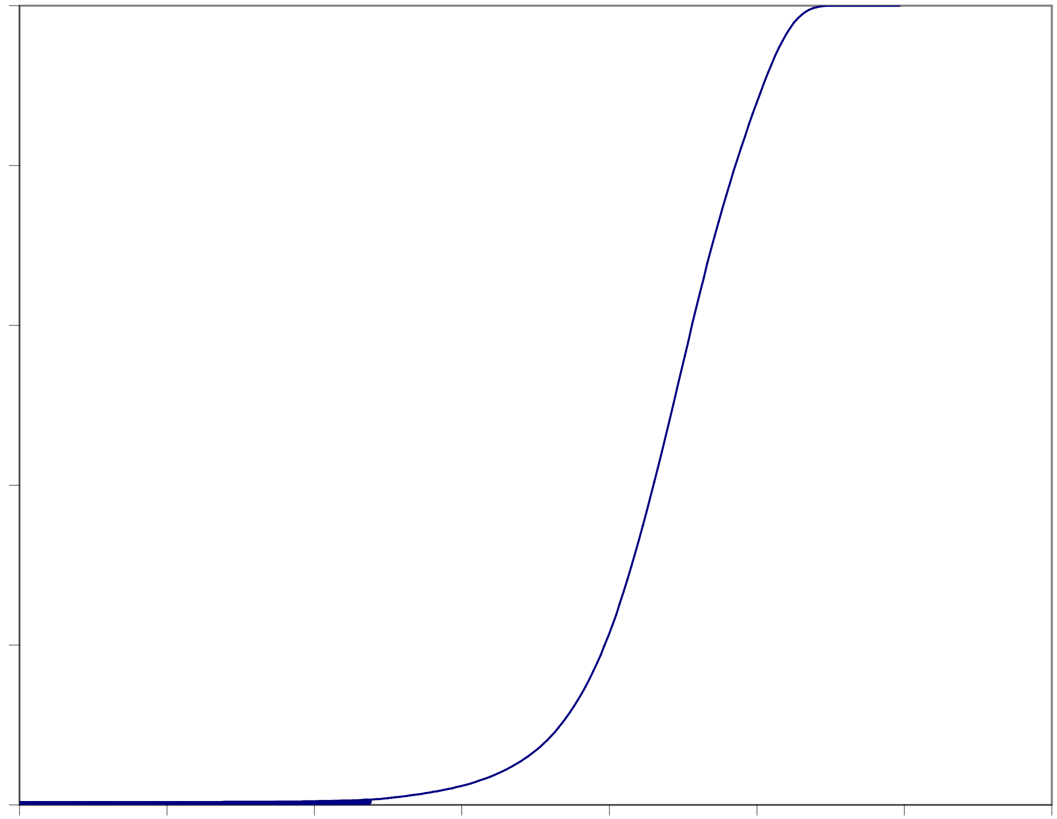
Dynamic Tests – 4cpm



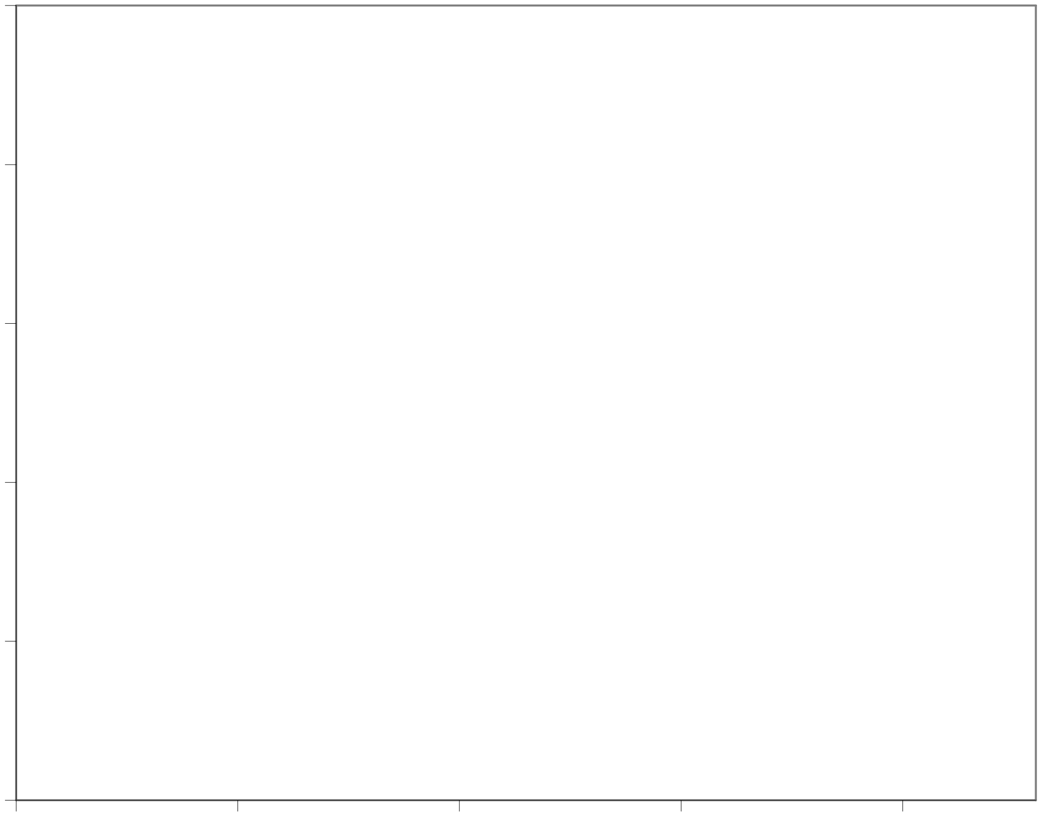
Dynamic Tests – 8cpm



Dynamic Tests – 9cpm



Isothermal Tests - 100ºC



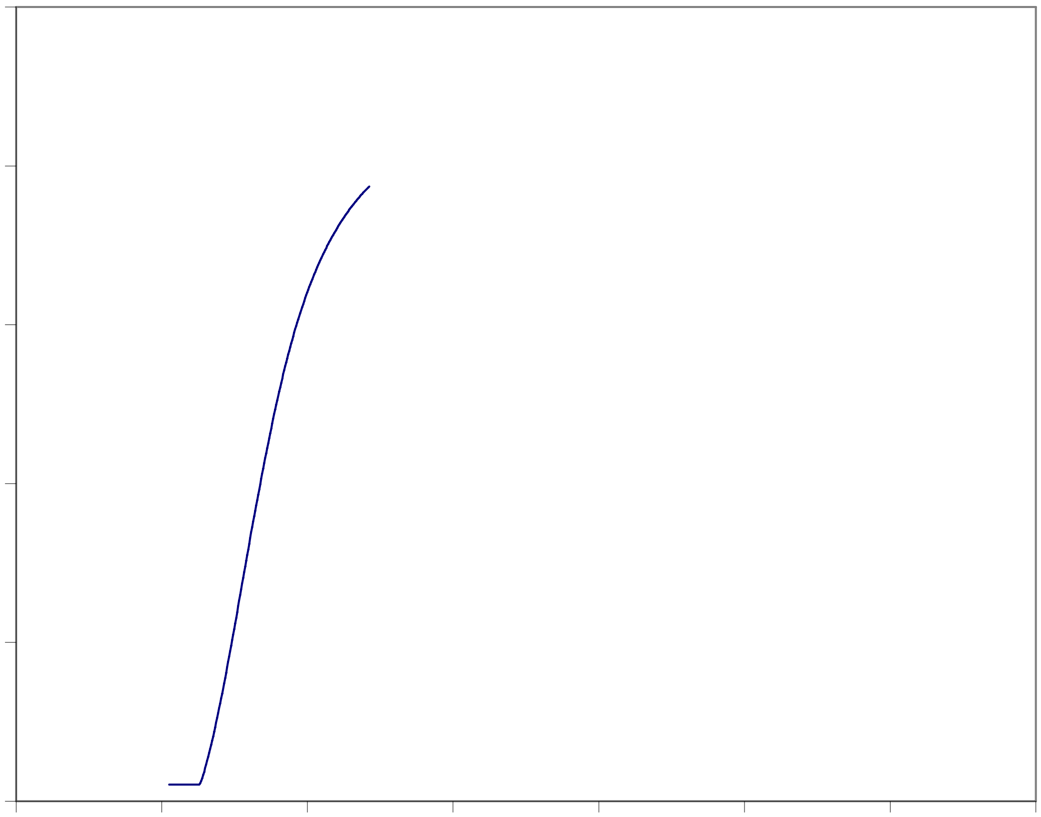
Isothermal Tests - 120ºC

Isothermal Tests - 130ºC

Isothermal Tests - 140ºC

Isothermal Tests - 150ºC

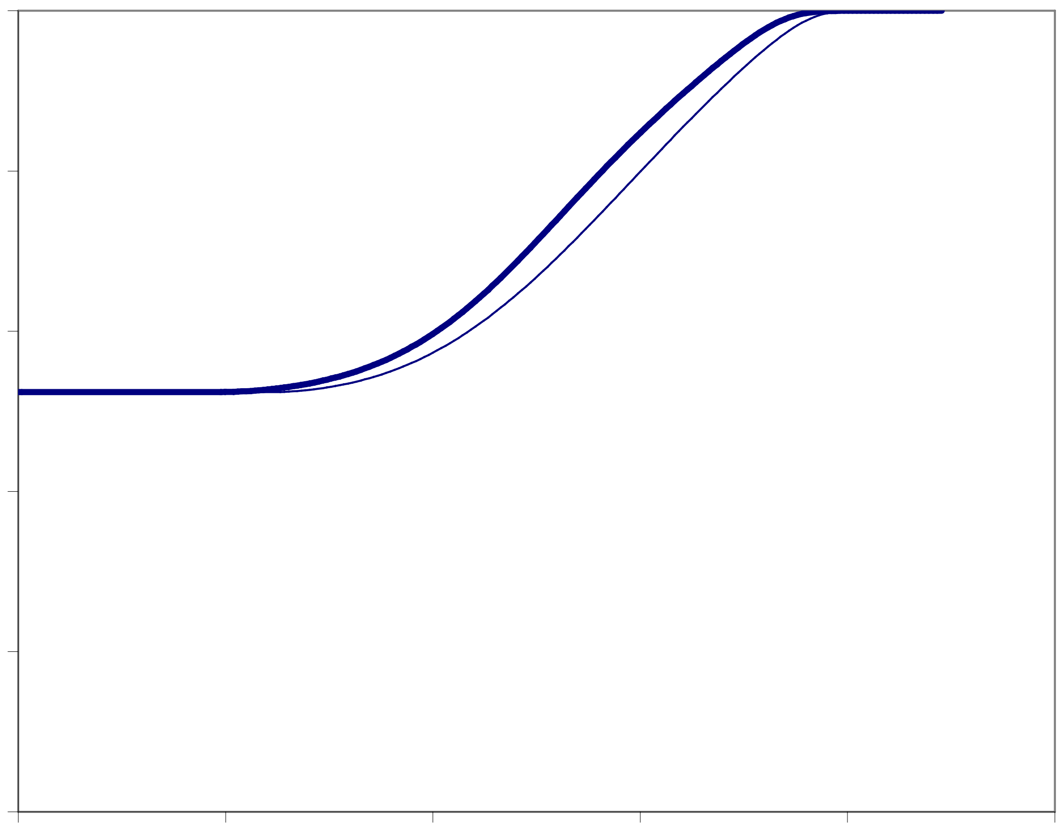
Isothermal Tests - 170ºC

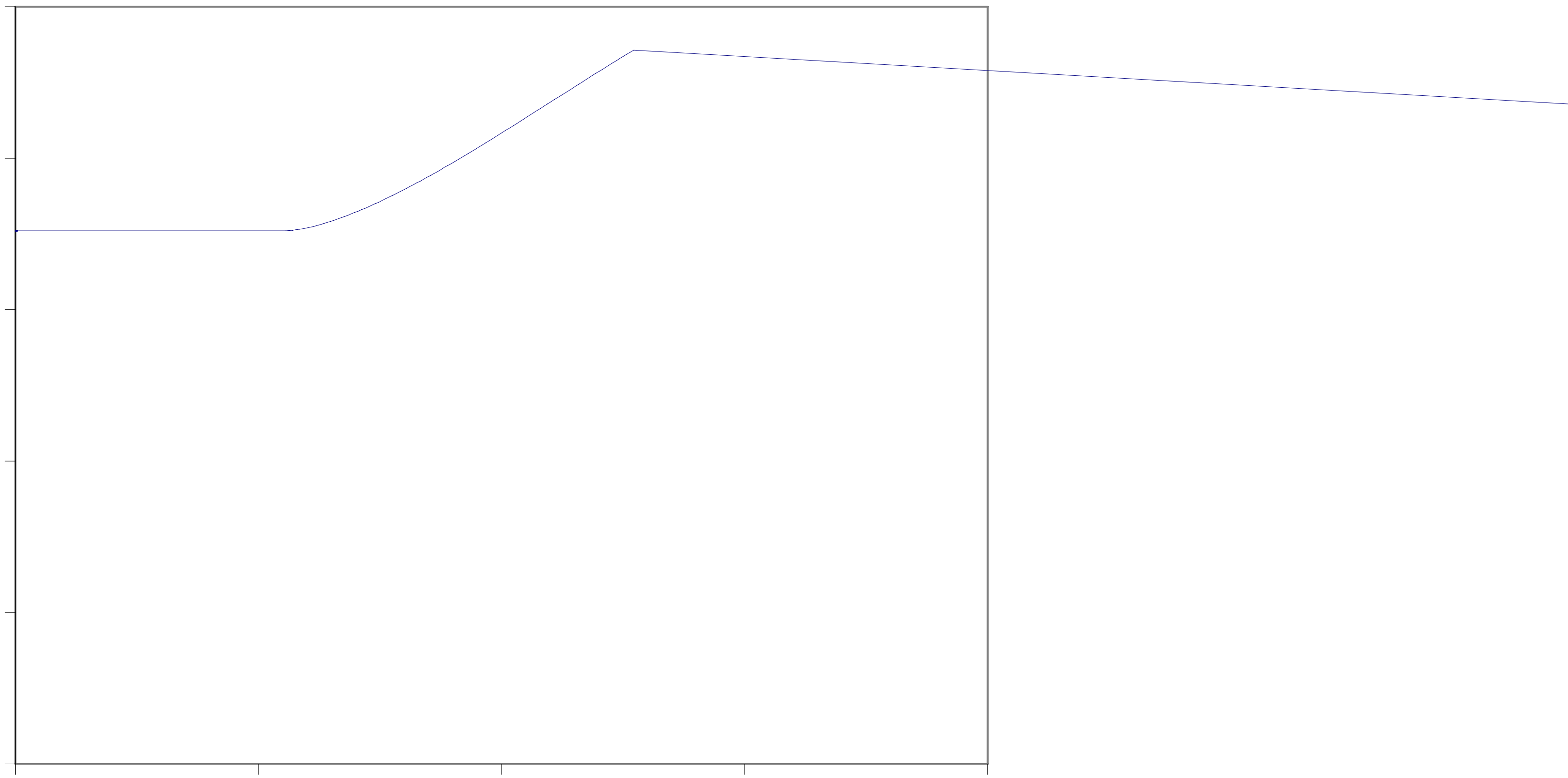


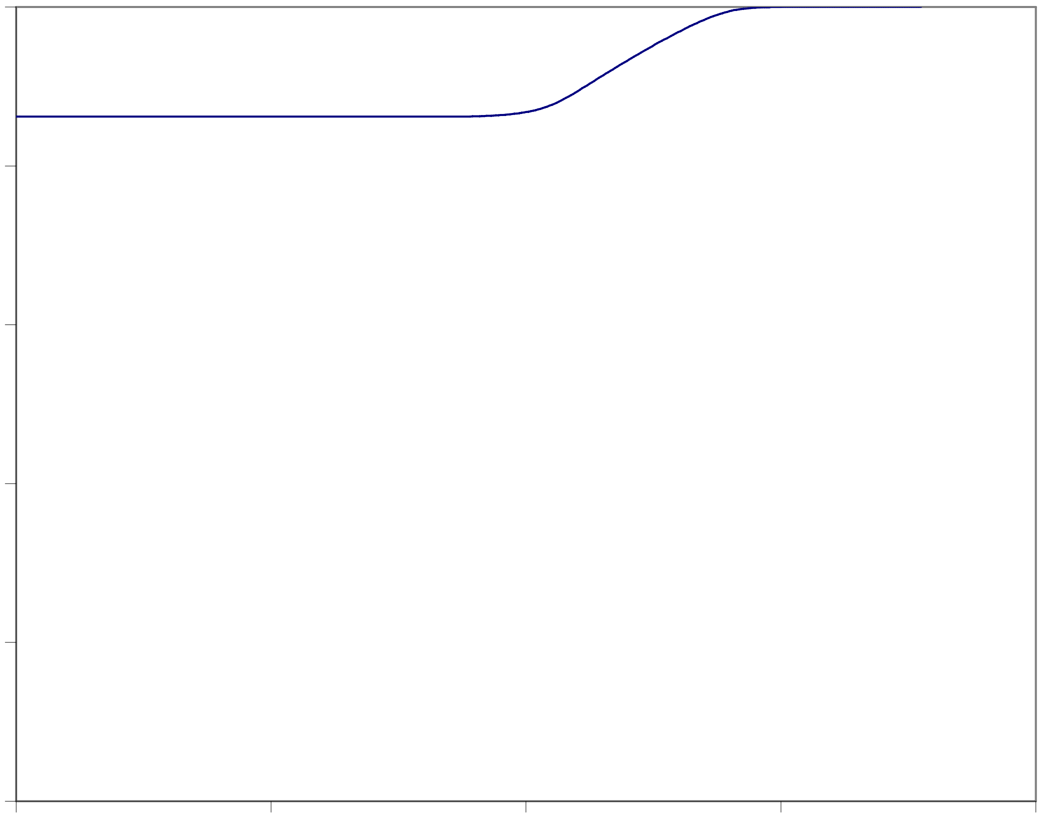
Isothermal Tests - 180ºC-1

Isothermal Tests - 180ºC-2

Isothermal Tests - 190ºC





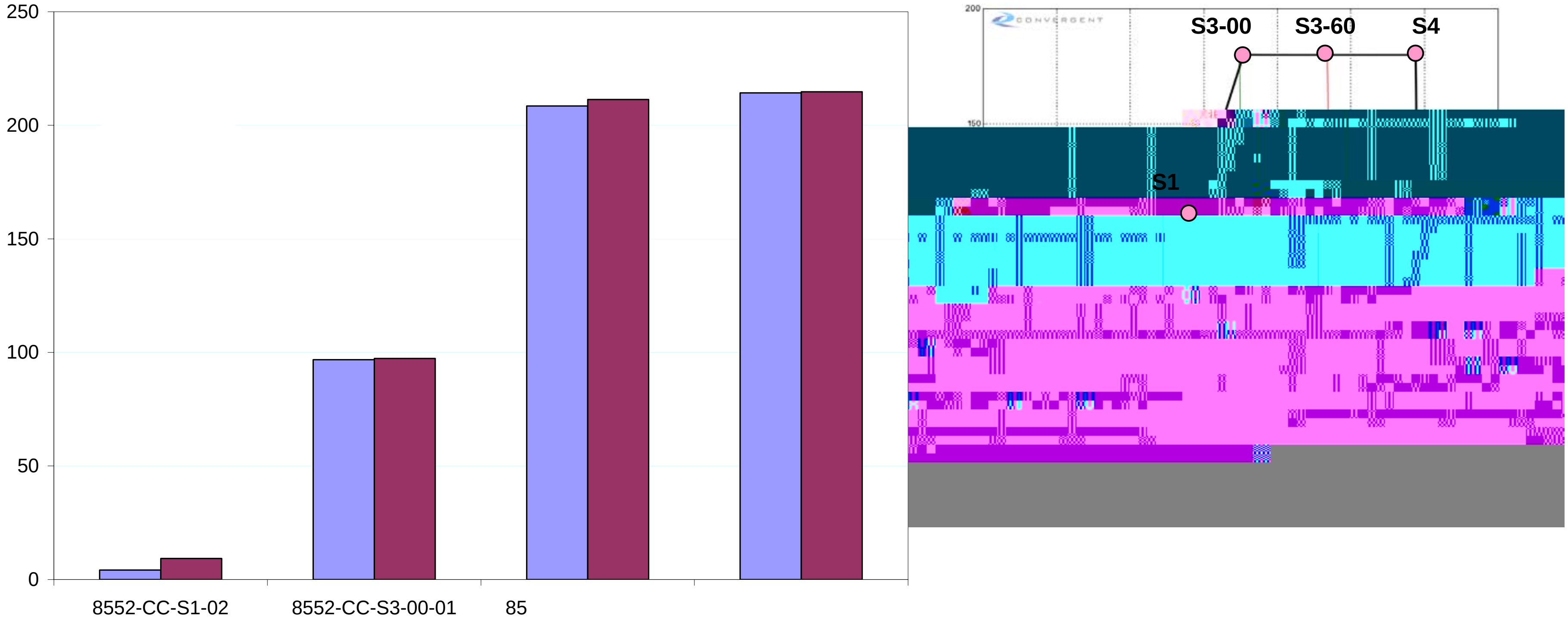


Isothermal Tests – All

Post-Hold Tg

Post-Hold Tg

Tg values at various points along the MRCC were measured and then compared to the predictions of the cure kinetics model. Good agreement was observed.



