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)
  - Fabric Prepreg: AGP193P/8552S, 38%, 193AW, 60" (received Oct 18, 2006)

- Cure cycle suggested by Manufacturer (US version) is as follows:
  - Heat at 3-5ºF/min to 225ºF
  - Hold at 225ºF for 30-60 min
  - Heat to 350ºF at 3-5ºF/min
  - Hold at 350ºF for 120±10 min
  - Cool at 2-5ºF/min 150ºF

- The tests were done on resin and comparison drawn with other forms

Cure Kinetics Model

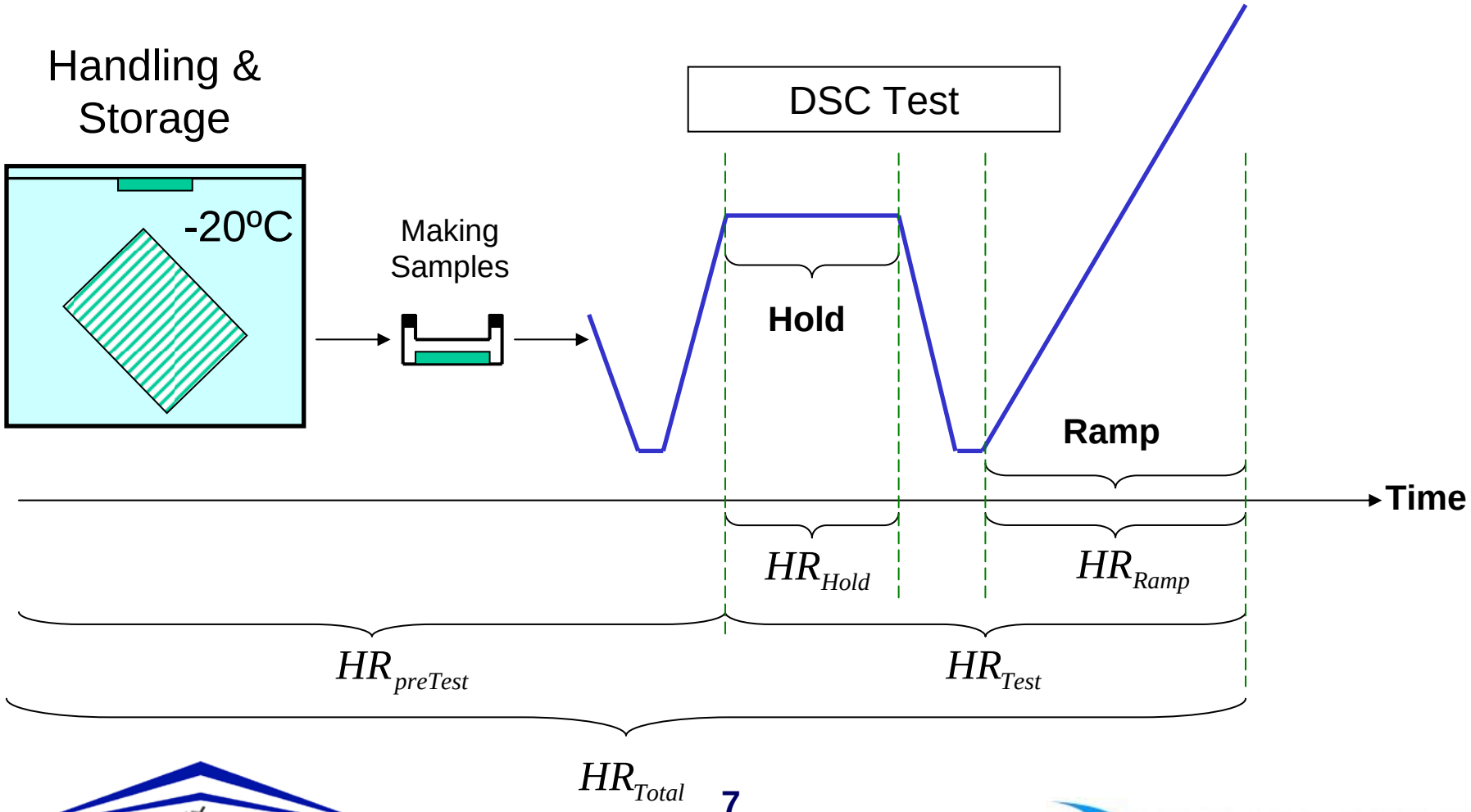
Nomenclature

Iso	or Isothermal: Conditioned isothermal DSC tests where the specimen is held at a constant temperature long enough so that the reaction slows down significantly due to diffusion
Int	or Interrupted: Isothermal tests where the hold segment terminates before the reaction stops due to diffusion
Dyn	or dynamic: DSC tests where the specimen temperature is increased at a constant rate until the reaction completes
RES	or Residual: Dynamic DSC tests performed after the hold segment of Iso and Int tests to complete the remainder of the chemical reaction
CC	or CureCycle: DSC tests designed to recreate the recommended cure cycles for the material, interrupted at certain points to investigate the cure advancement during the cure cycle
x	or DoC: Degree of Cure
x_{Hold}	Degree of cure at the end of the hold segment of isothermal tests
$\dot{x}$	or xdot: Cure Rate
T_g	Glass transition temperature

Nomenclature

HF	Heat Flow, measured in the DSC experiments (W)
HR_{Ramp}	The heat of reaction released during the ramp segment of the DSC tests (in dynamic tests and the residual ramps of the isothermal tests) (J/g)
HR_{Hold}	The heat of reaction released during the hold segment of the DSC tests (mostly in all variation of isothermal tests) (J/g)
$HR_{preTest}$	The heat of reaction released during the cure advancement in material before the start of the test (material handling, storing, etc.) (J/g)
HR_{Test}	The total heat of reaction measured through the two stages of the DSC test (J/g) $\left(= HR_{Hold} + HR_{Ramp}\right)$
HR_{Total}	The total heat of reaction of the material tested (J/g) $\left(= HR_{preTest} + HR_{Test}\right)$
HR_{Iso}^{avg}	The average of the total heat of reaction measured through the various stages of the isothermal DSC tests (J/g)
HR_{Dyn}^{avg}	The average of the total heat of reaction measured through the various stages of the dynamic DSC tests (J/g)
HR_{Total}^{avg}	The average of the total heat of reaction measured through the various stages of all the DSC tests (J/g)
HR_{Model}	Model heat of reaction; the nominal value of total heat of reaction used in the model (J/g)

HR Definition



Summary

• Differential Scanning Calorimetry (DSC) tests were performed to characterize the cure kinetics of Hexcel 8552. A Q1000 TA Instrument DSC was used for this purpose.

• Normal pans were used to perform the DSC tests on neat resin.

• Characterisation was performed on the resin. Other forms of this resin (film and fabric prepreg) were also tested and compared to the neat resin.

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 equilibrated at a very low temperature and then heated up at a predetermined rate. The goal is to fully cure the sample on the ramp. A second ramp follows to determine the final glass transition temperature (fully cured Tg).

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 complete temperature range (100ºC to 190ºC) was consistent. However, a number of tests were dismissed due to anomalies and the remaining isothermal and dynamic tests (see table on next slide) were considered in the analysis.