Goodness of Fit - Overview

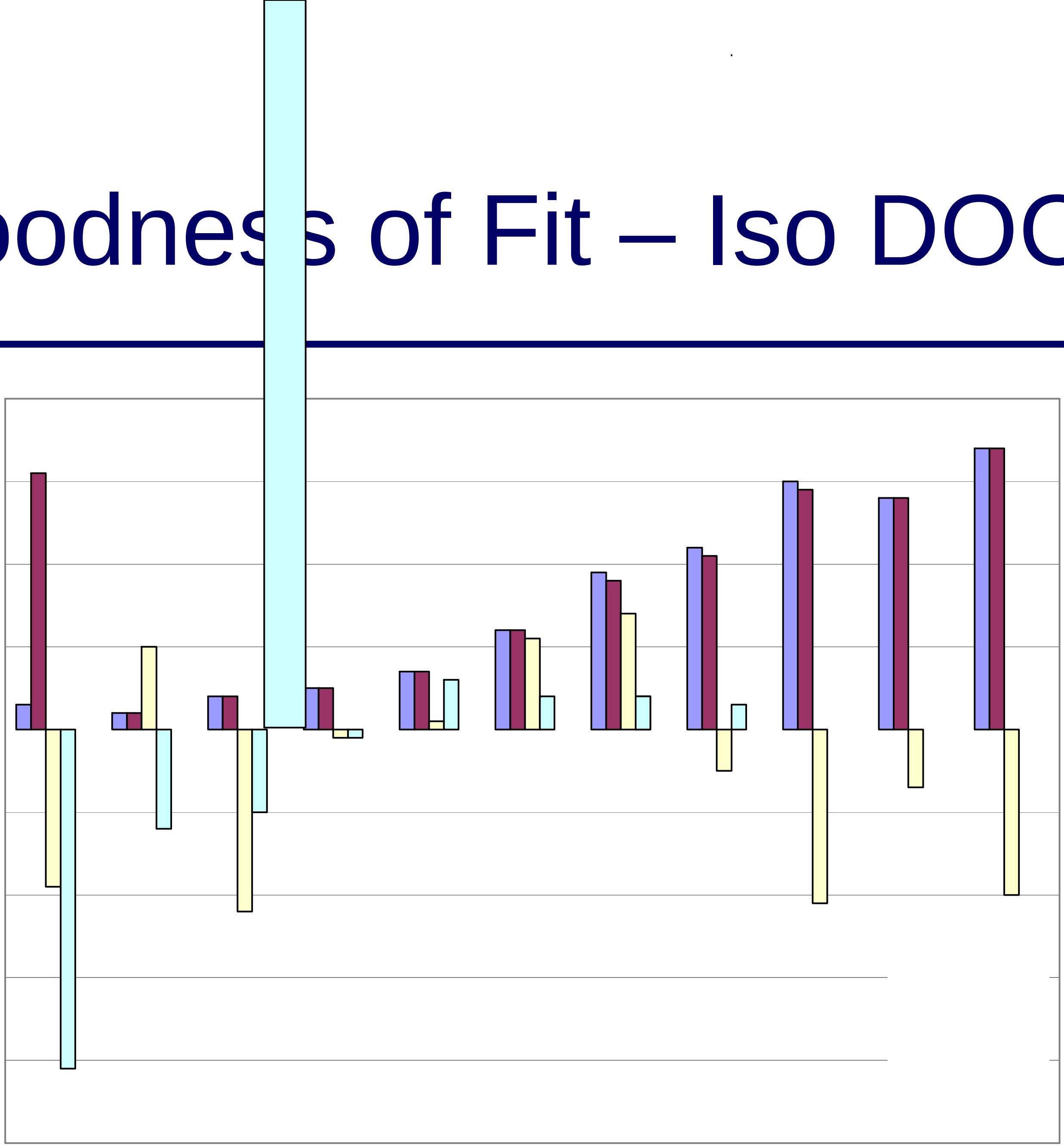
- Goodness of fit is measured by comparing the test to the model prediction at several key points in the

CONV[illegible]RGENT
MANUFACTURIN

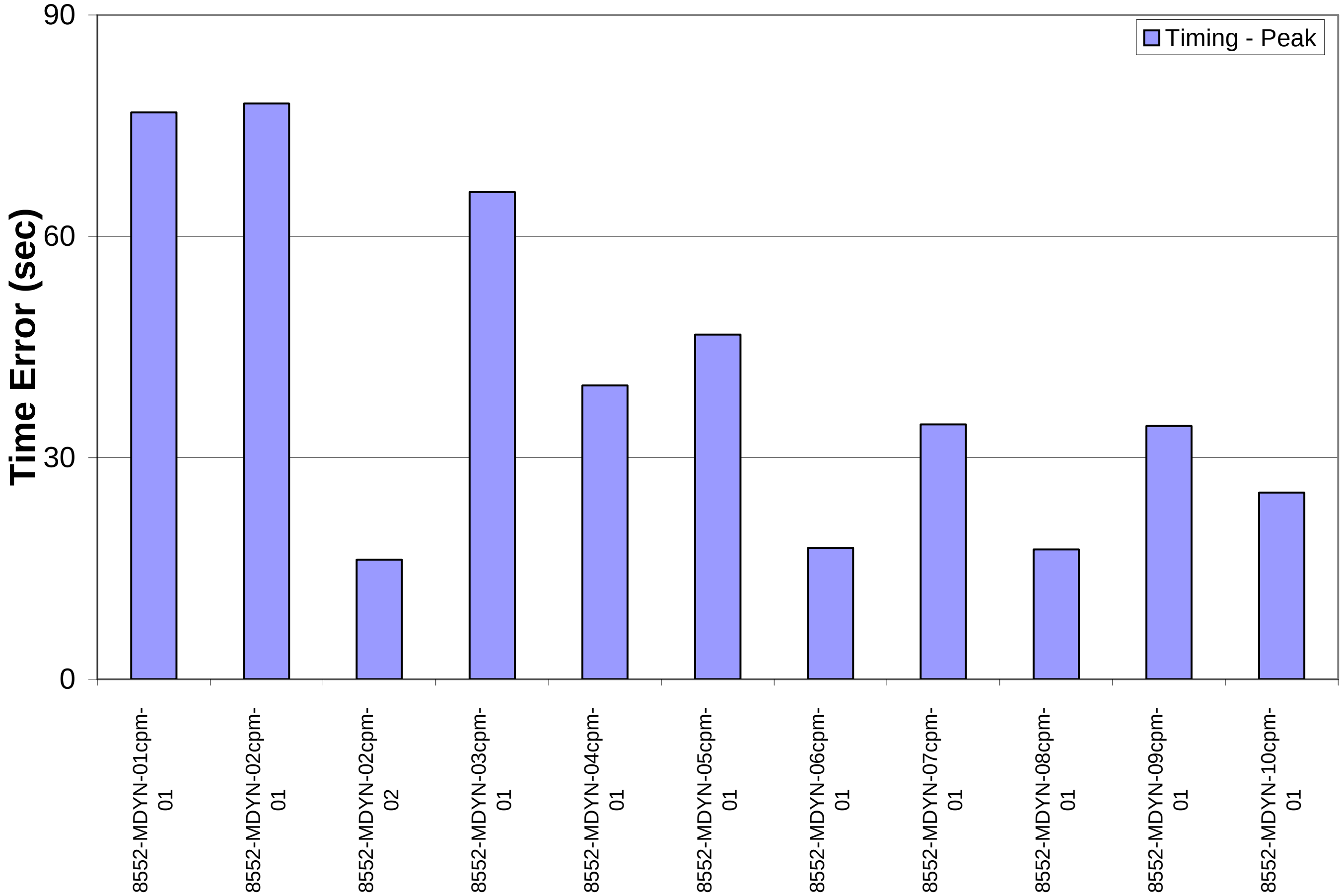
Goodness of Fit – Peak

Goodness of Fit – Other

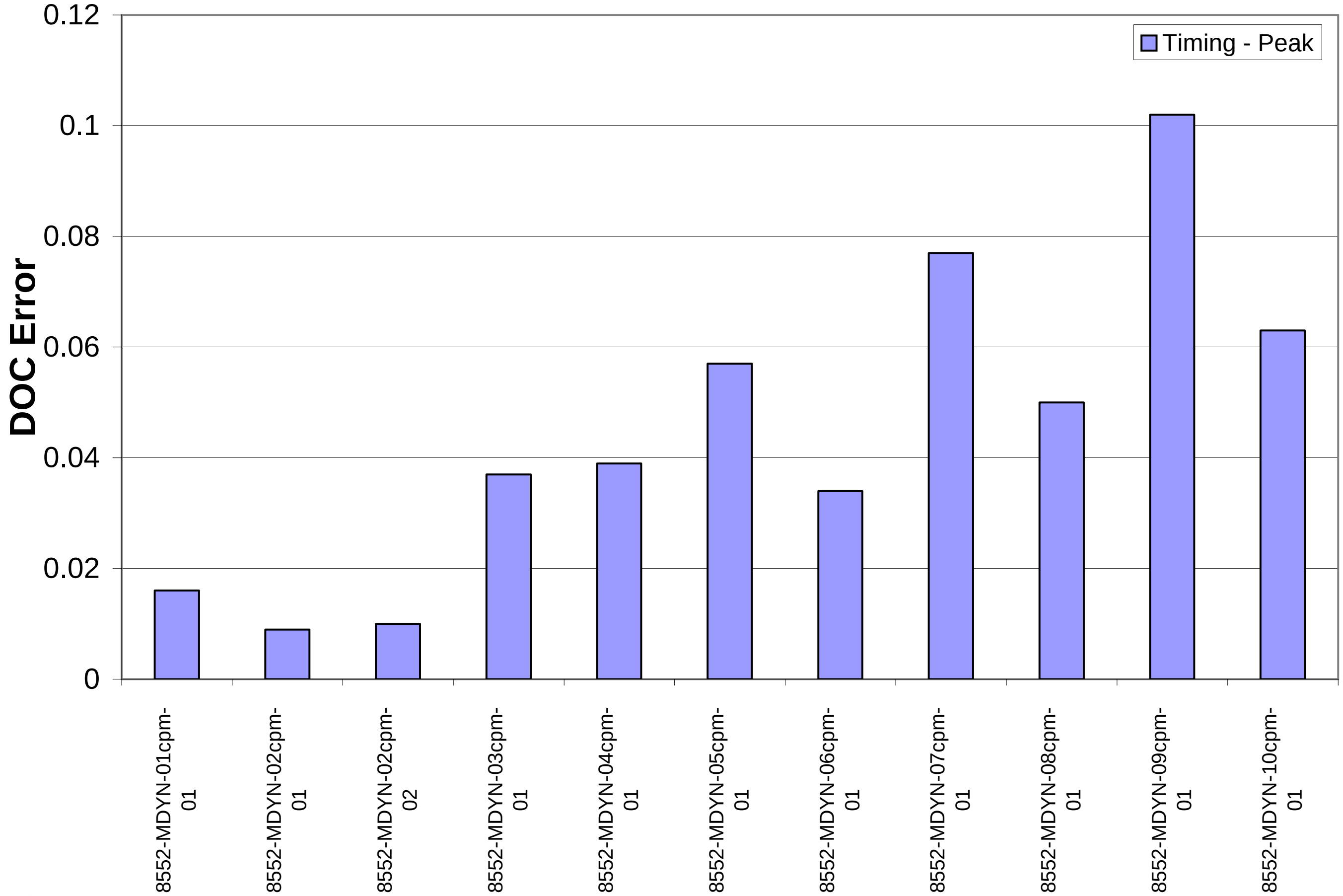
Goodness of Fit – Iso DOC



Goodness of Fit – Dyn Timing

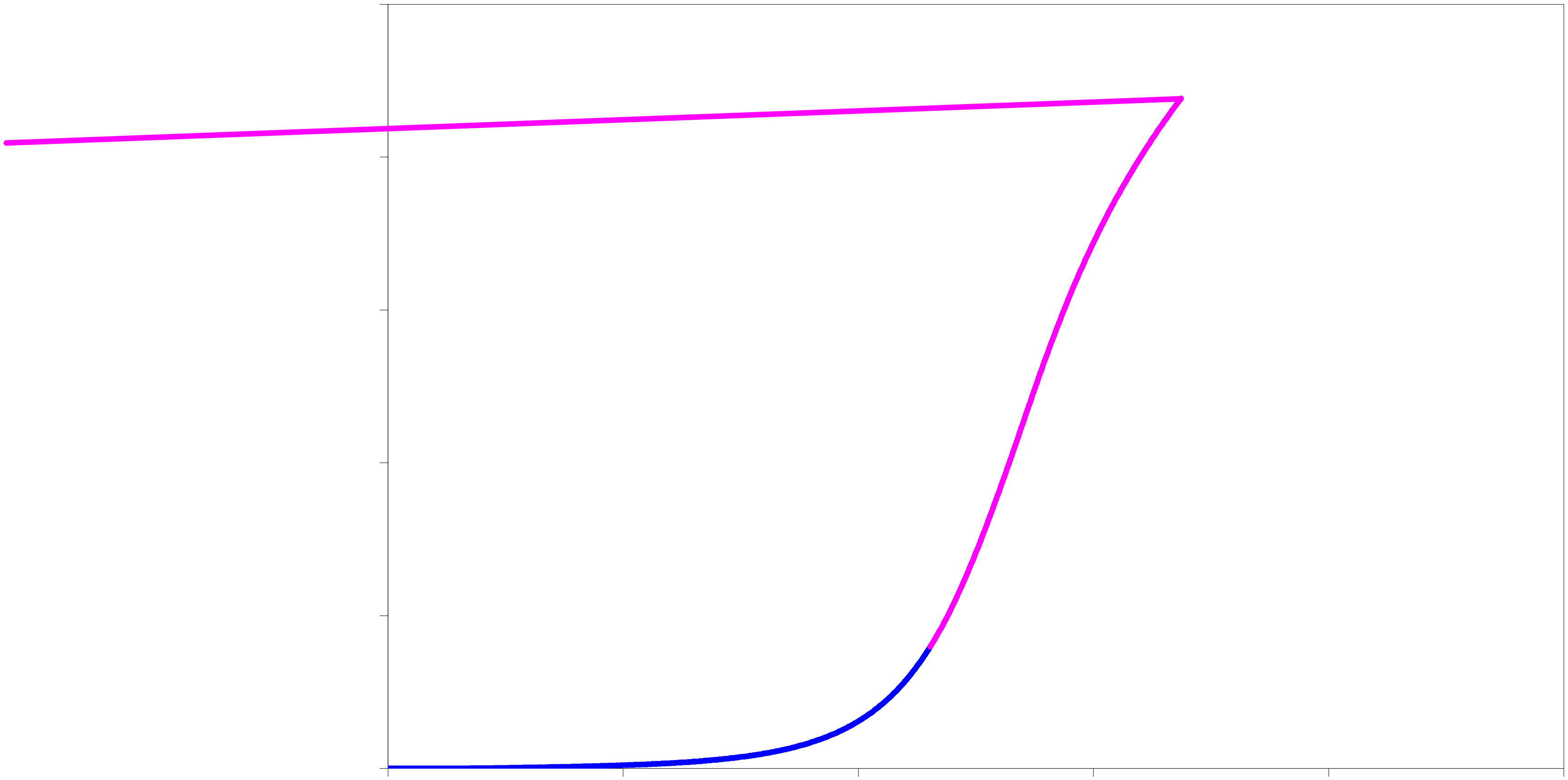


Goodness of Fit – Dyn DOC

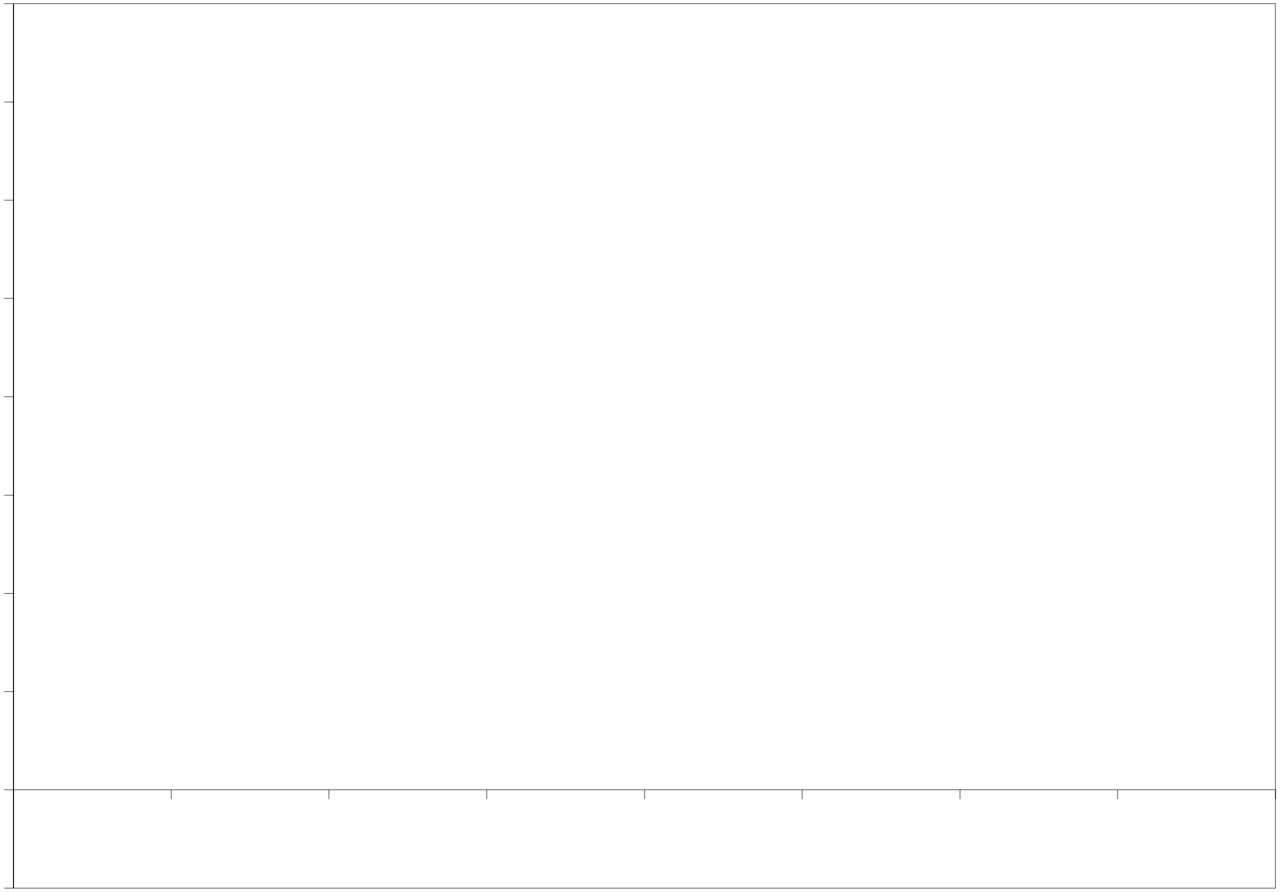


Other Forms - Fabric

Other Forms - Fabric

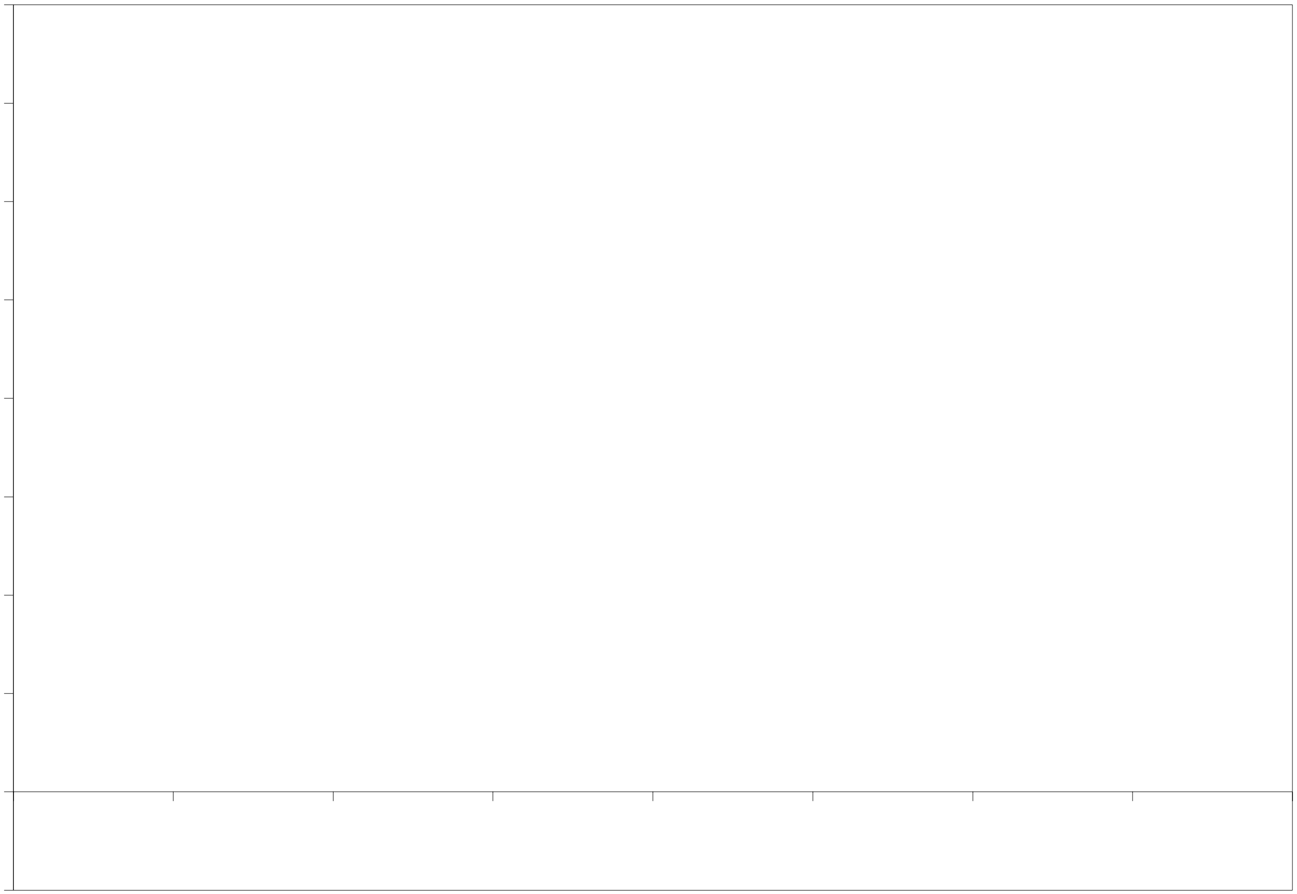


Other Forms - Film



Other Forms - Film

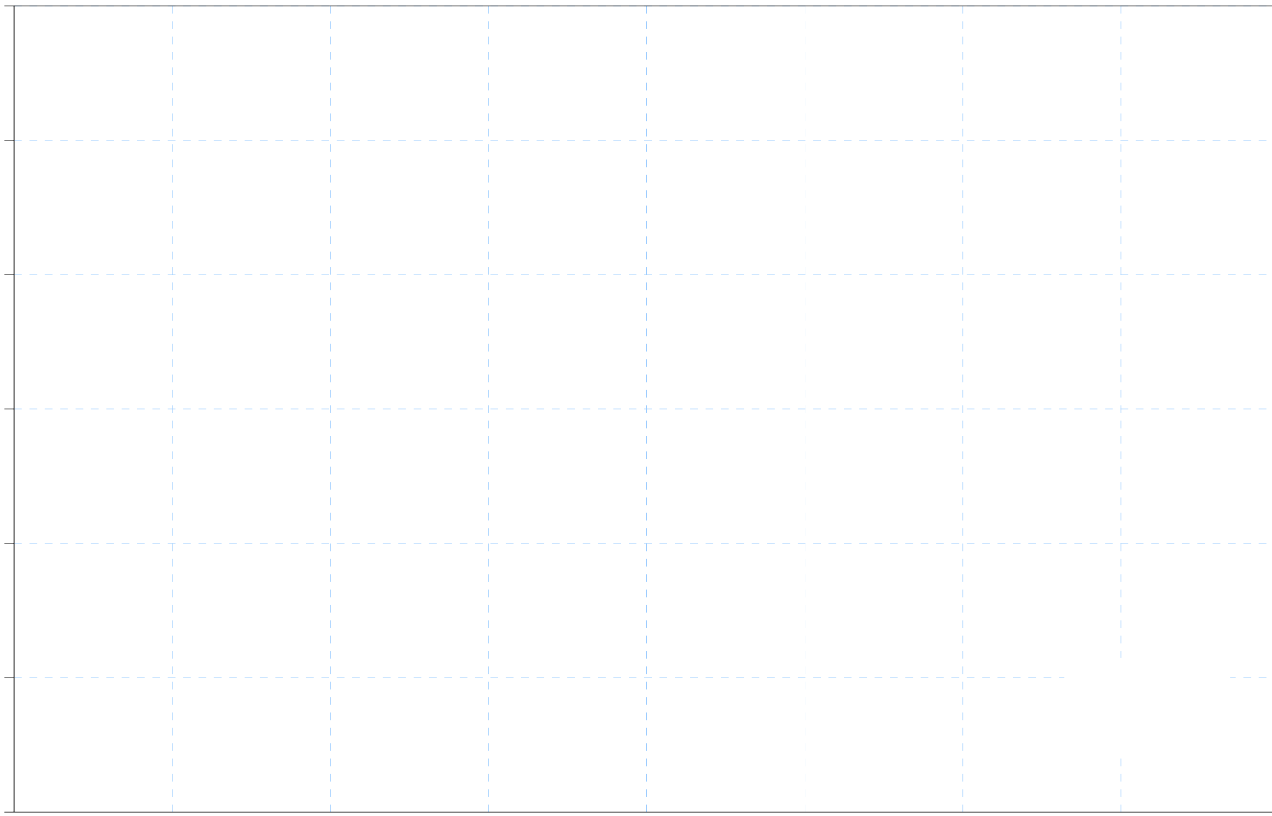
Other Forms - Film



Other Forms - Film

Other Forms – HR Values

Material Model Verification – 100C



Material Model Verification – 1Cpm

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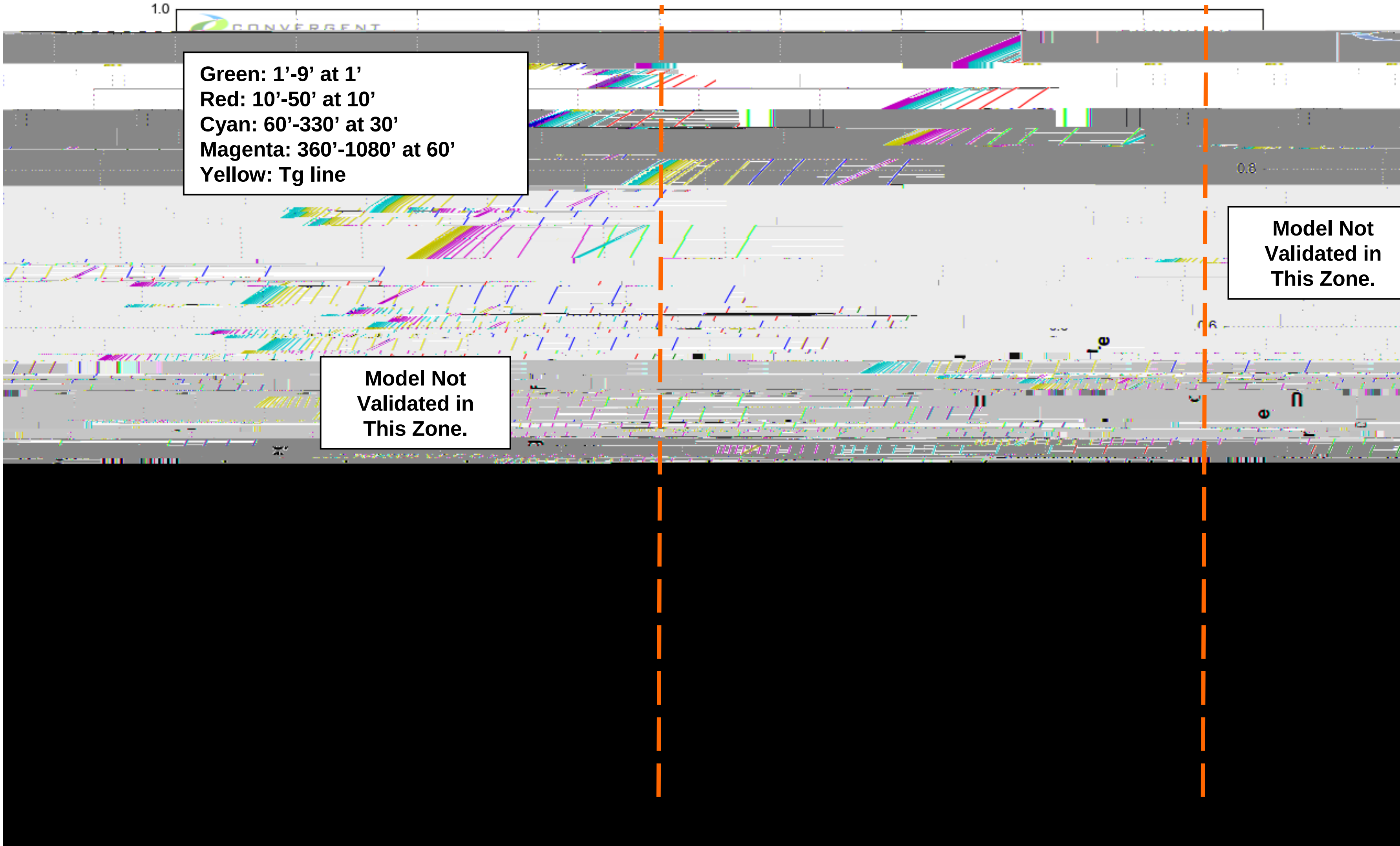

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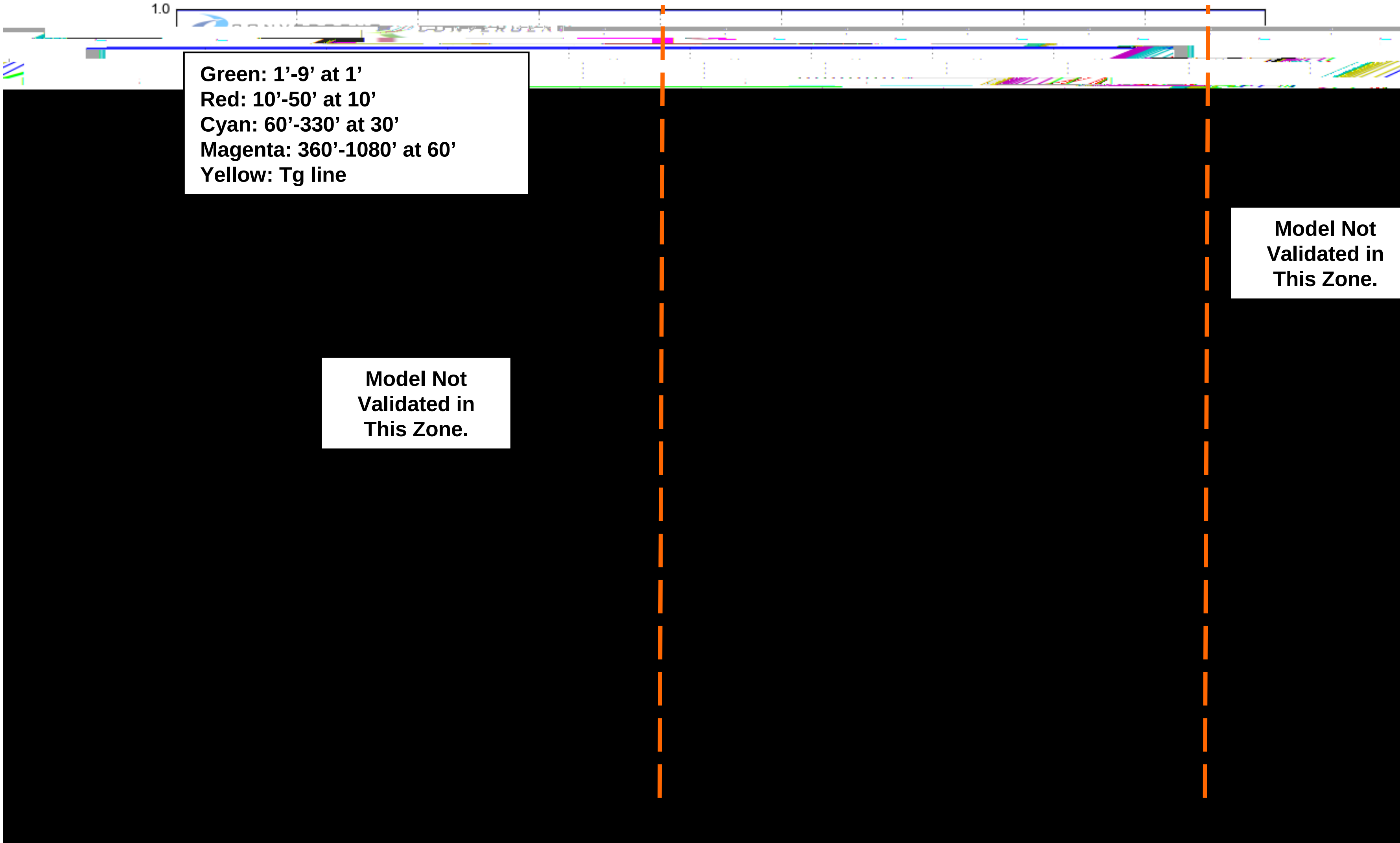
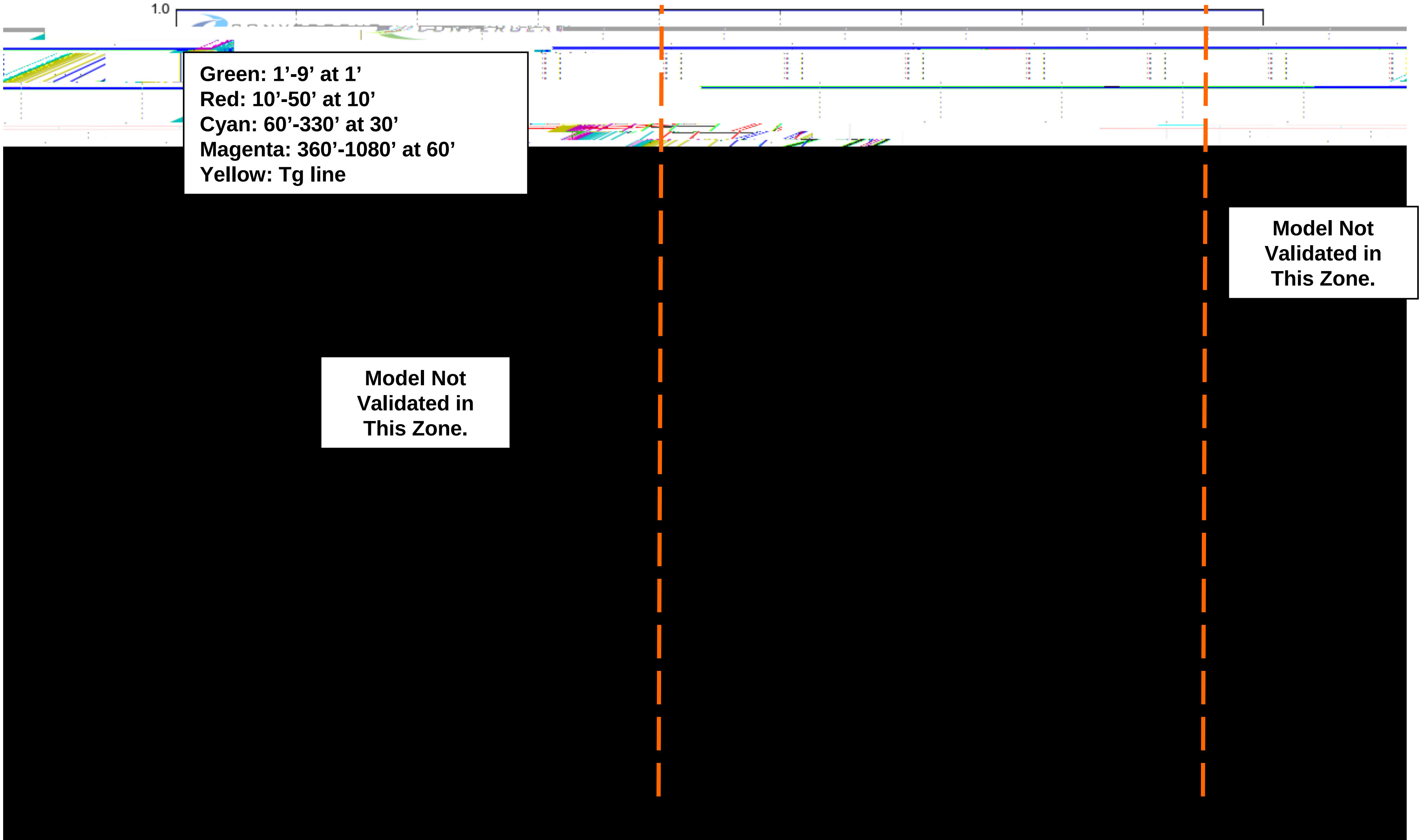
1°C/min