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

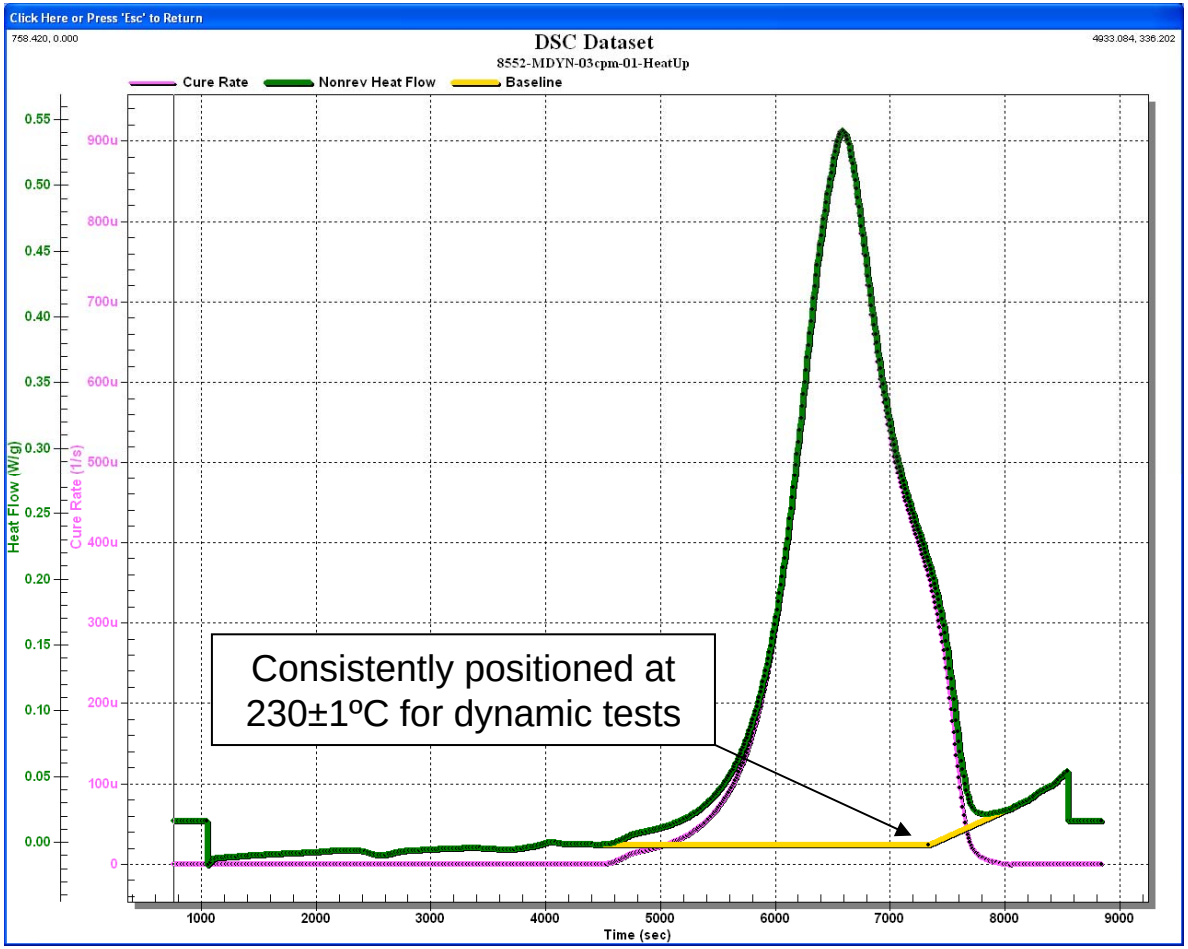
Test	Mass (mg)	Hold	
		Temp (ºC)	Time (min)
8552-MISO-100C-01	5.3	100	720
8552-MISO-110C-01	5.7	110	540
8552-MISO-120C-01	4.7	120	480
8552-MISO-130C-01	4.4	130	420
8552-MISO-140C-01	5.2	140	360
8552-MISO-150C-01	5.6	150	360
8552-MISO-160C-01	5.4	160	300
8552-MISO-170C-01	3.6	170	180
8552-MISO-180C-01	5.59	180	180
8552-MISO-180C-02	5.4	180	180
8552-MISO-190C-01	4.49	190	180
8552-160C-INT15	5	160	15
8552-160C-INT30	0	160	30

Test	Mass (mg)
8552-CC-S1-02	4.6
8552-CC-S3-00-01	5
8552-CC-S3-60-01	4.7
8552-CC-S4-01	4.9

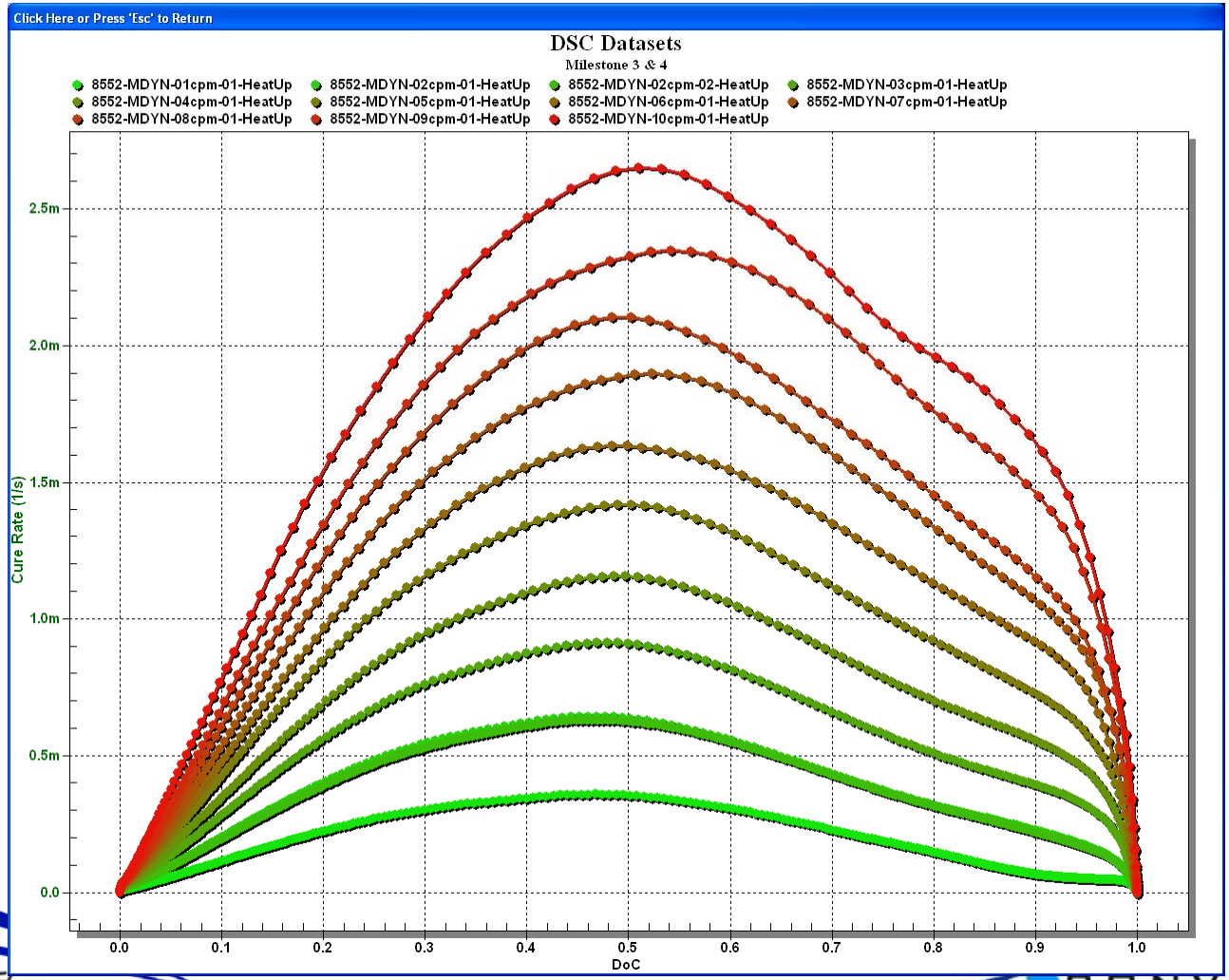
Degree of Cure Calculation

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 Isothermal tests were chosen to be linear.