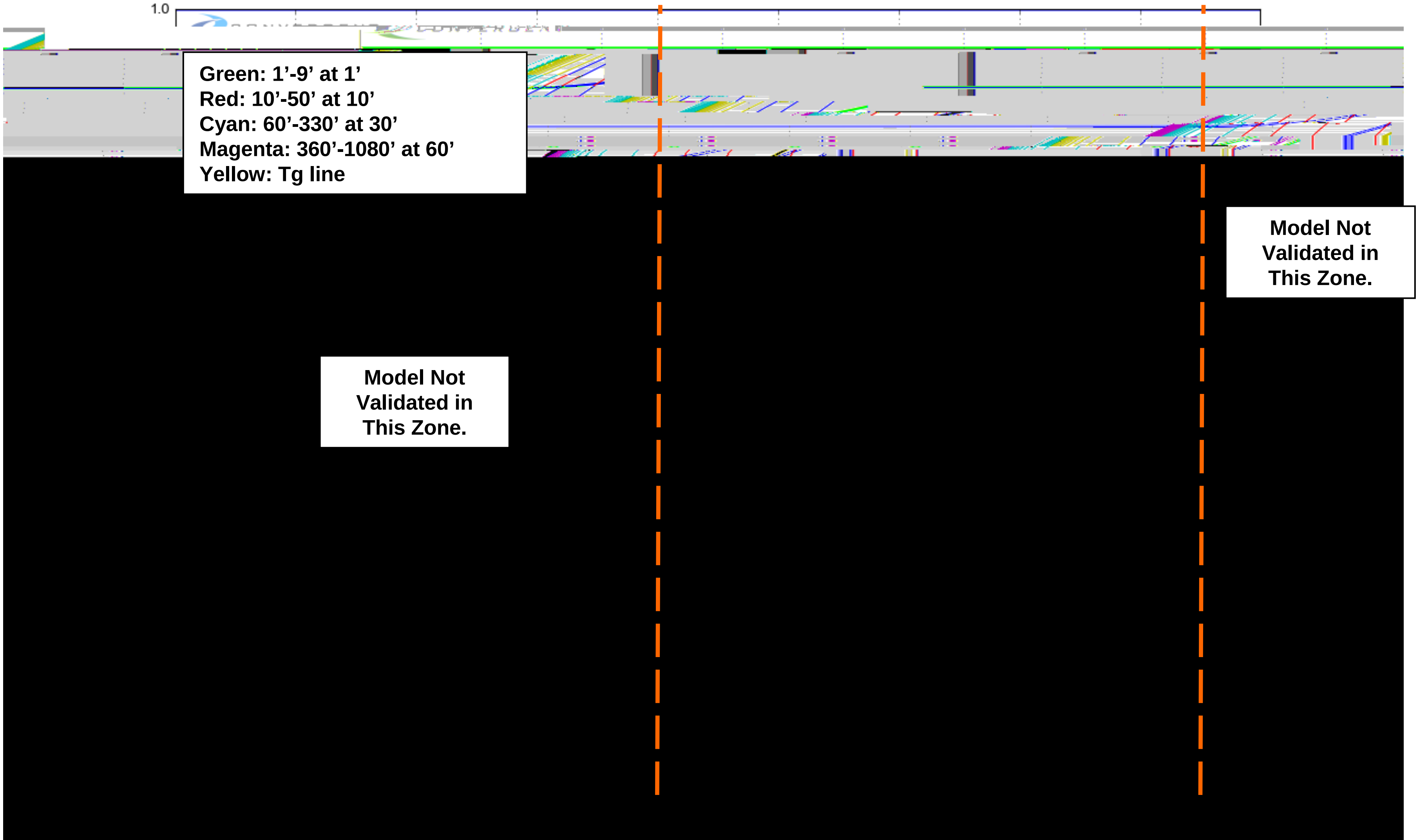
HEXPLY 8552 – Version 1.0

3°C/min



HEXPLY 8552 – Version 1.0

4°C/min



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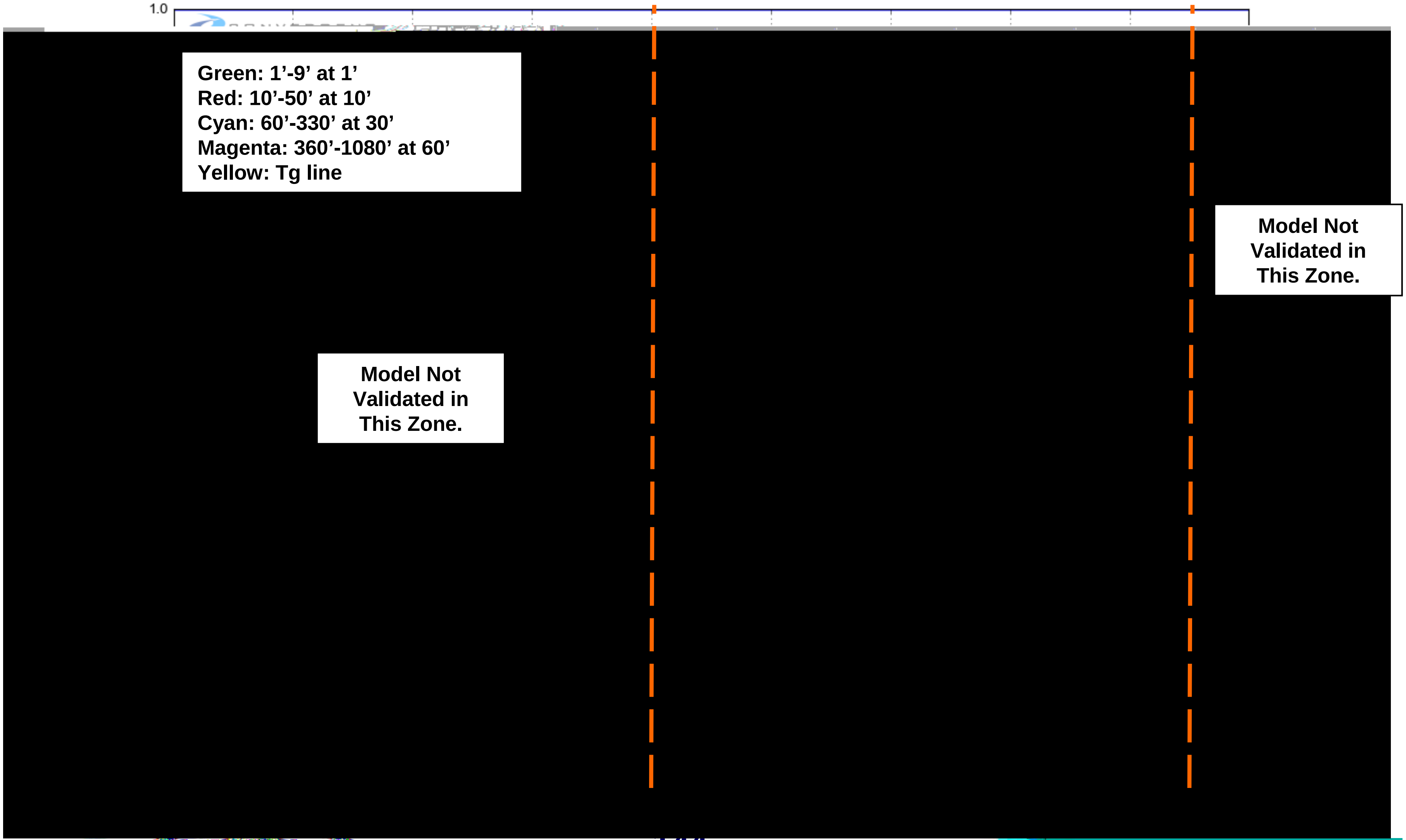
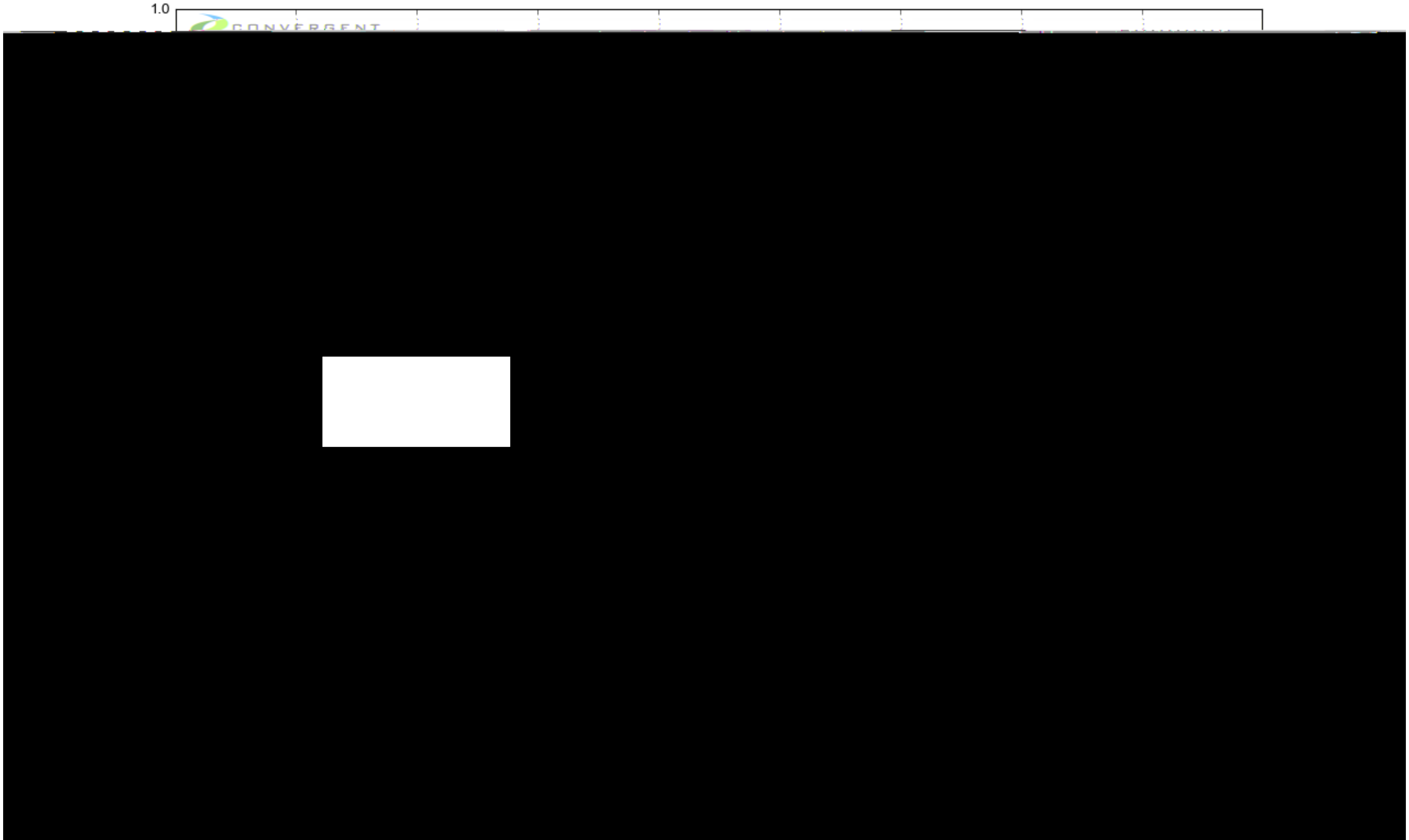
5°C/min



HEXPLY 8552 – Version 1.0



Viscosity Model

Nomenclature

η'	Dynamic viscosity or the real part of the complex viscosity
η''	Elastic complex part of the complex viscosity
η^*	Complex viscosity
μ	Material viscosity calculated by the viscosity model

Viscosity Tests

- Material viscosity was measured using an AR2000 Rheometer with parallel plate geometry.

- Dynamic tests at different ramp rates were performed to capture the changes in material viscosity as a function of temperature and degree of cure. A total of 8 tests at 1, 2, 3, and 4 C/min (two at each rate) were performed.

- Disposable aluminum plates of diameter 25mm were used at a gap of 1mm (sample thickness). Frequency of oscillation was chosen to be 1Hz.

Viscosity Measure

Rheometry tests are performed under sinusoidal oscillatory loads. The classical solution of the viscoelastic behaviour of materials in such circumstances is usually expressed in terms of storage and loss moduli. Considering the following strain function, the resulting stress would be:

Strain applied: $\gamma = \gamma_0 \sin \omega t$

Resulting stress: $\sigma = \sigma_0 \sin(\omega t + \delta)$

where δ is the phase angle between the strain and the stress. Decomposing the stress into in-phase and out-of-phase components, we get:

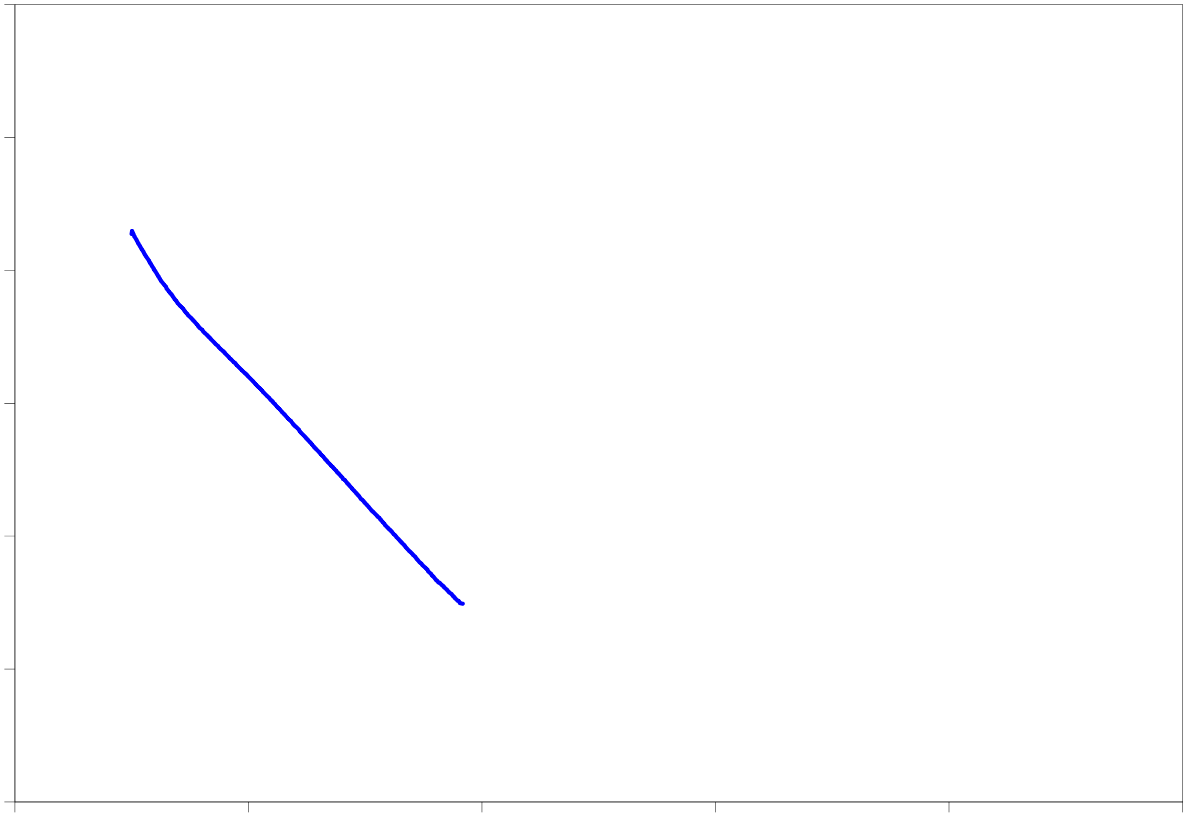
$$\sigma = \gamma_0 (G' \sin \omega t + G'' \cos \omega t)$$

where *G'* is the in-phase (elastic or storage) modulus and *G"* is the out-of-phase (viscous or loss) modulus.

Viscosity Measure

Alternatively, a complex viscosity η* can be defined with "dynamic viscosity" (η

Raw Data



Mathematical Model

The viscosity model chosen is in the following form[1]:

$$\mu = \mu_1(T) + \mu_2(T)\left(\frac{x_g}{x_g - x}\right)^{\left(A + Bx + Cx^2\right)}$$

where

$$\mu_i(T) = \mu_{0i} e^{\frac{E_i}{RT}} \qquad i = 1 \text{ or } 2$$

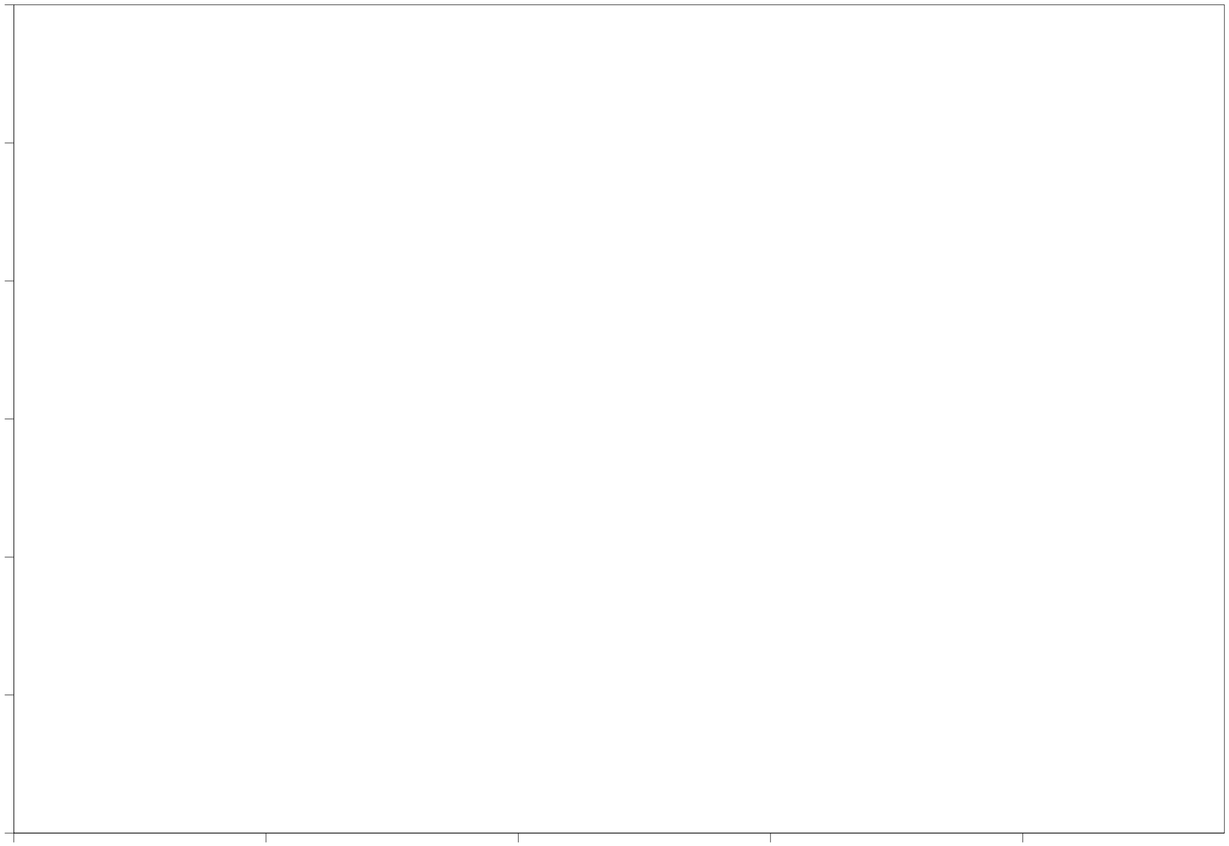
[1]Khoun and Hubert, Processing Characterization of a RTM Carbon Epoxy System for Aeronautical Applications.

Mathematical Model

The parameters of the viscosity model are determined by fitting to the experimental results.