Cure Rate vs. DoC – Dyns



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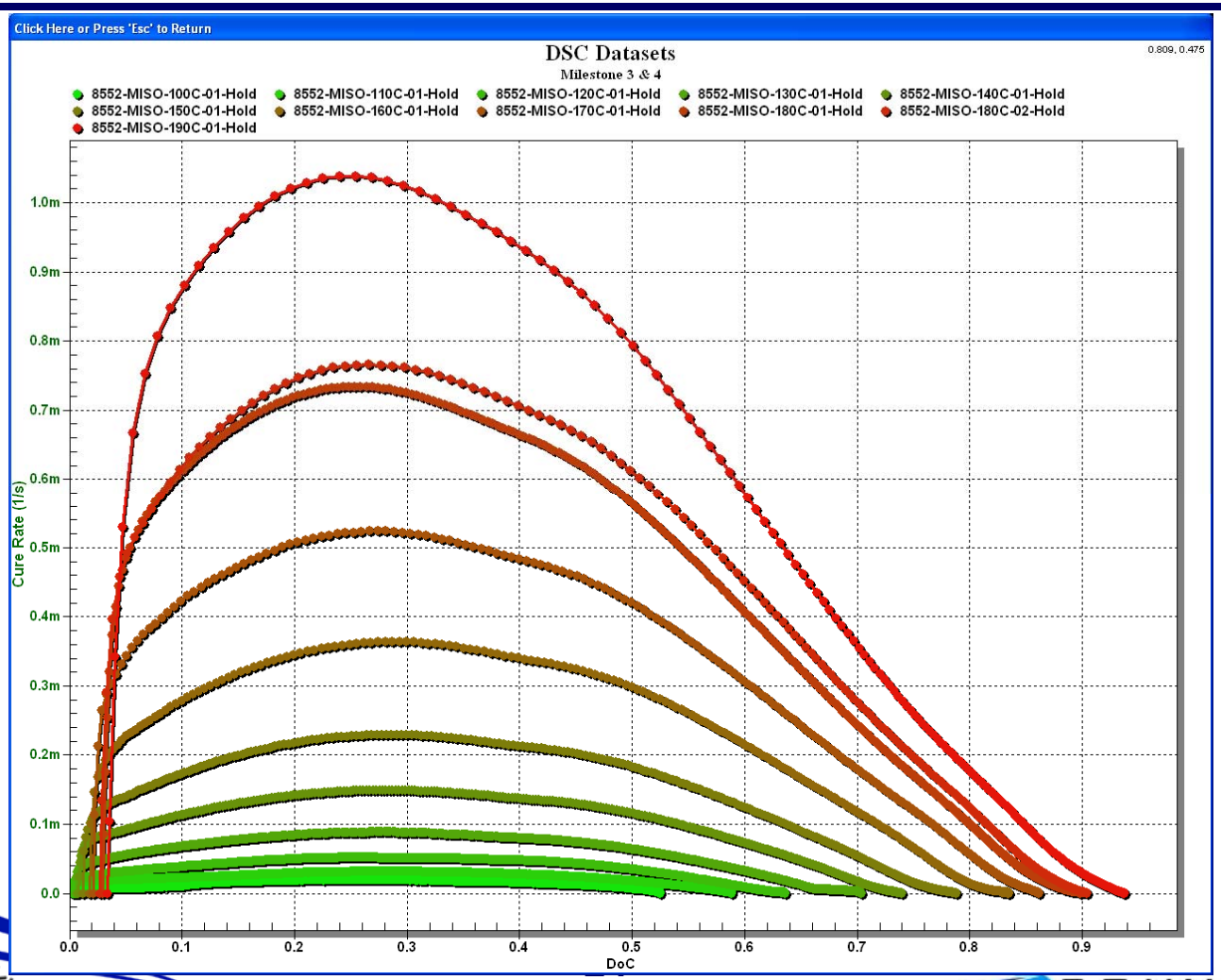
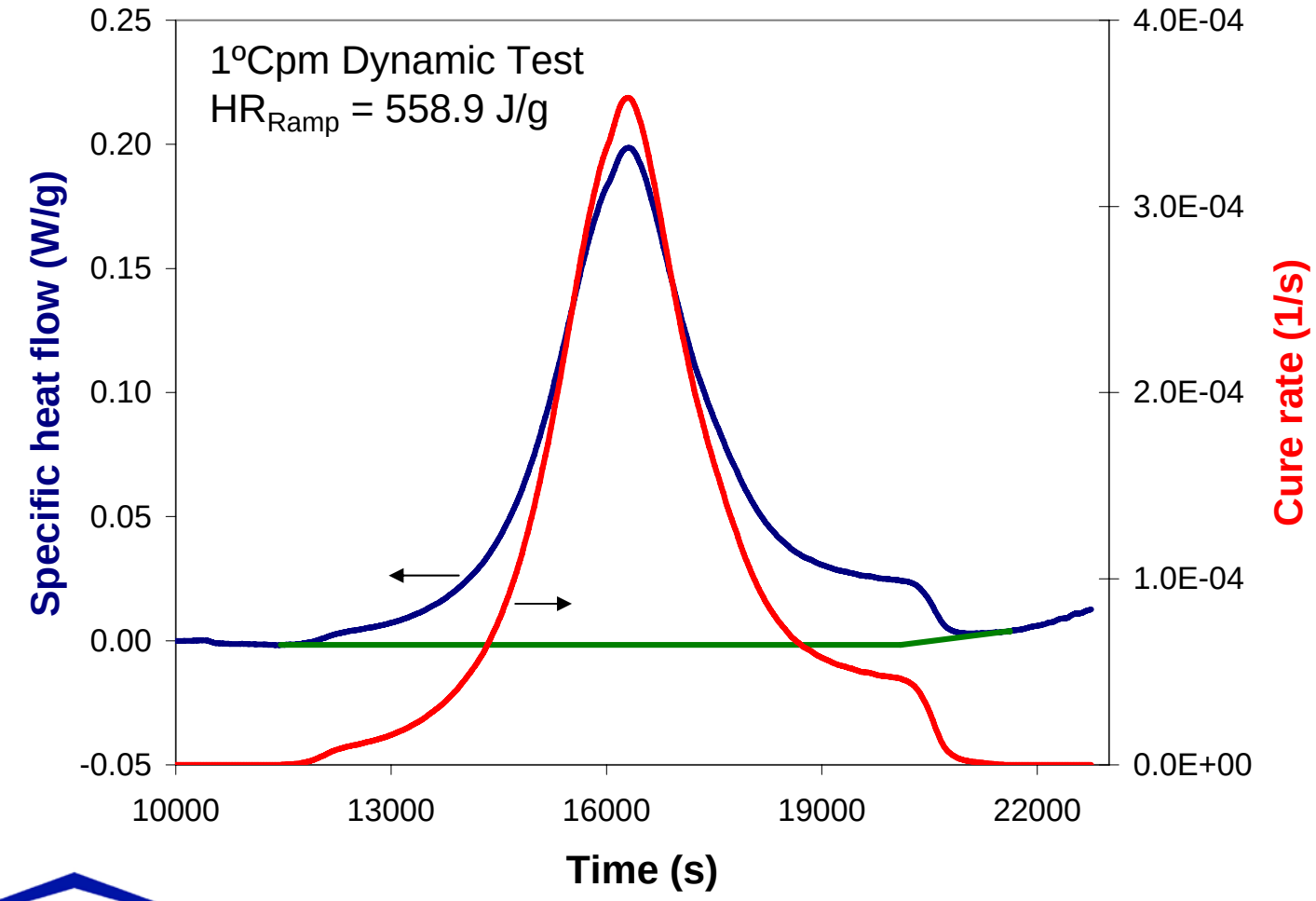
CONVERGENT MANUFACTURING TECHNOLOGIES