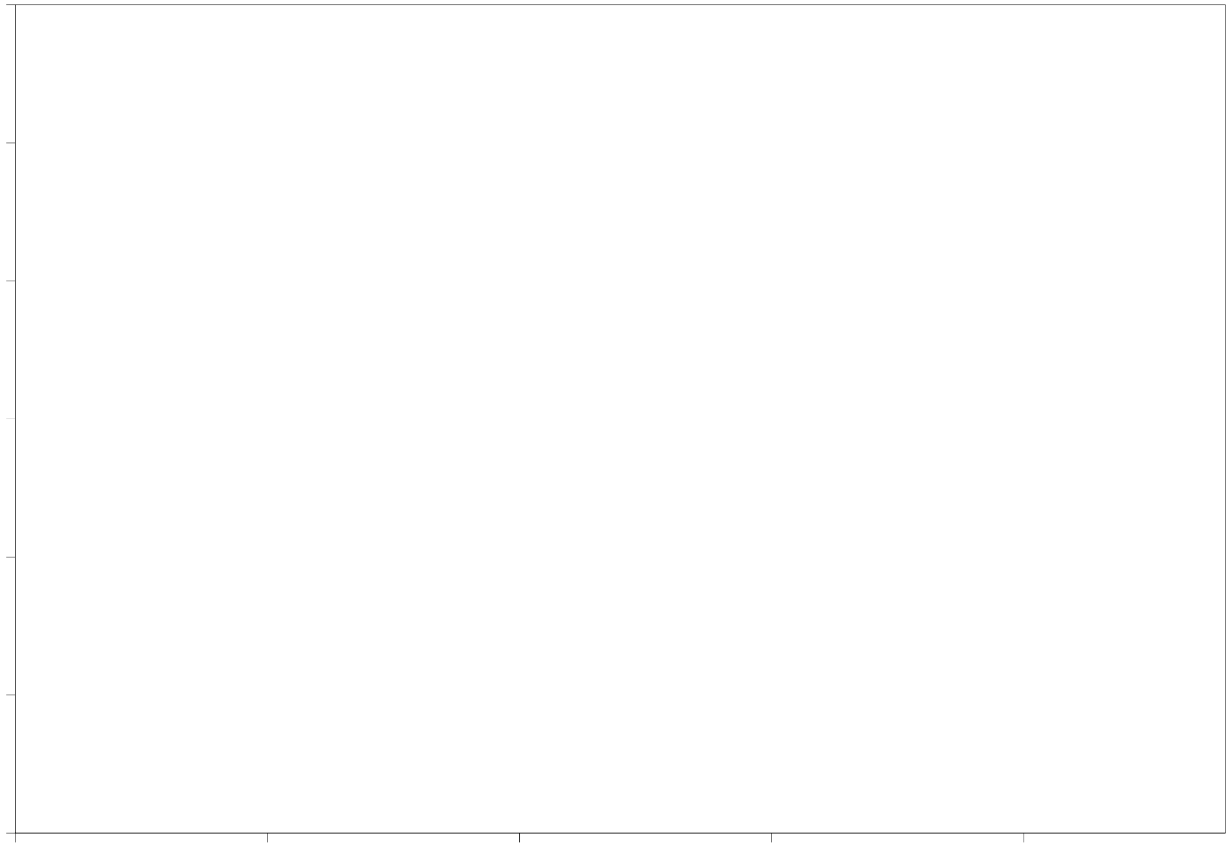
$$E_1 = 81{,}908$$

Dynamic Tests – 1cpm-1



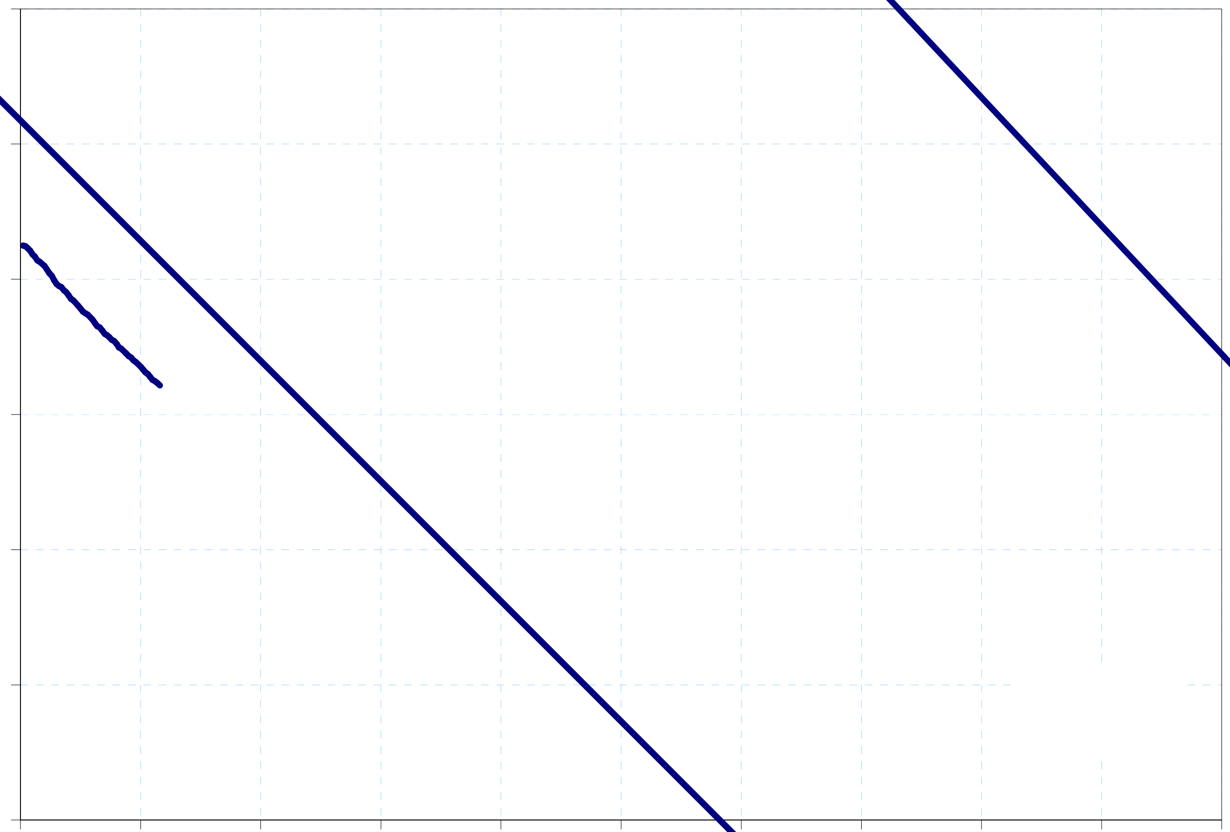
Dynamic Tests – 3cpm-1

Dynamic Tests – 3cpm-2



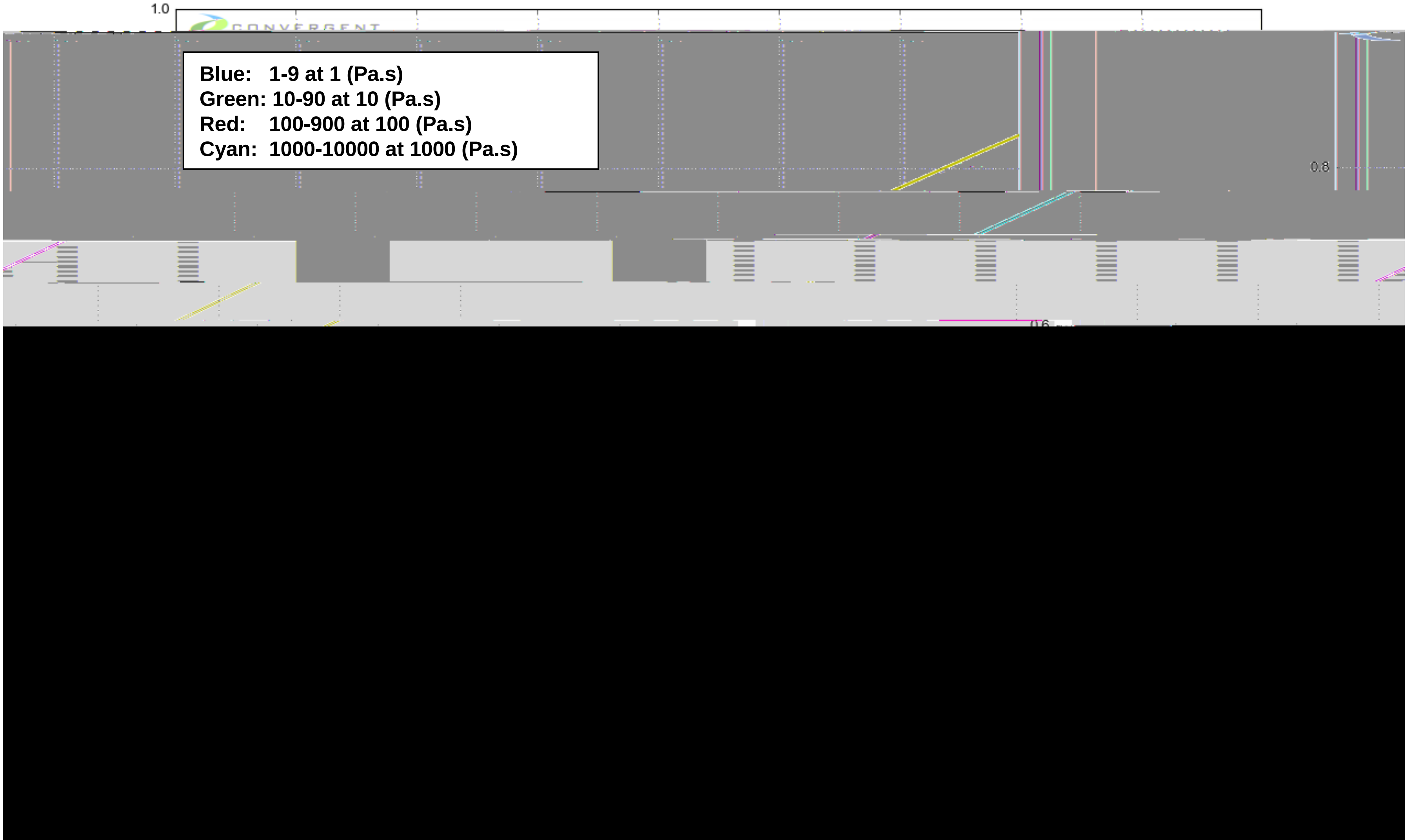
Dynamic Tests – 4cpm-2

Material Model Verification – 4Cpm-2



HEXPLY 8552 – Version 1.0

Resin Viscosity



Heat Capacity (Cp) Model

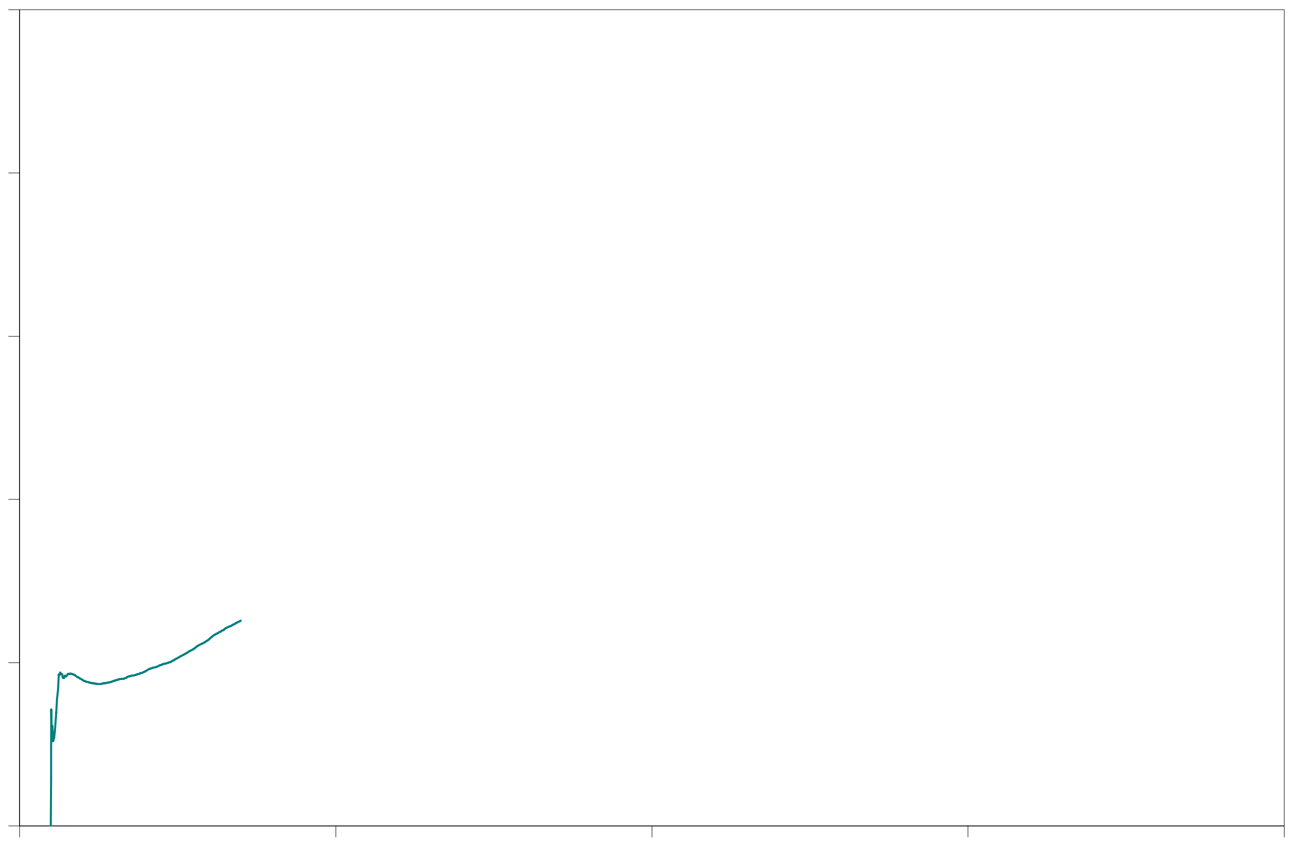
Nomenclature

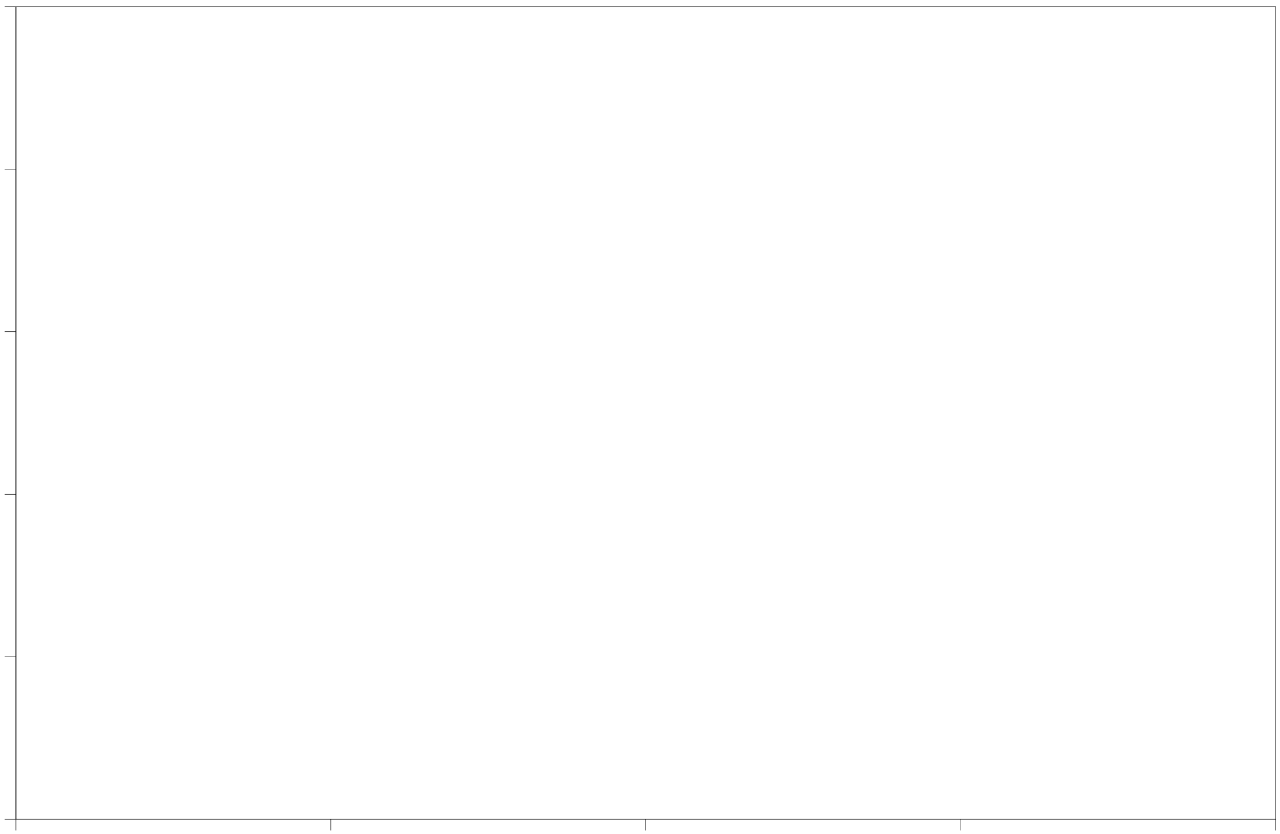
Cp Heat Capacity (J/gC)

Cp Measurement

- The DSC tests were temperature modulated to obtain
-

Raw Data





Cp Model Basics

Cp Model

The parameters of the Cp model are determined by fitting to the experimental results. The model is formulated as shown below:

$$C_{p_{ij}} = s_{ij} T + c_{ij} \quad \left(i = r, g \quad \text{and} \quad j = 0, \infty \right)$$

$$(\; = \;) \qquad + \qquad \infty$$

$$C_p = C_{p_r} + \frac{C_{p_g} - C_{p_r}}{1 + e^{k\left[\left(T - T_g\right) - \Delta T_c\right]}}$$

Glassy

$s_{g0} = 0.003775$

Rubbery

Other parameters

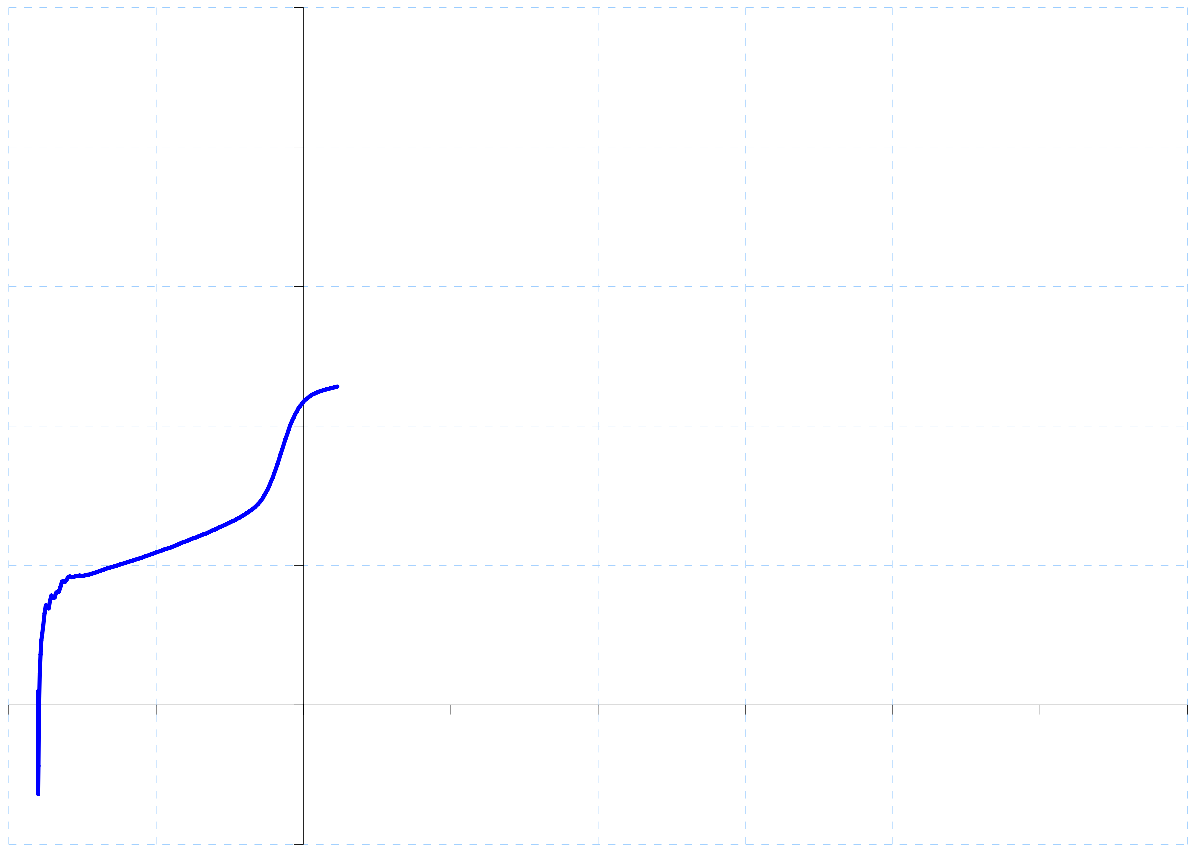
$k = 0.278$

$\Delta T_c = -1.5$

Valid for temperature values between -70ºC to 275ºC.