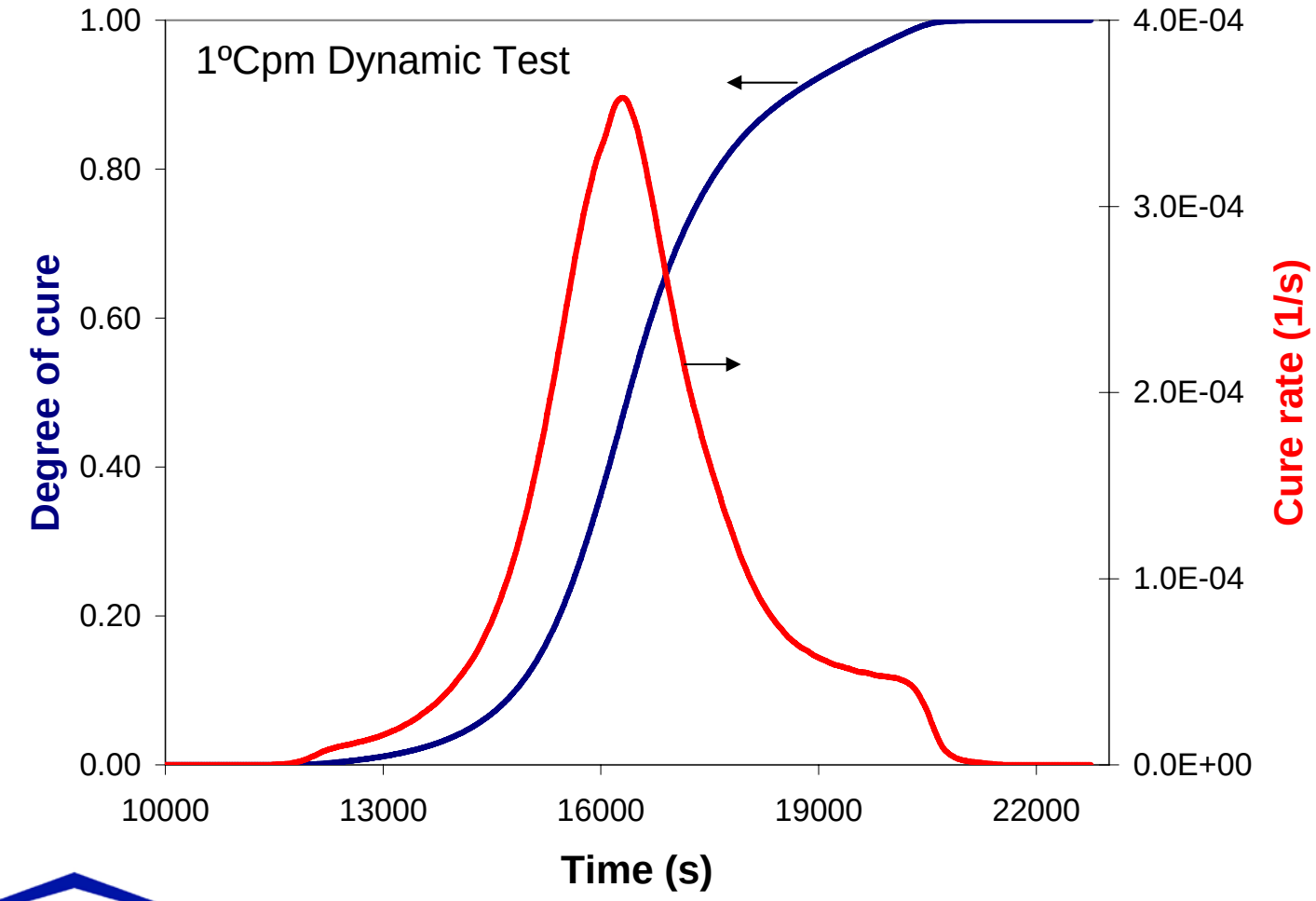
Cure Rate vs. DoC – Isos



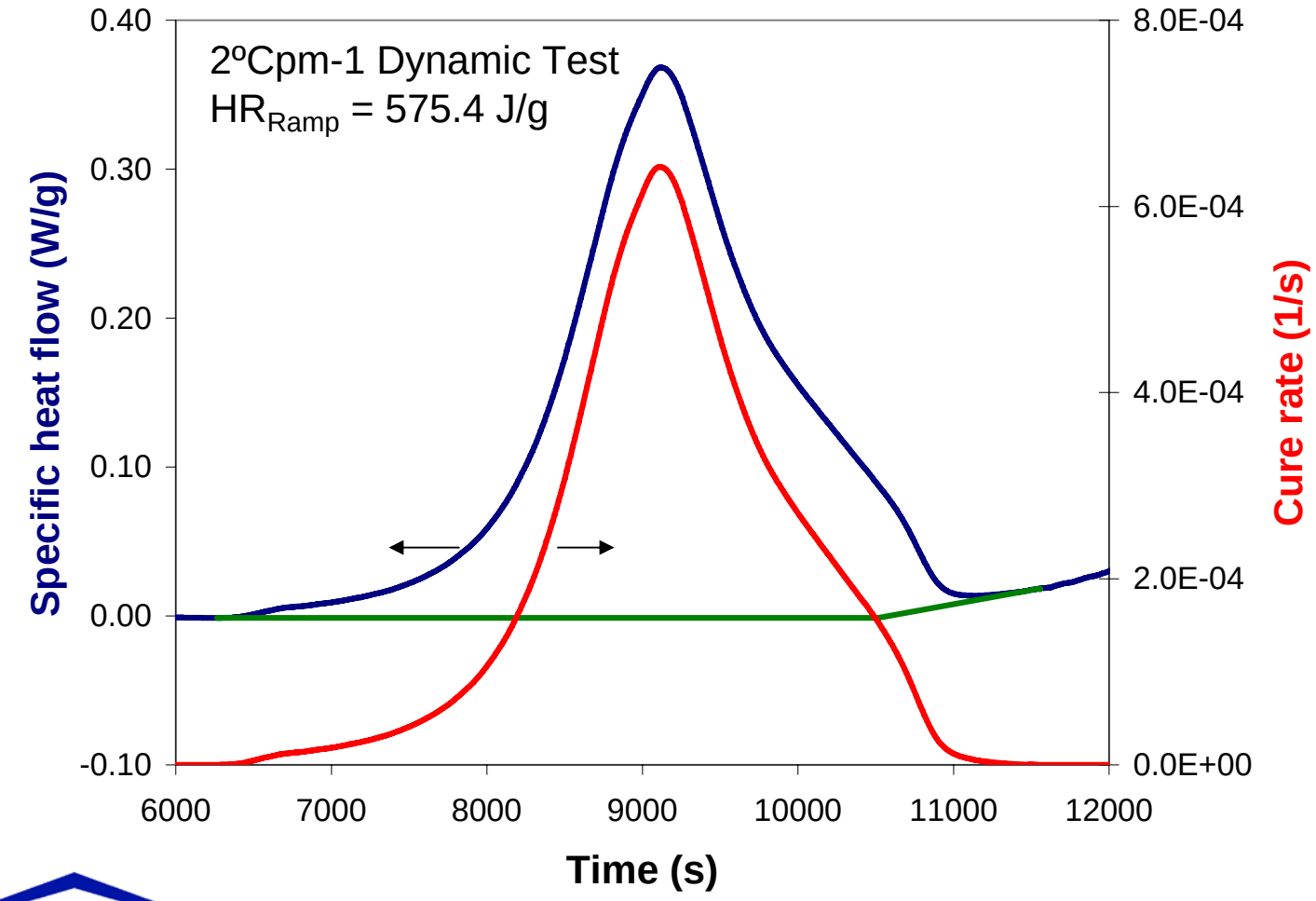
Heat Flow Response – 1cpm



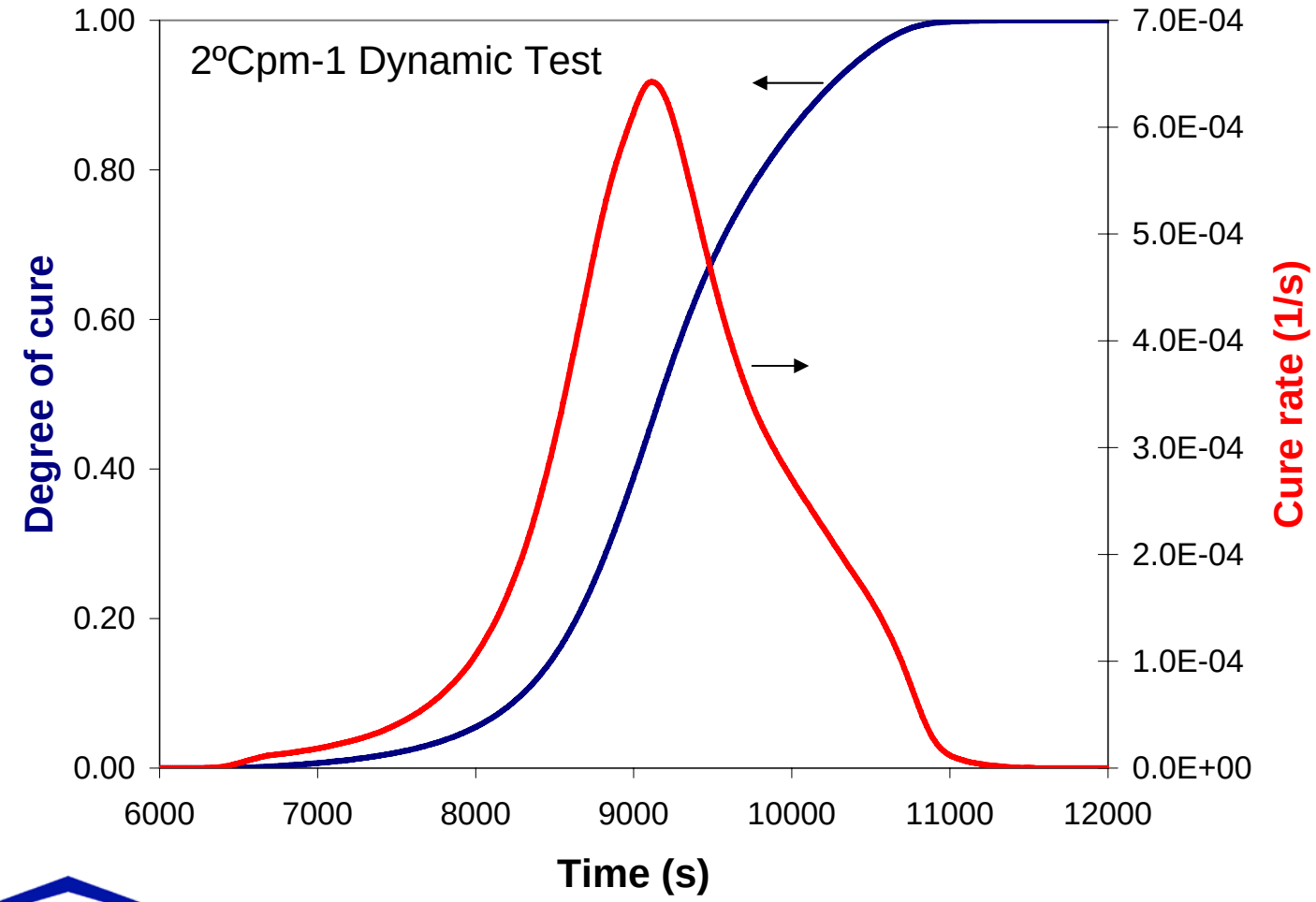