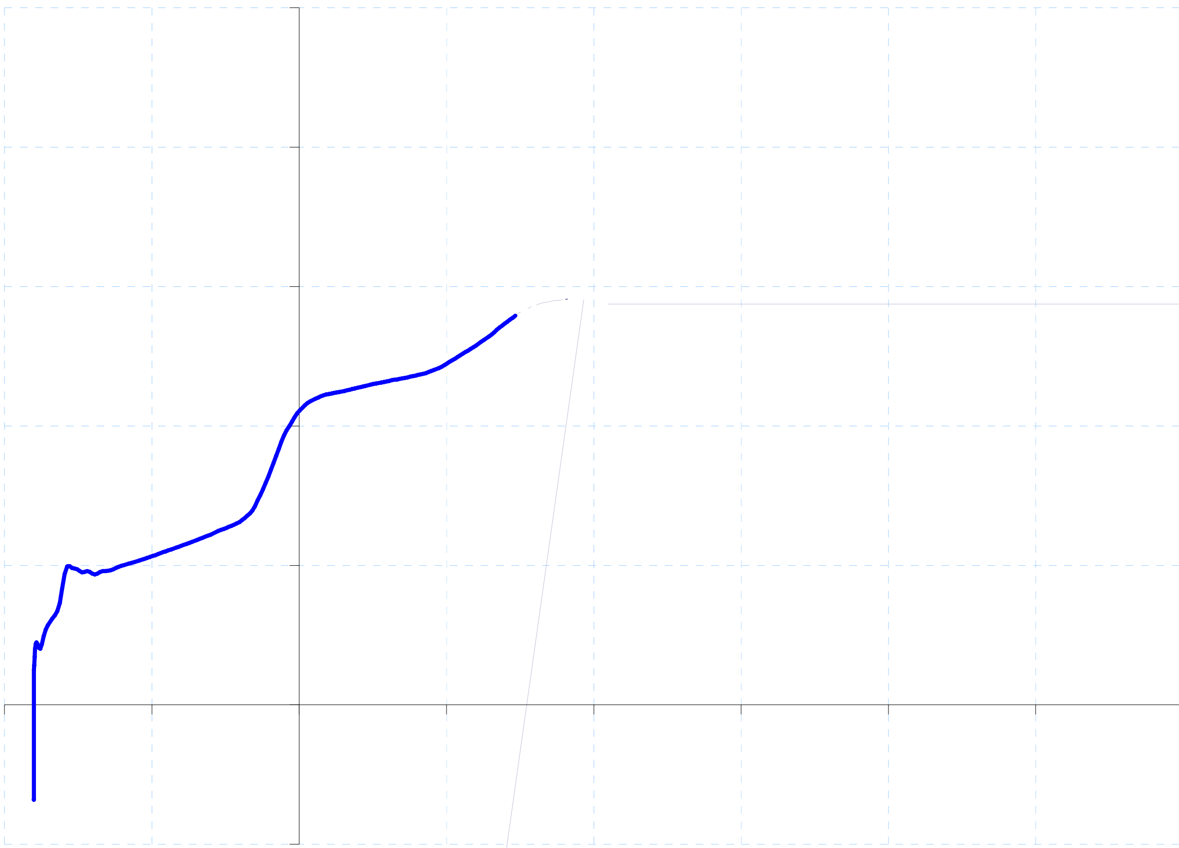
Dynamic Tests – 2cpm-2

Dynamic Tests – 3cpm



Dynamic Tests – 5cpm

Dynamic Tests – 6cpm



Dynamic Tests – 8cpm

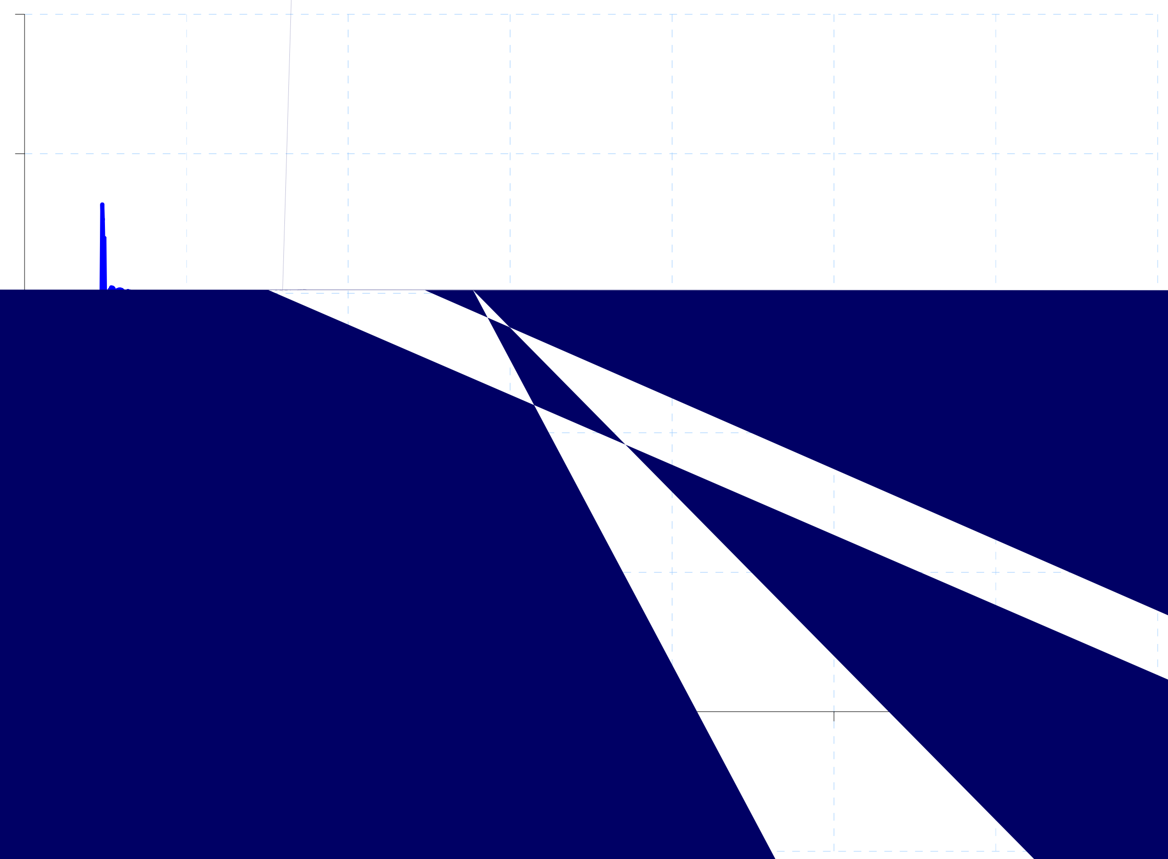
Dynamic Tests – 9cpm

Dynamic Tests – 10cpm

Isothermal Tests – 100C

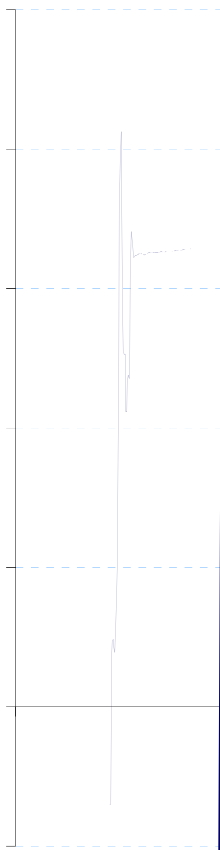
Isothermal Tests – 110C

Isothermal Tests – 120C



Ism0.0006 Tc55/WSrmal Tests –

Isothe[illegible] 160C

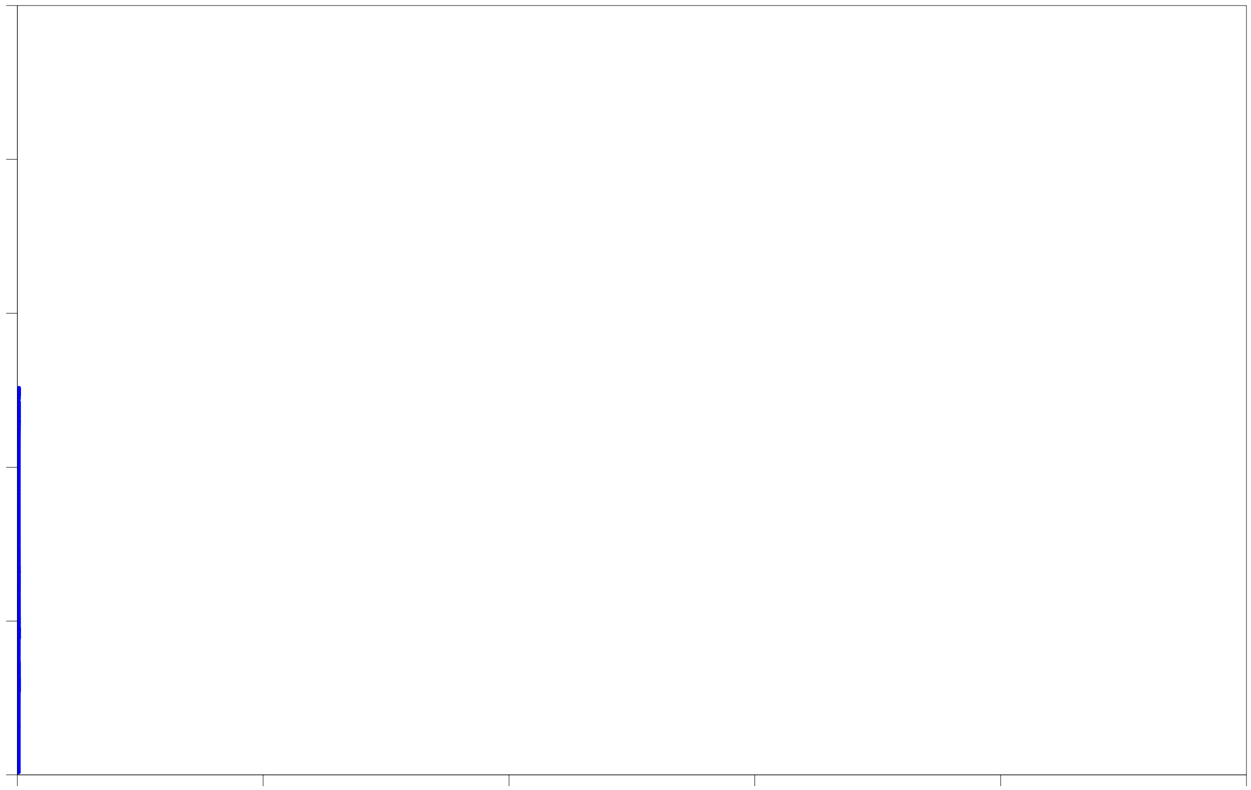


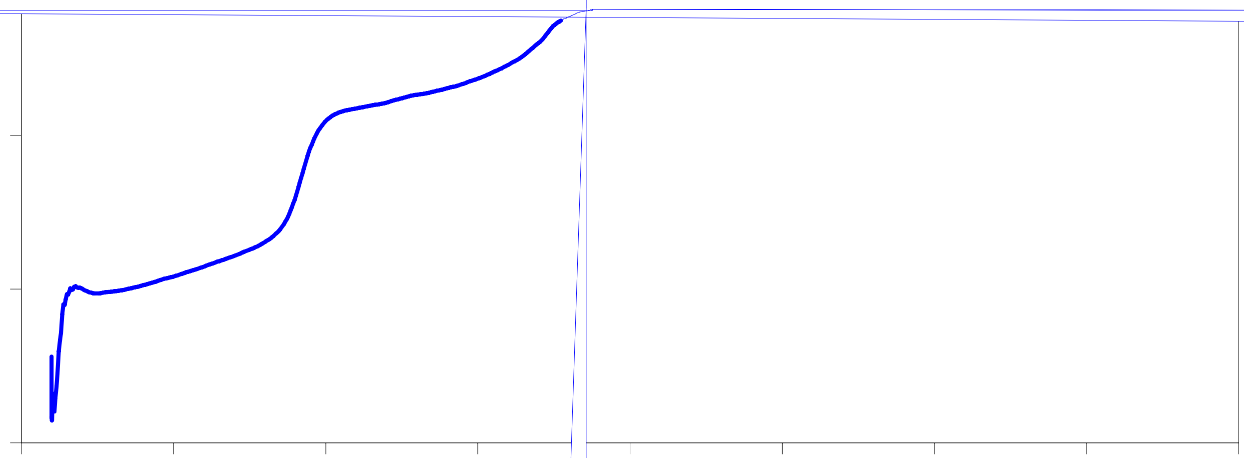
Isothermal Tests – 180C-1

Isothermal Tests – 180C-2

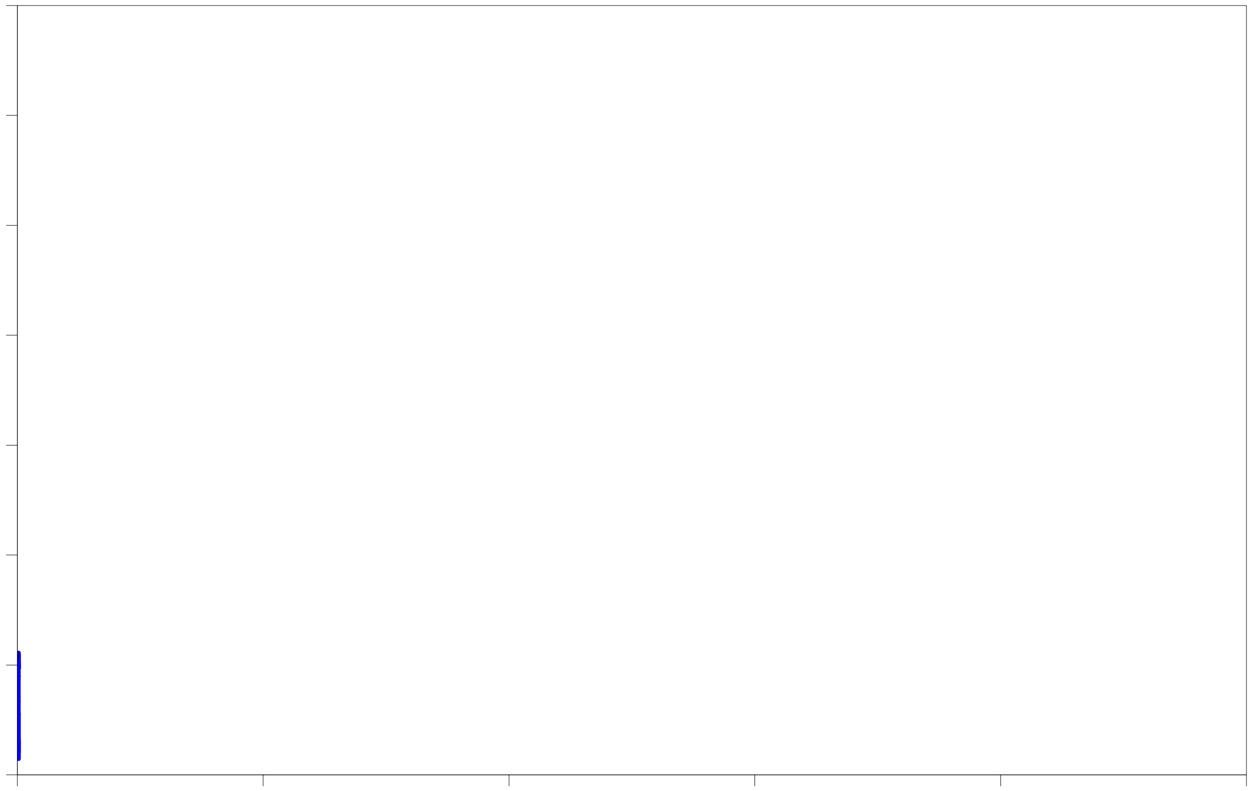