DoC and Cure Rate – 1cpm



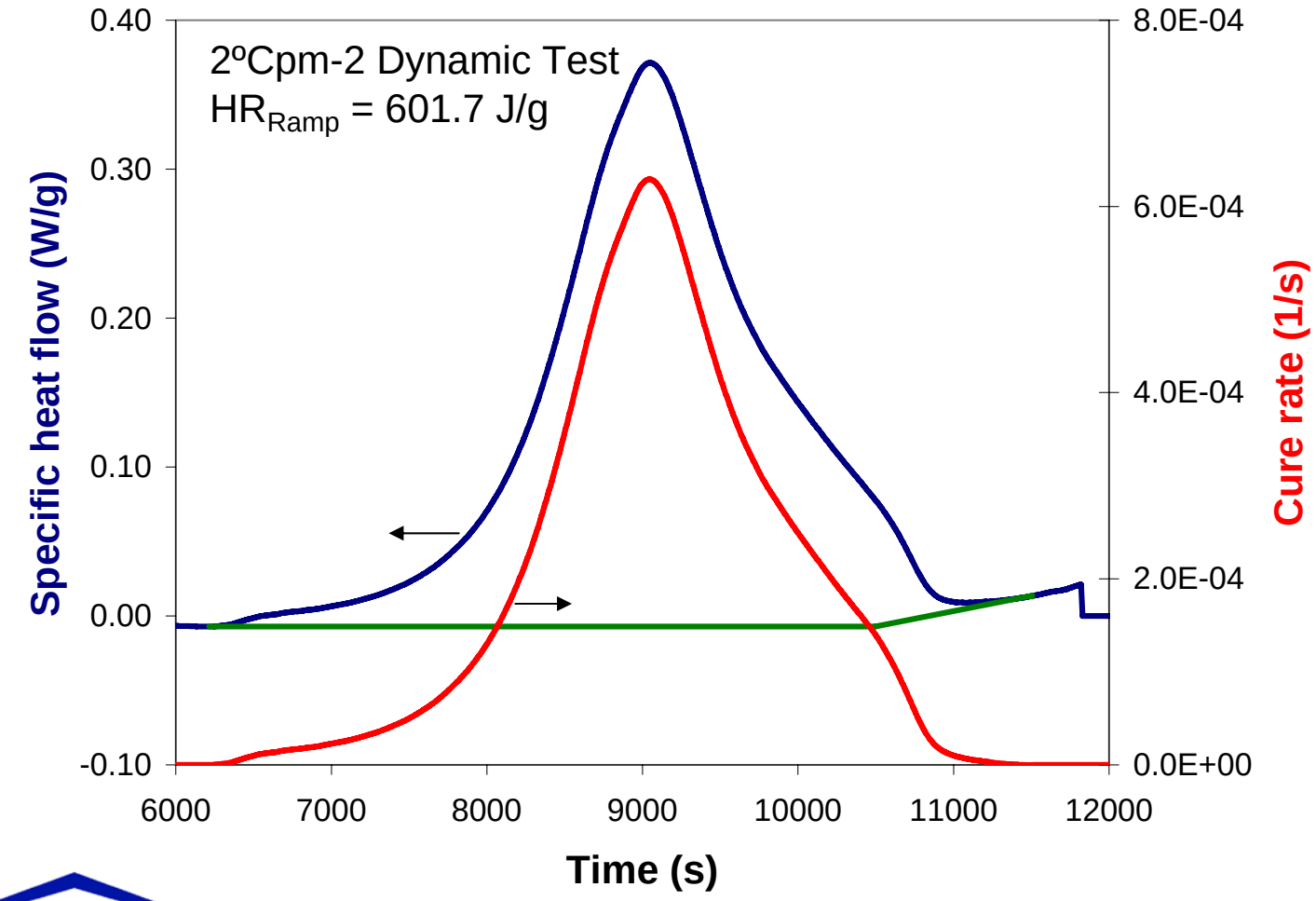
Heat Flow Response – 2cpm-1



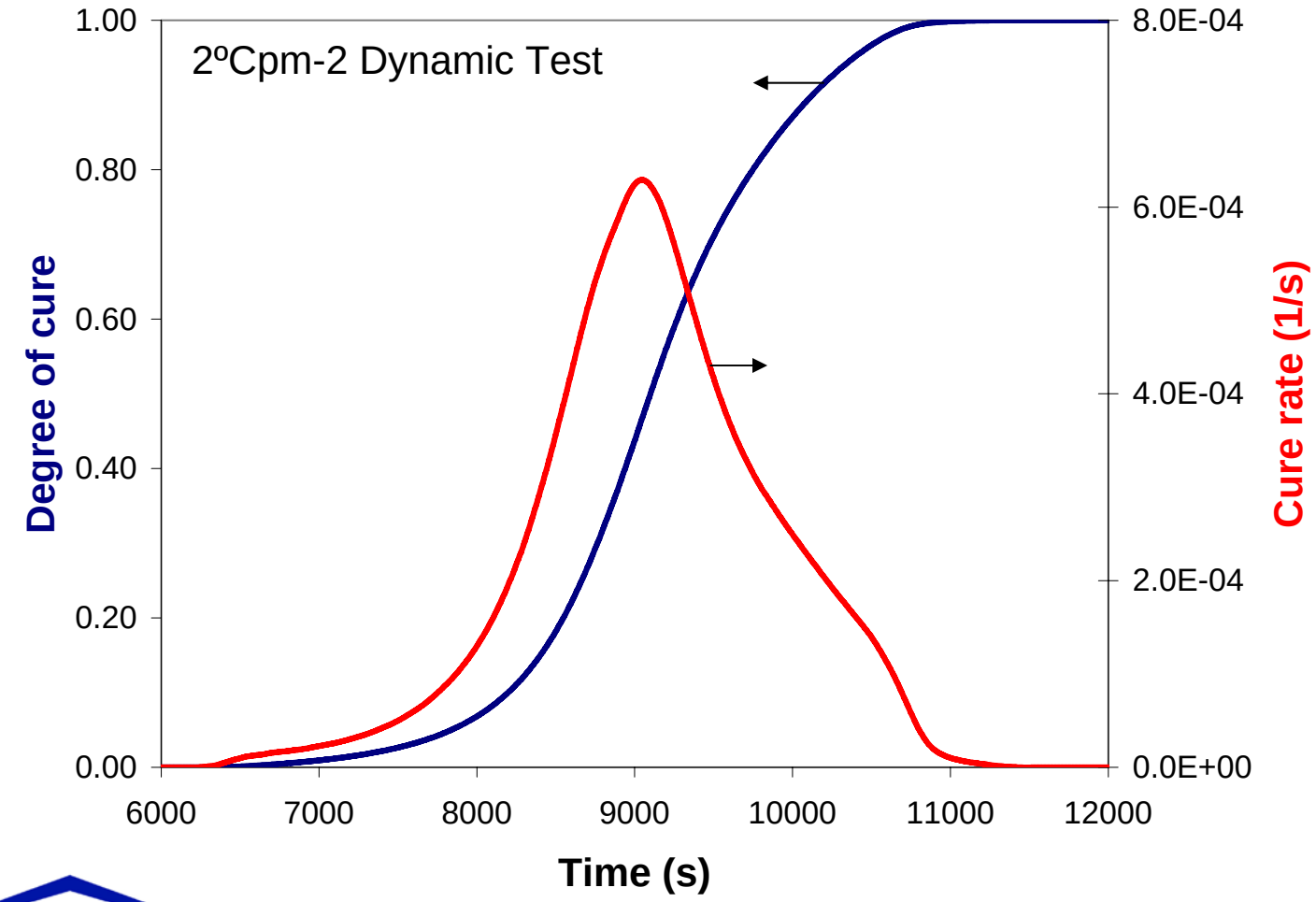
DoC and Cure Rate – 2cpm-1