Dynamic Tests – 1Cpm

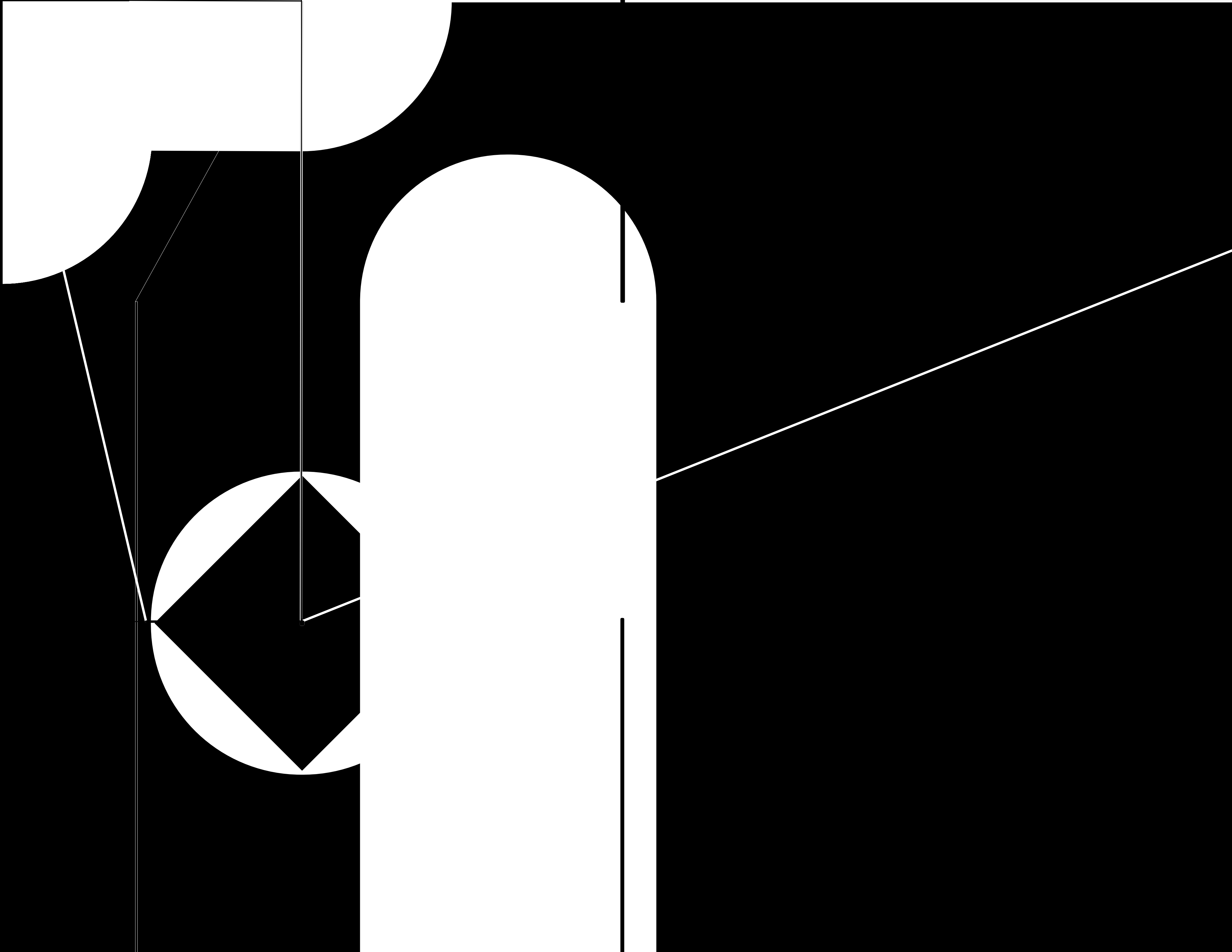
Dynamic Tests – 1Cpm



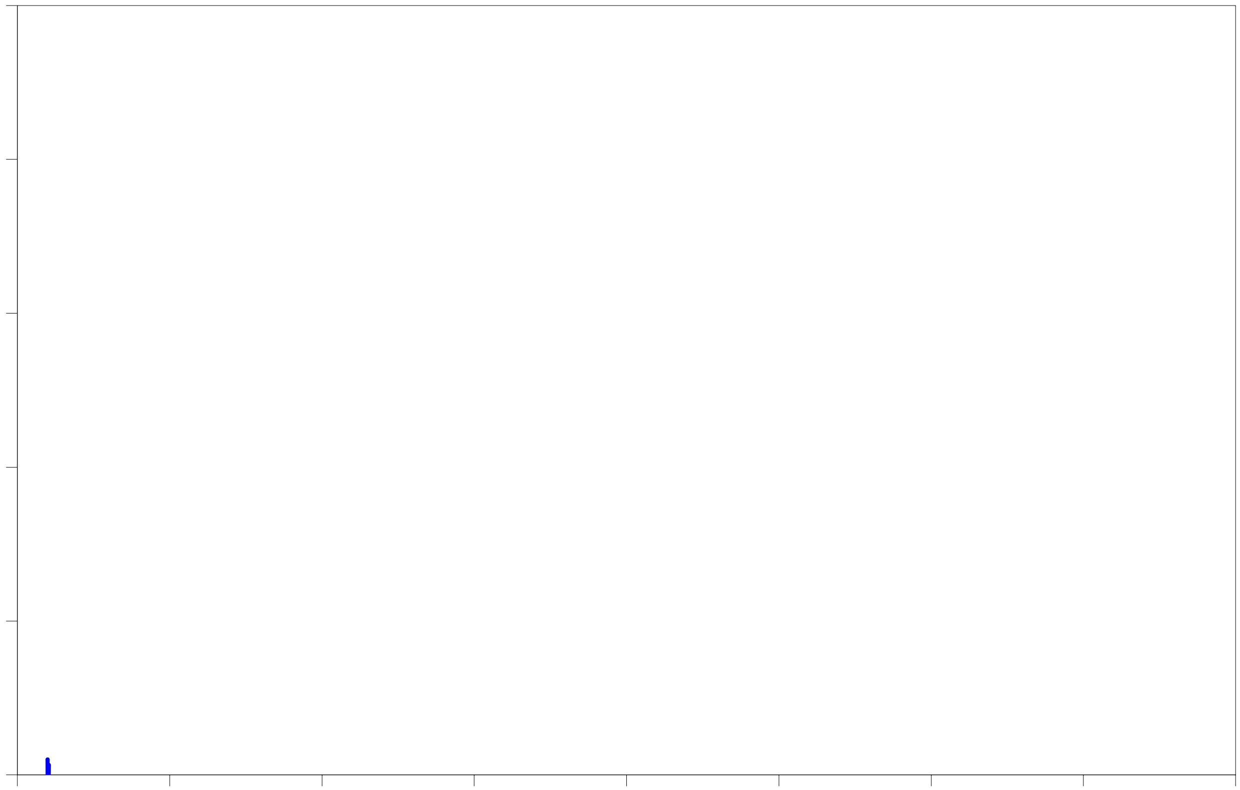


Dynamic Tests – 2Cpm-1

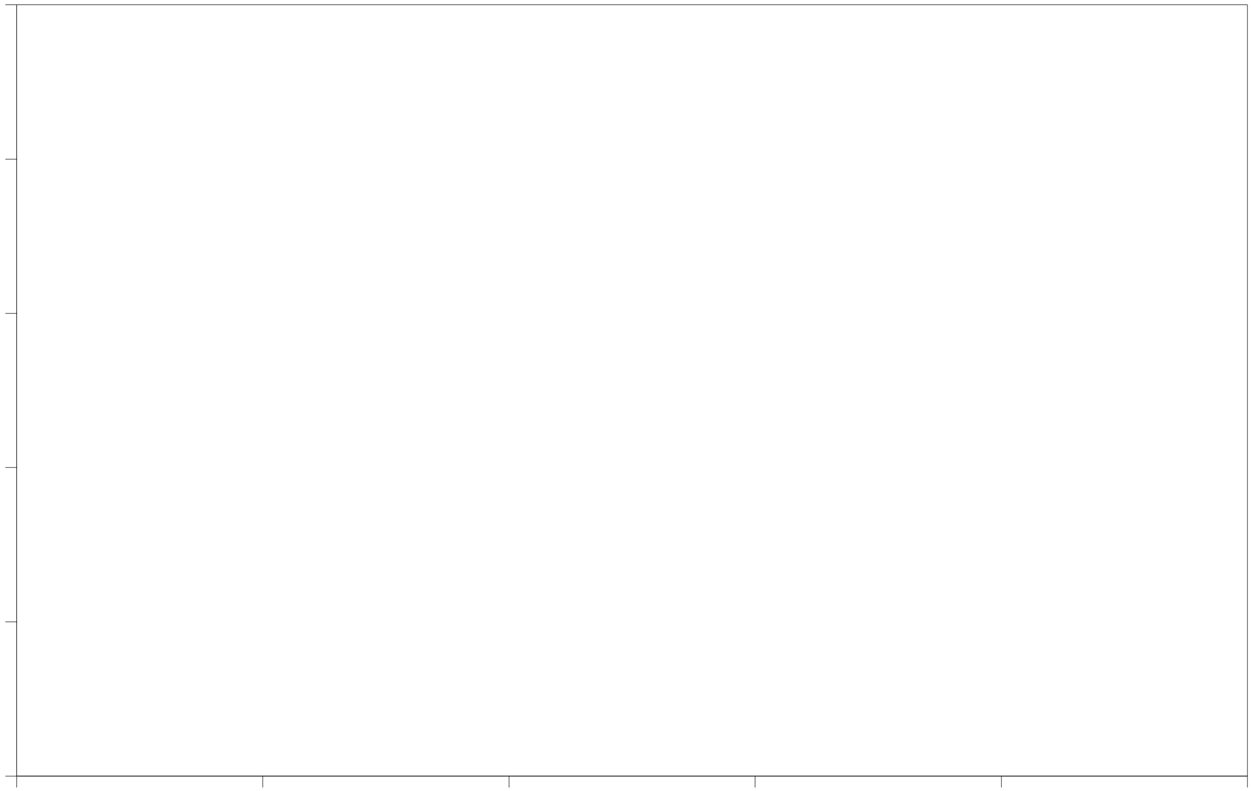




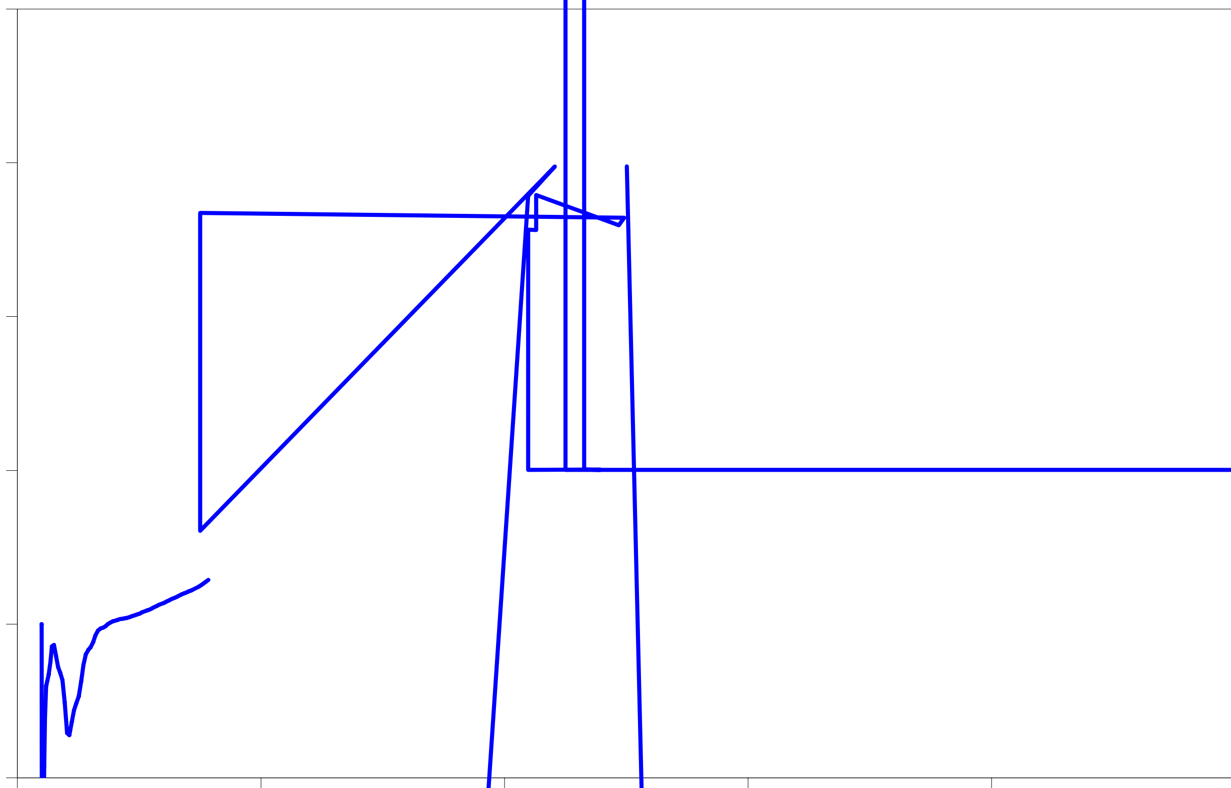
Dynamic Tests – 3Cpm



Dynamic Tests – 4Cpm

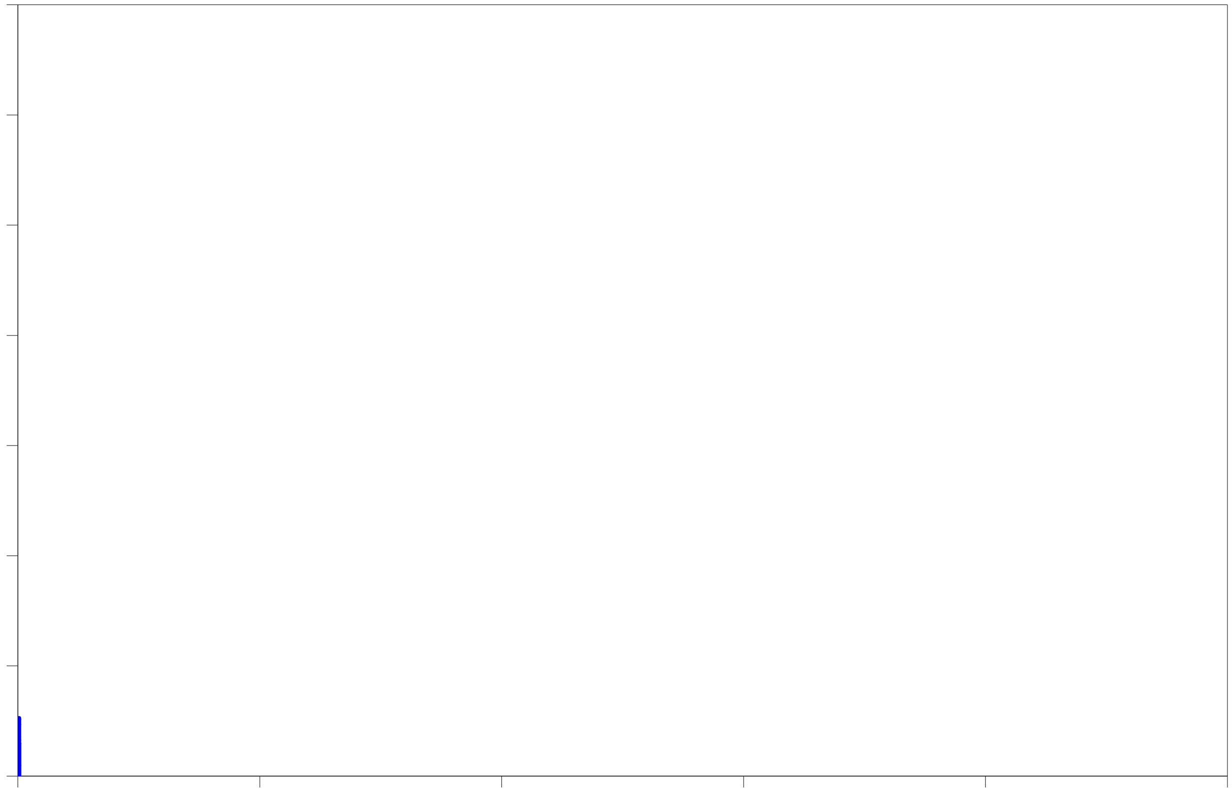


Dynamic Tests – 7Cpm

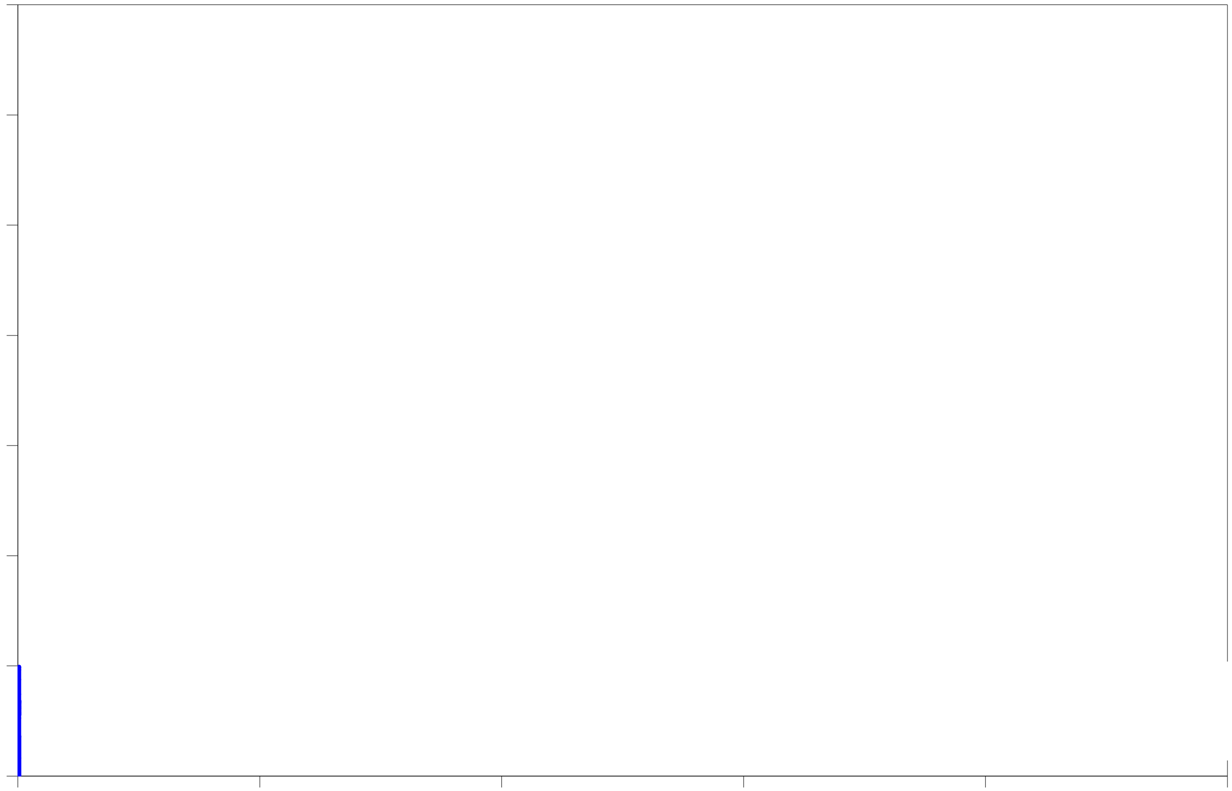


Dynamic Tests – 9Cpm

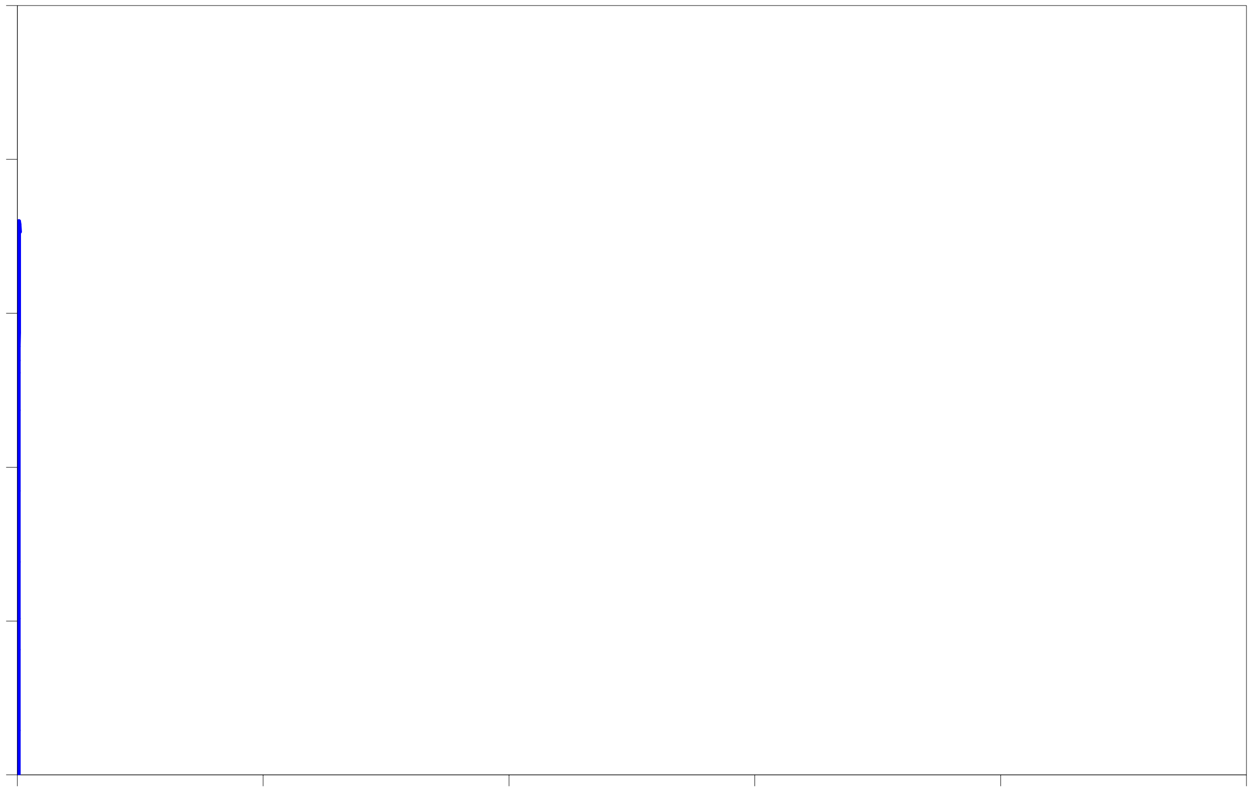
Dynamic Tests – 9Cpm



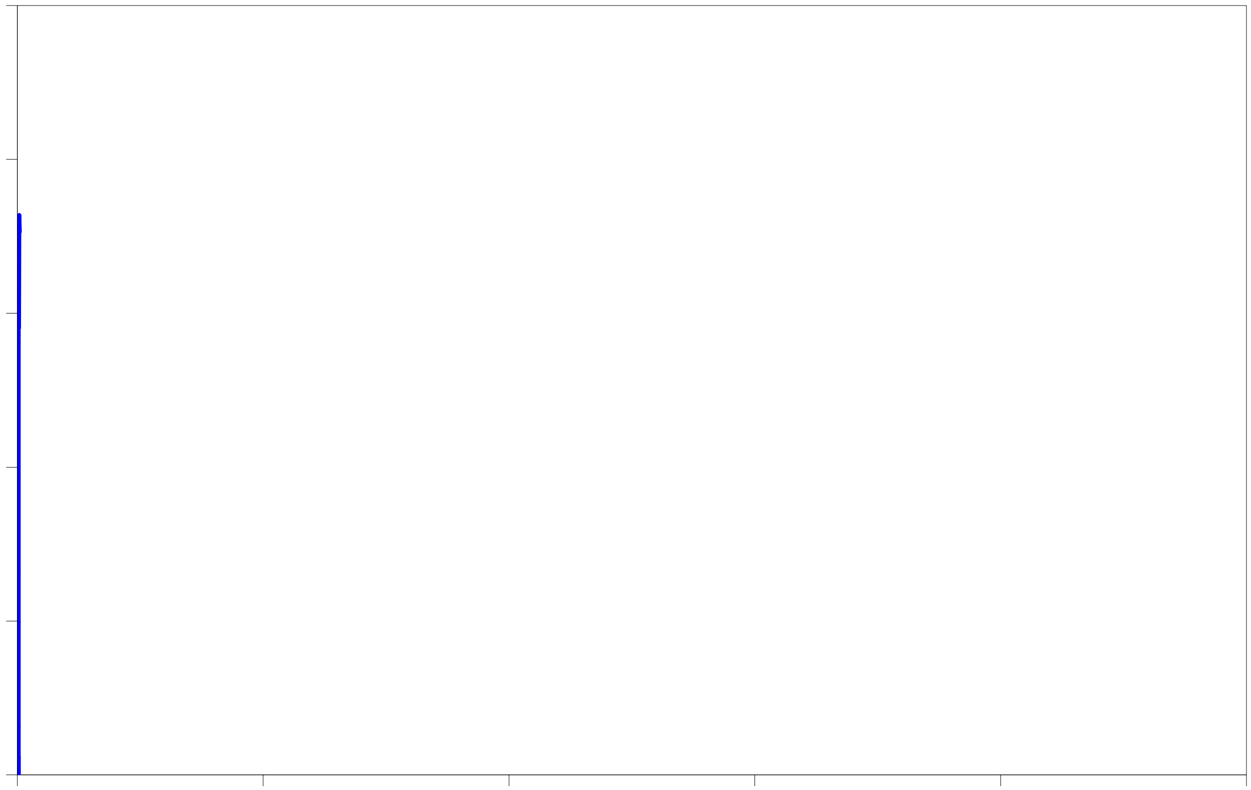
Dynamic Tests – 10Cpm



Isothermal Tests – 110C



Isothermal Tests – 120C

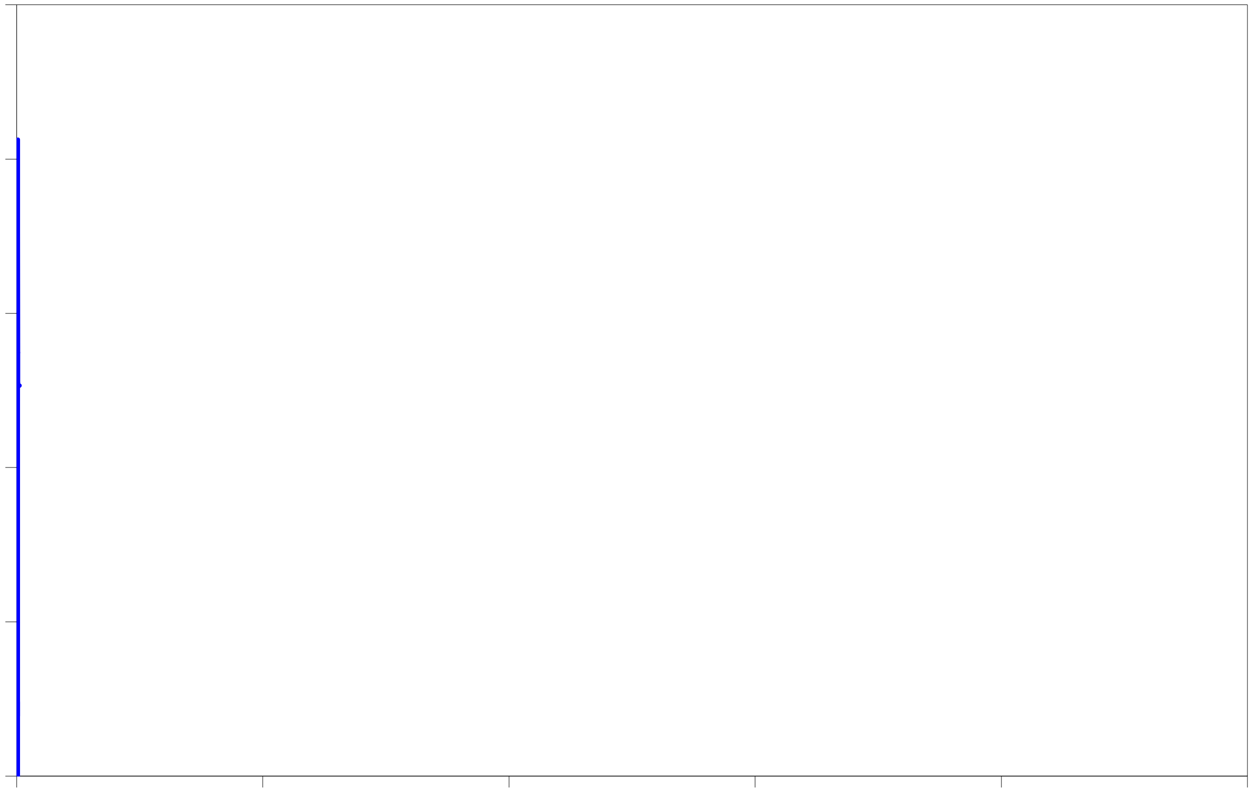


Isothermal Tests – 130C

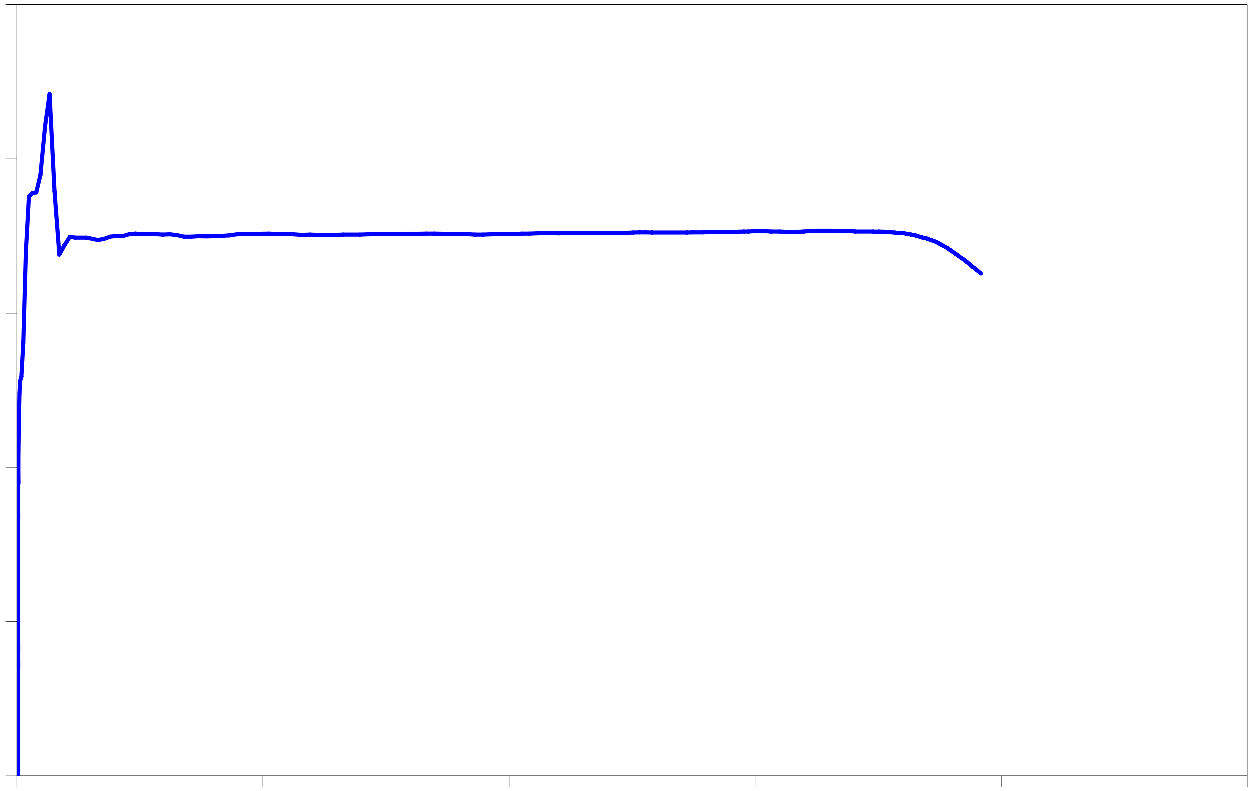
Isothermal Tests – 140C

Isothermal Tests – 150C

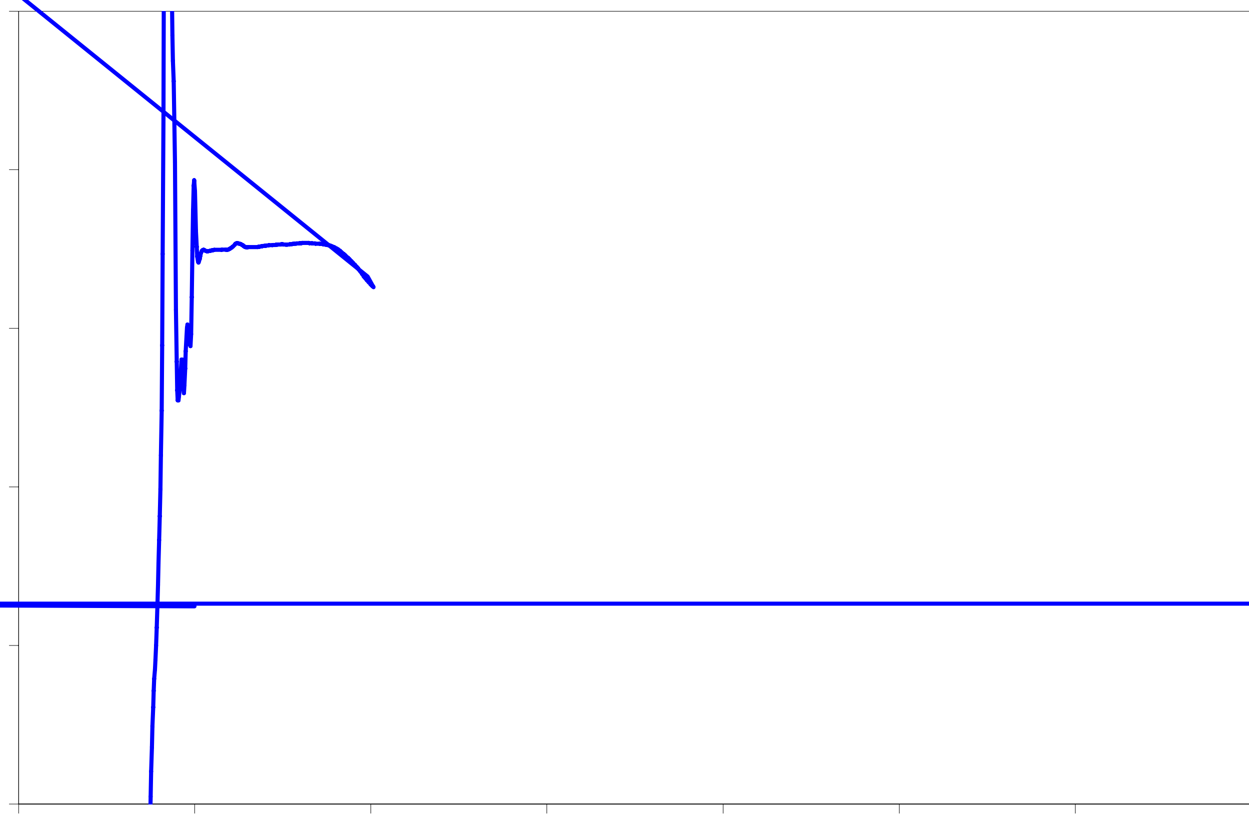
Isothermal Tests – 160C



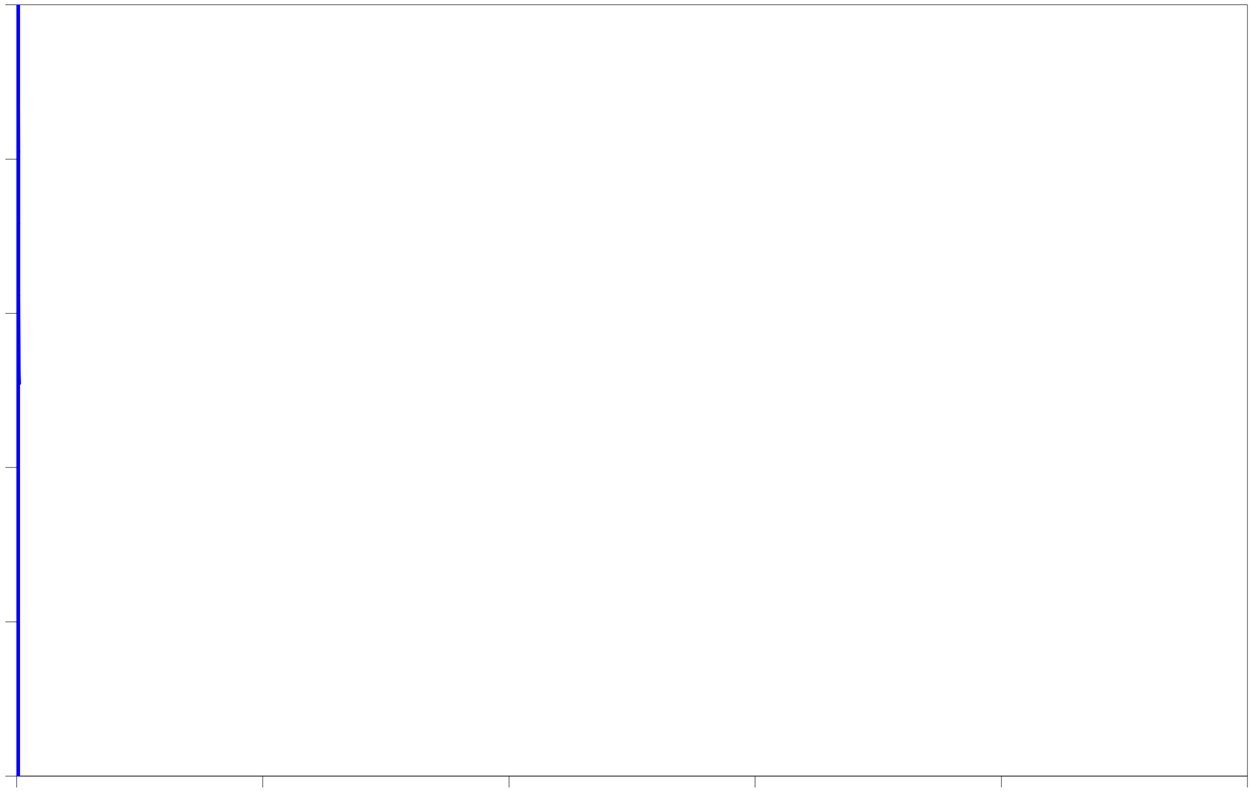
Isothermal Tests – 170C



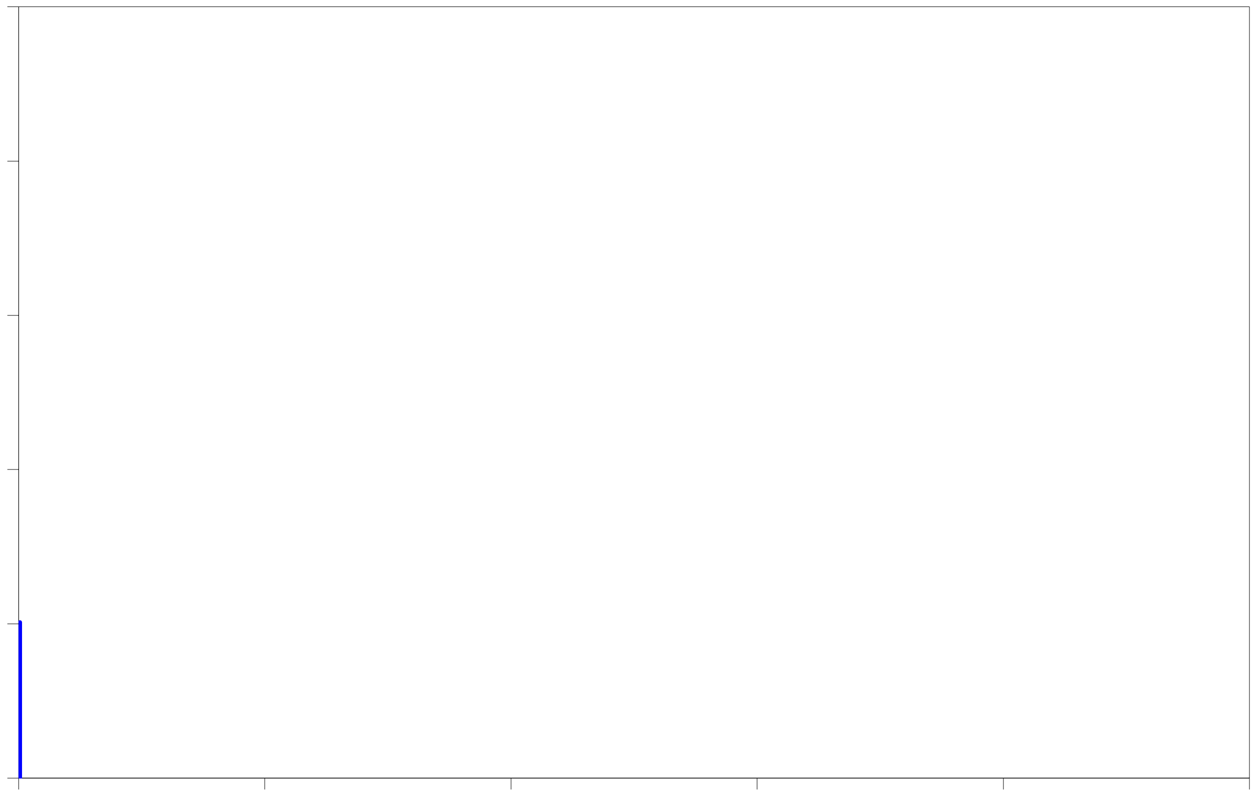
Isothermal Tests – 180C-1012015



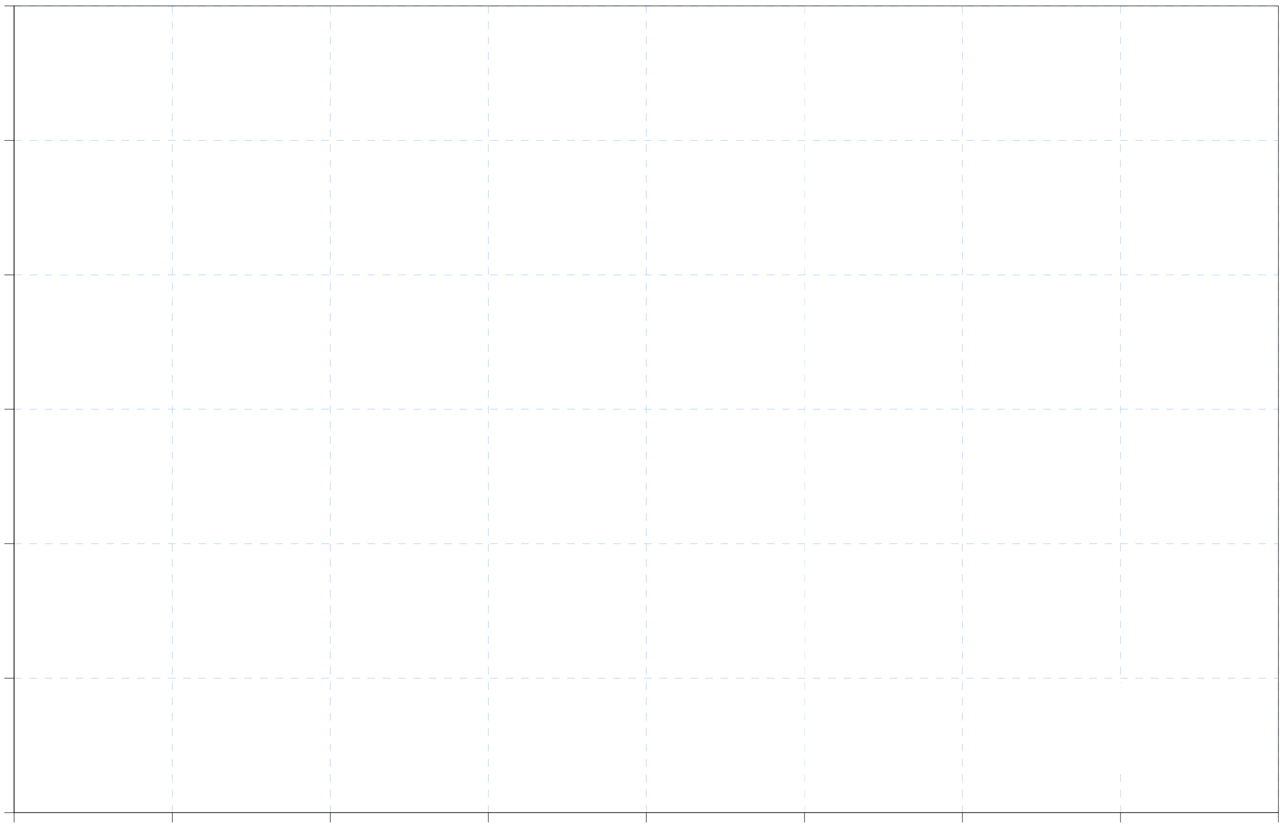
Isothermal Tests – 180C-1



Isothermal Tests – 190C



Material Model Verification – 100C



Material Model Verification – 1Cpm