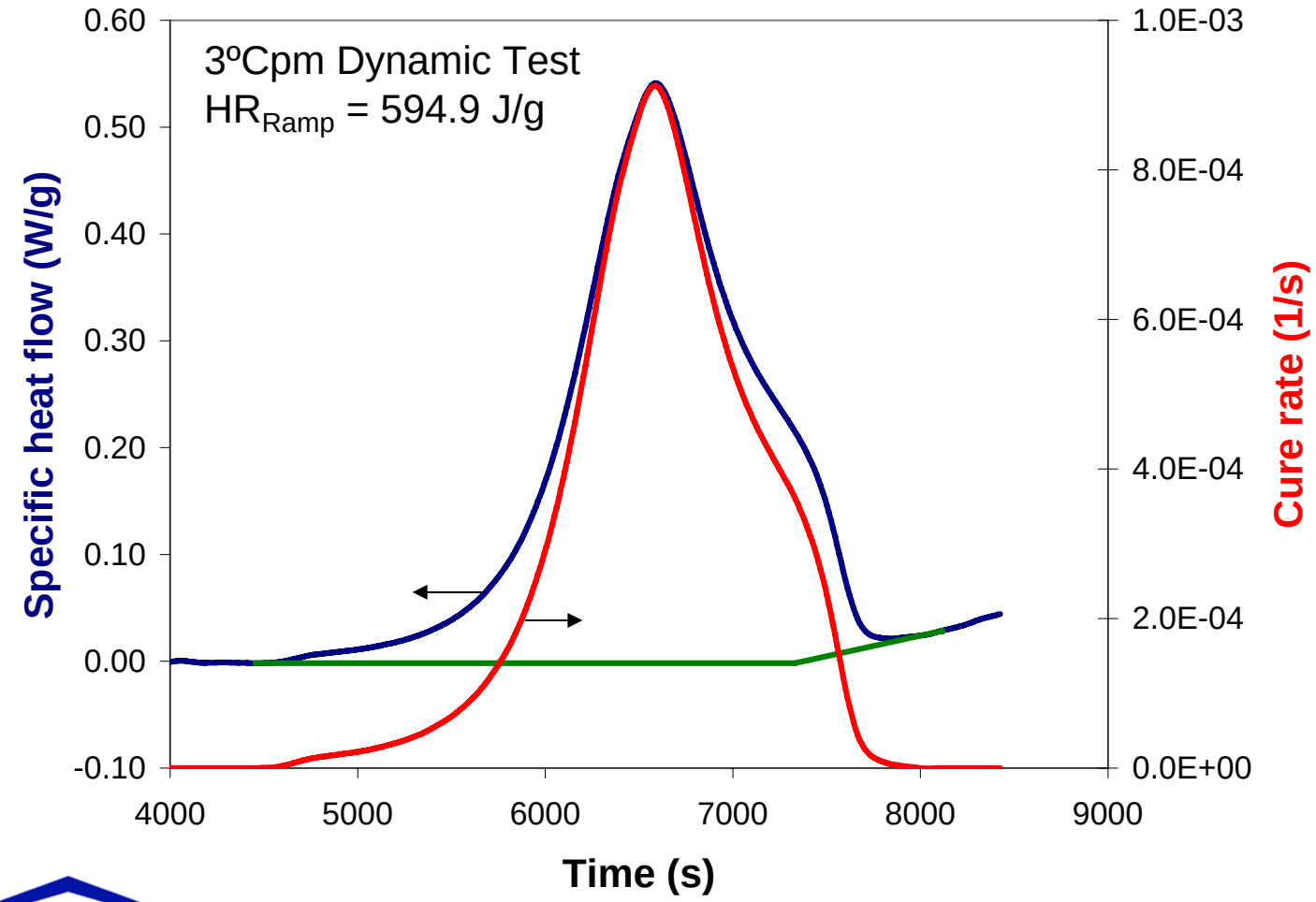
Heat Flow Response – 2cpm-2



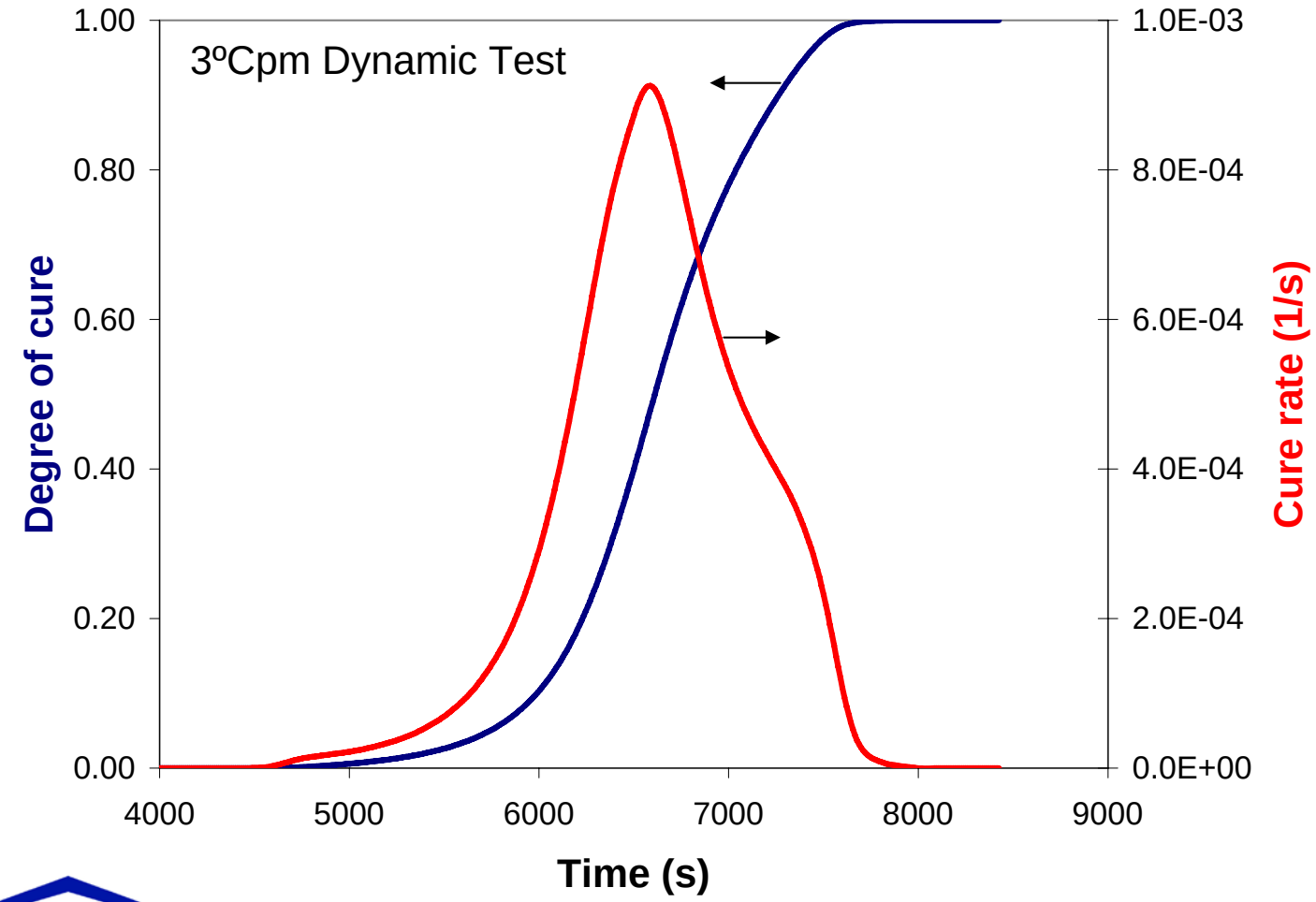
DoC and Cure Rate – 2cpm-2



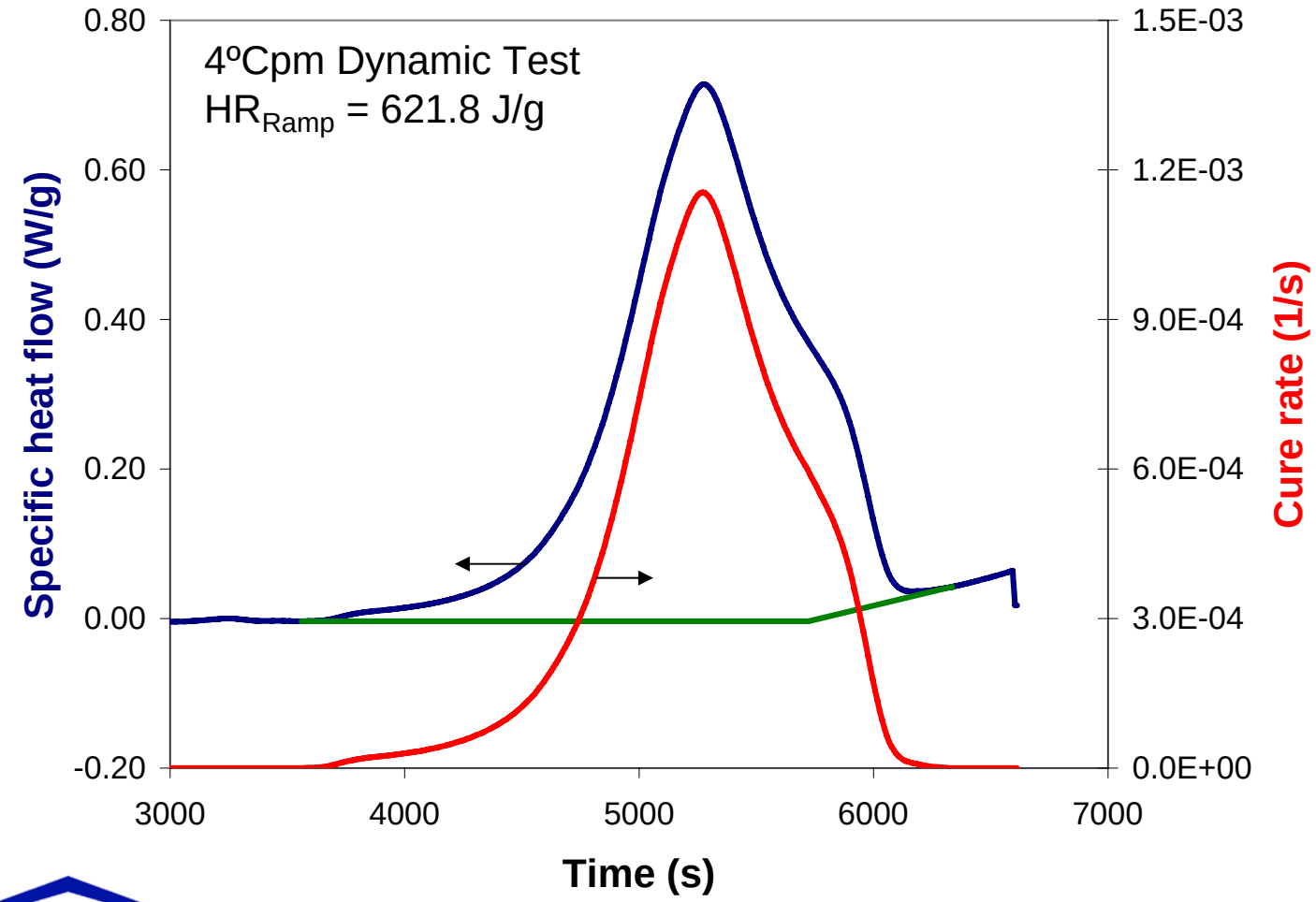
Heat Flow Response – 3cpm



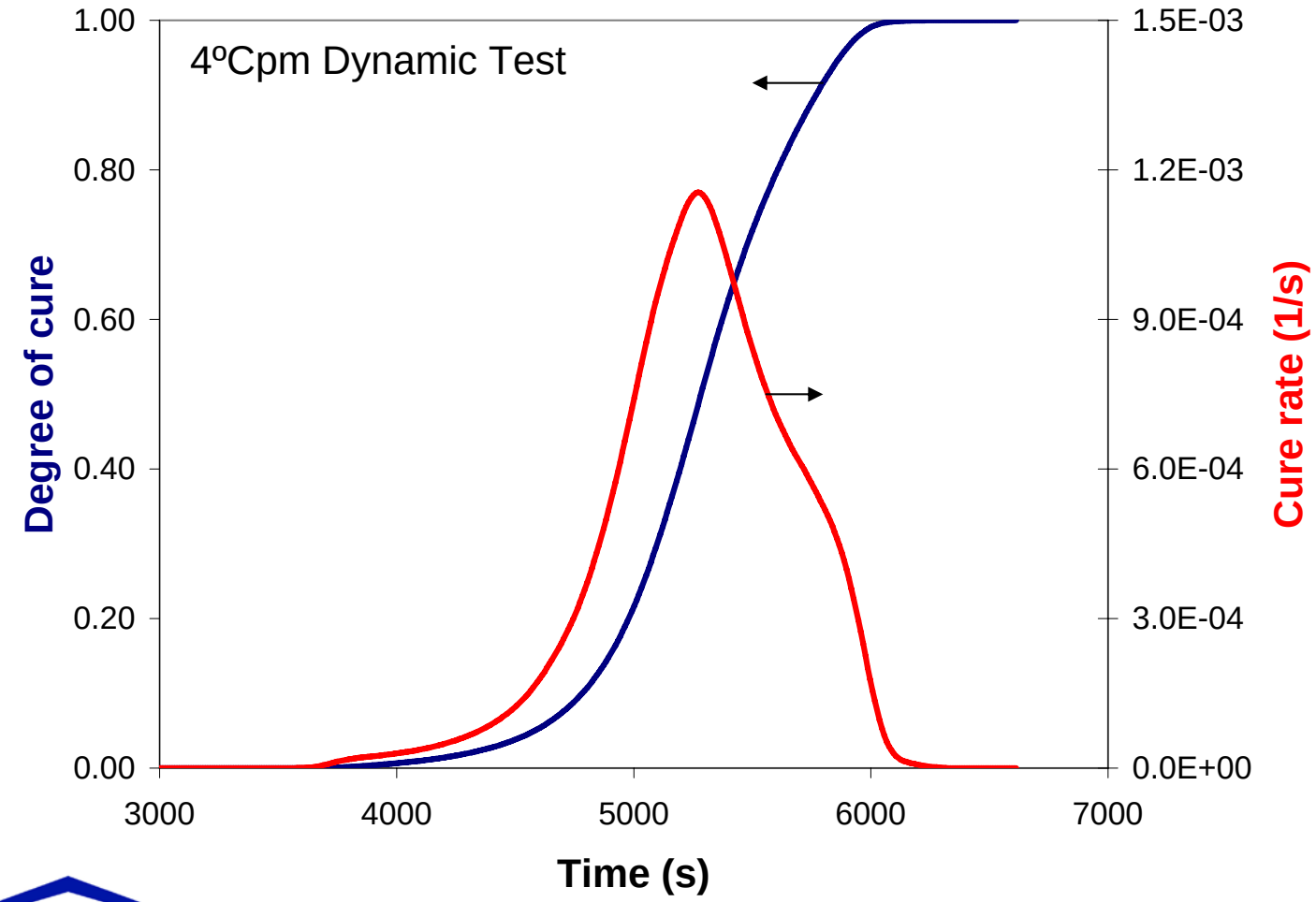
DoC and Cure Rate – 3cpm



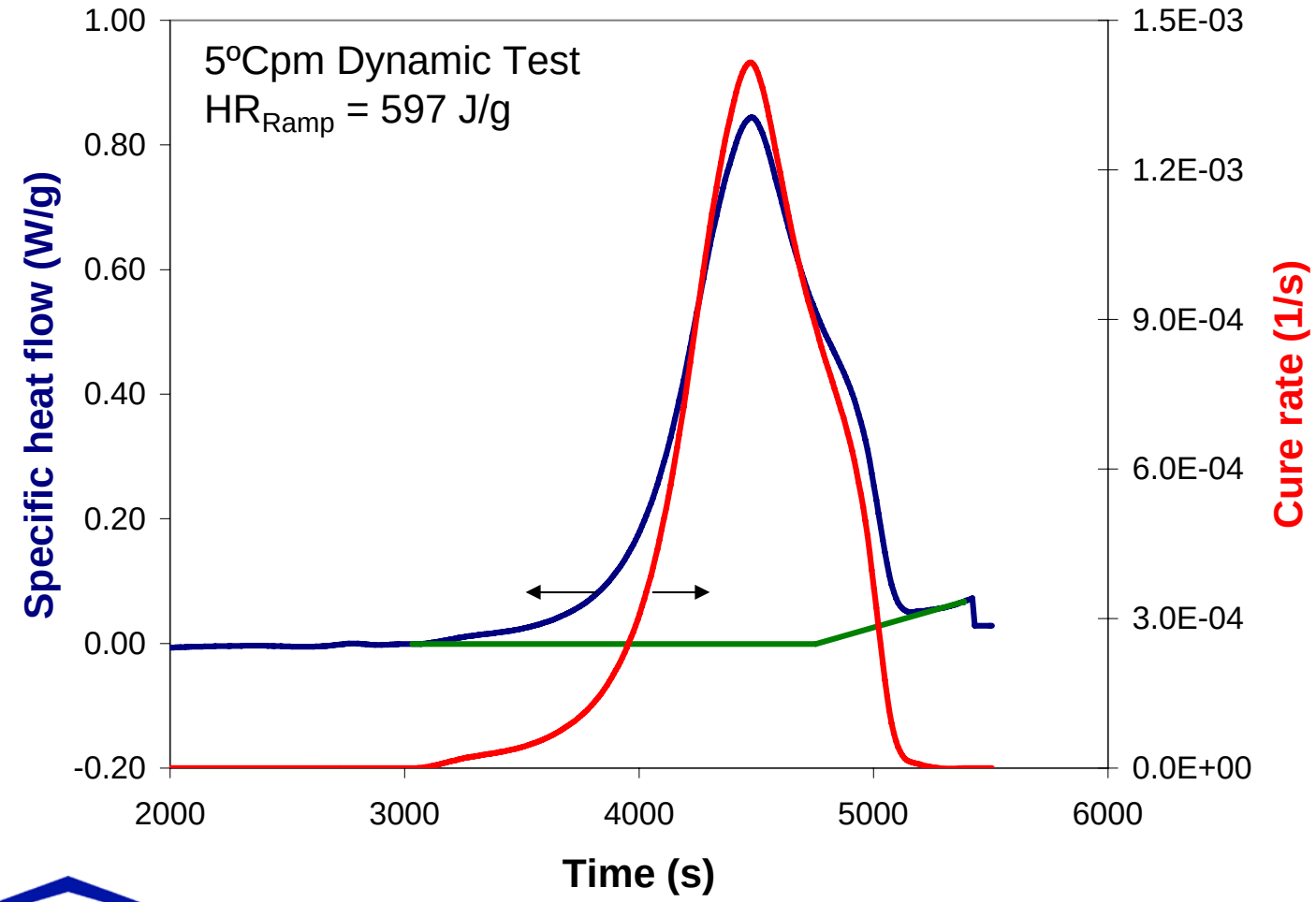
Heat Flow Response – 4cpm



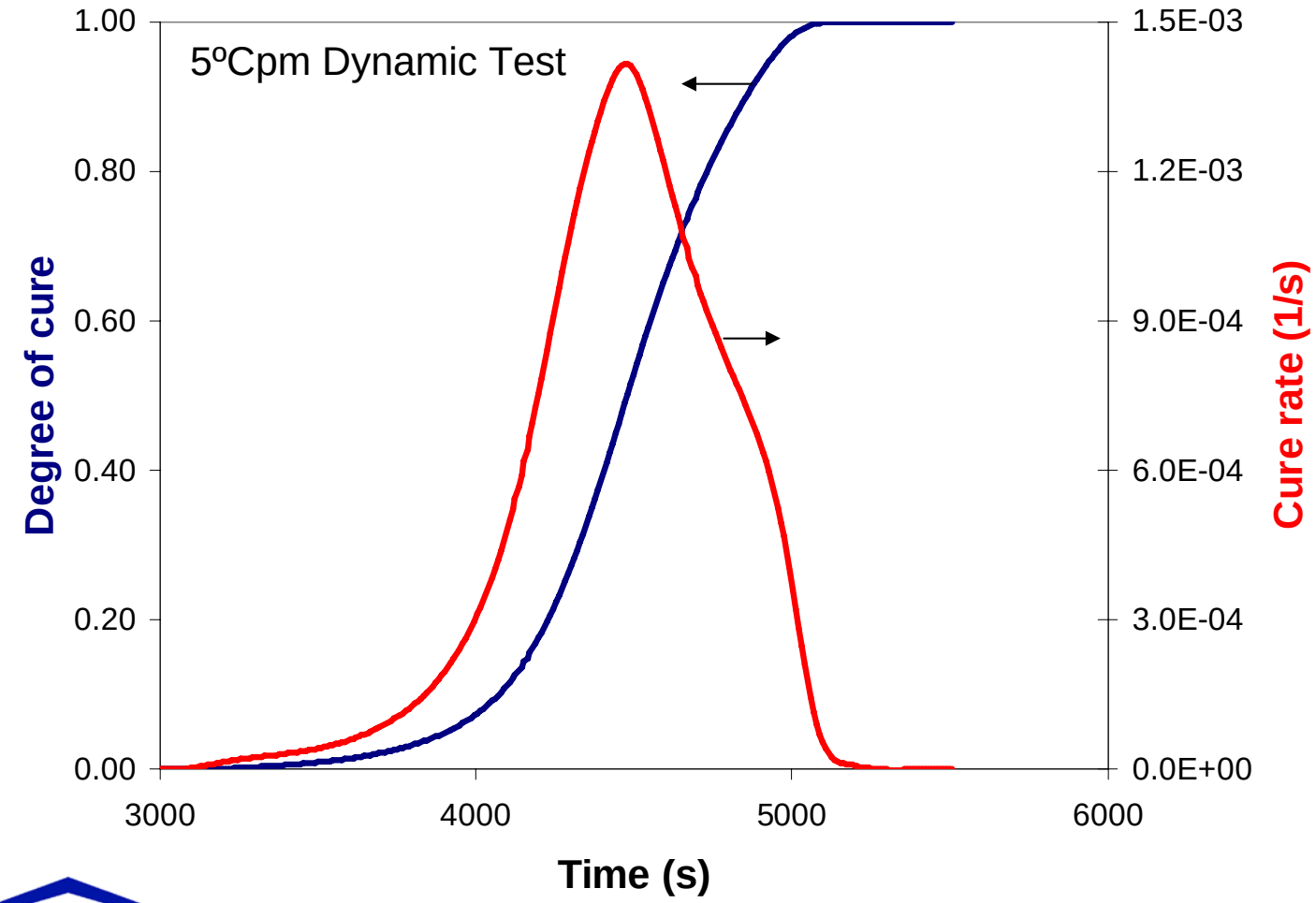
DoC and Cure Rate – 4cpm



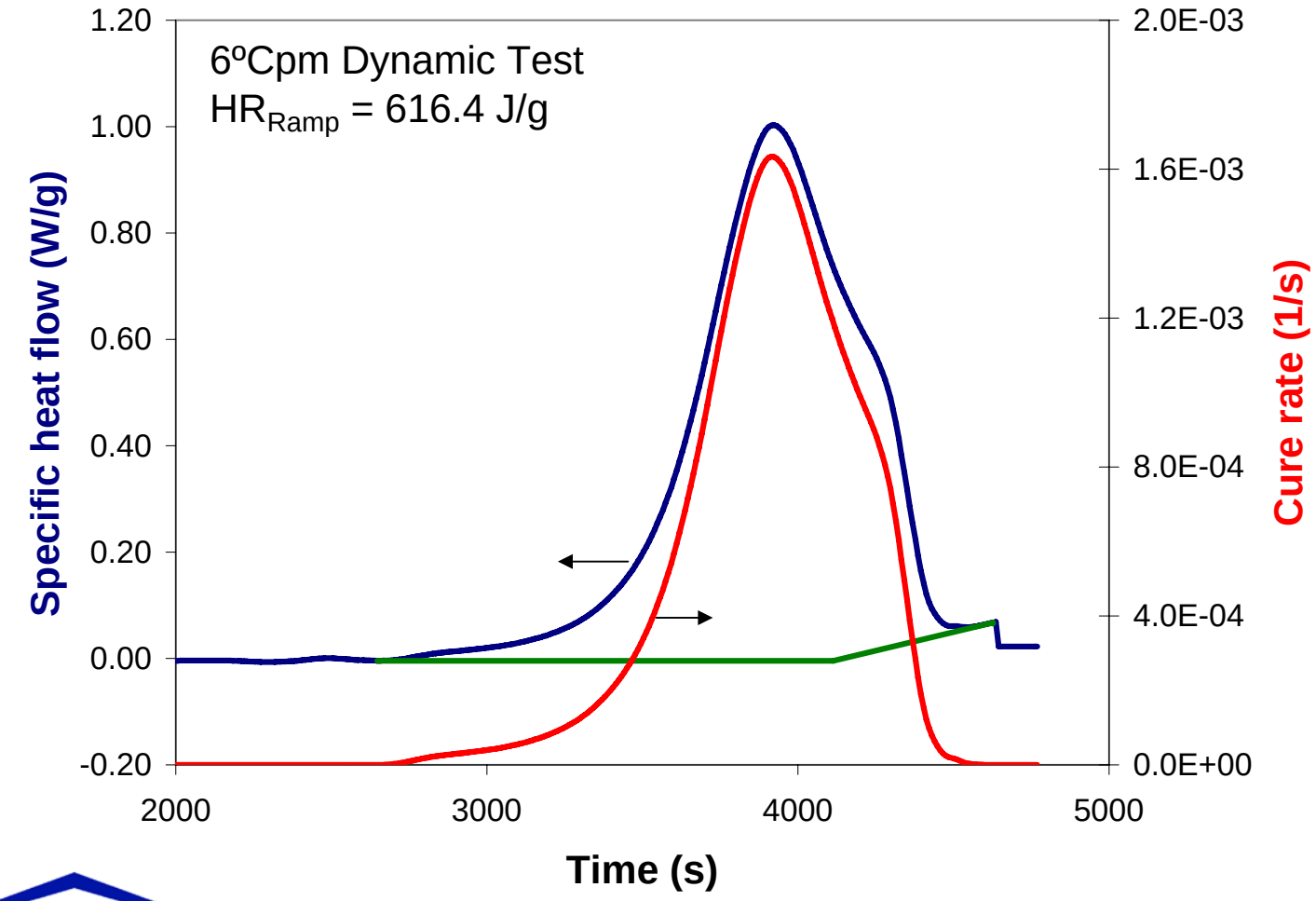
Heat Flow Response – 5cpm



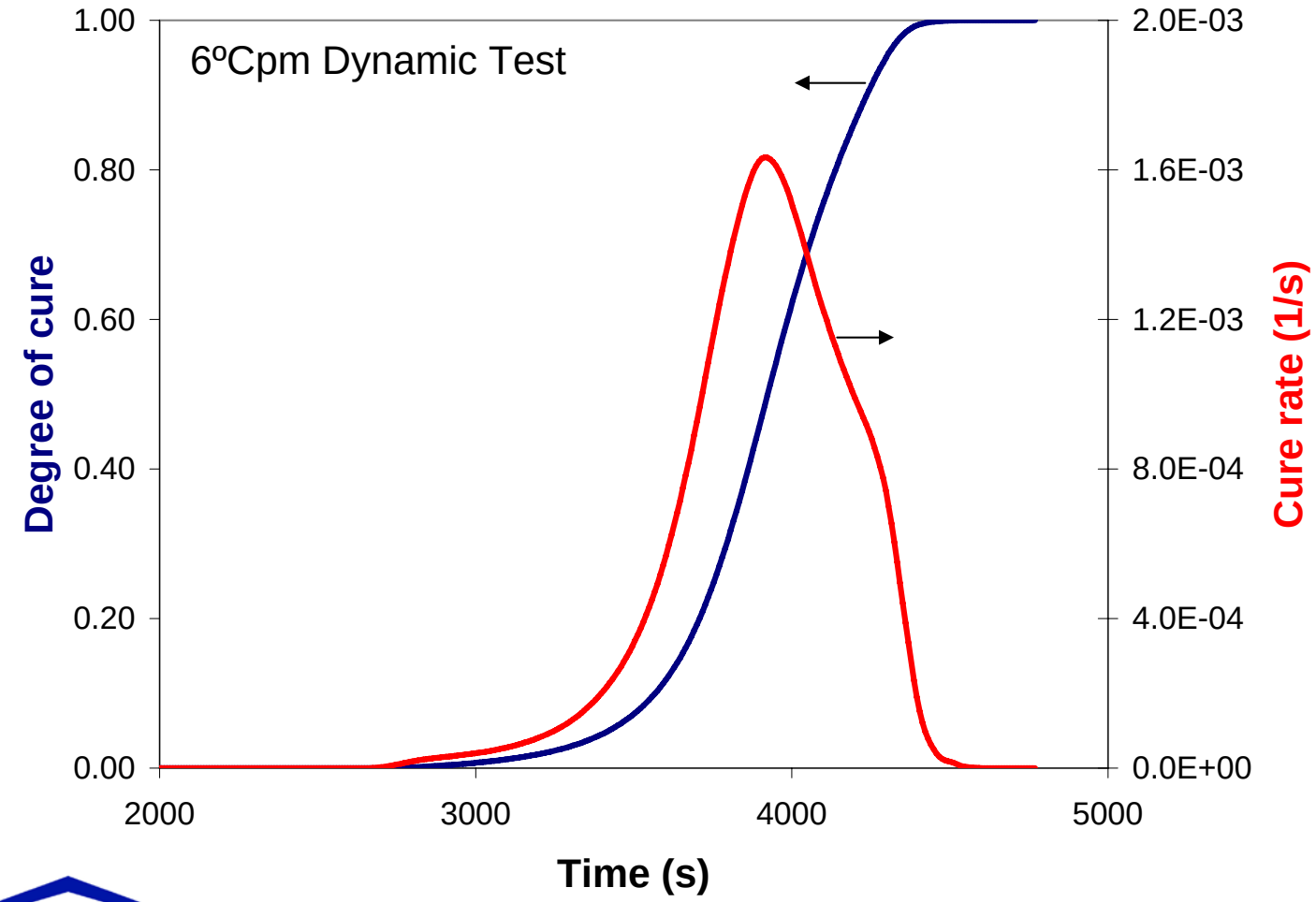
DoC and Cure Rate – 5cpm



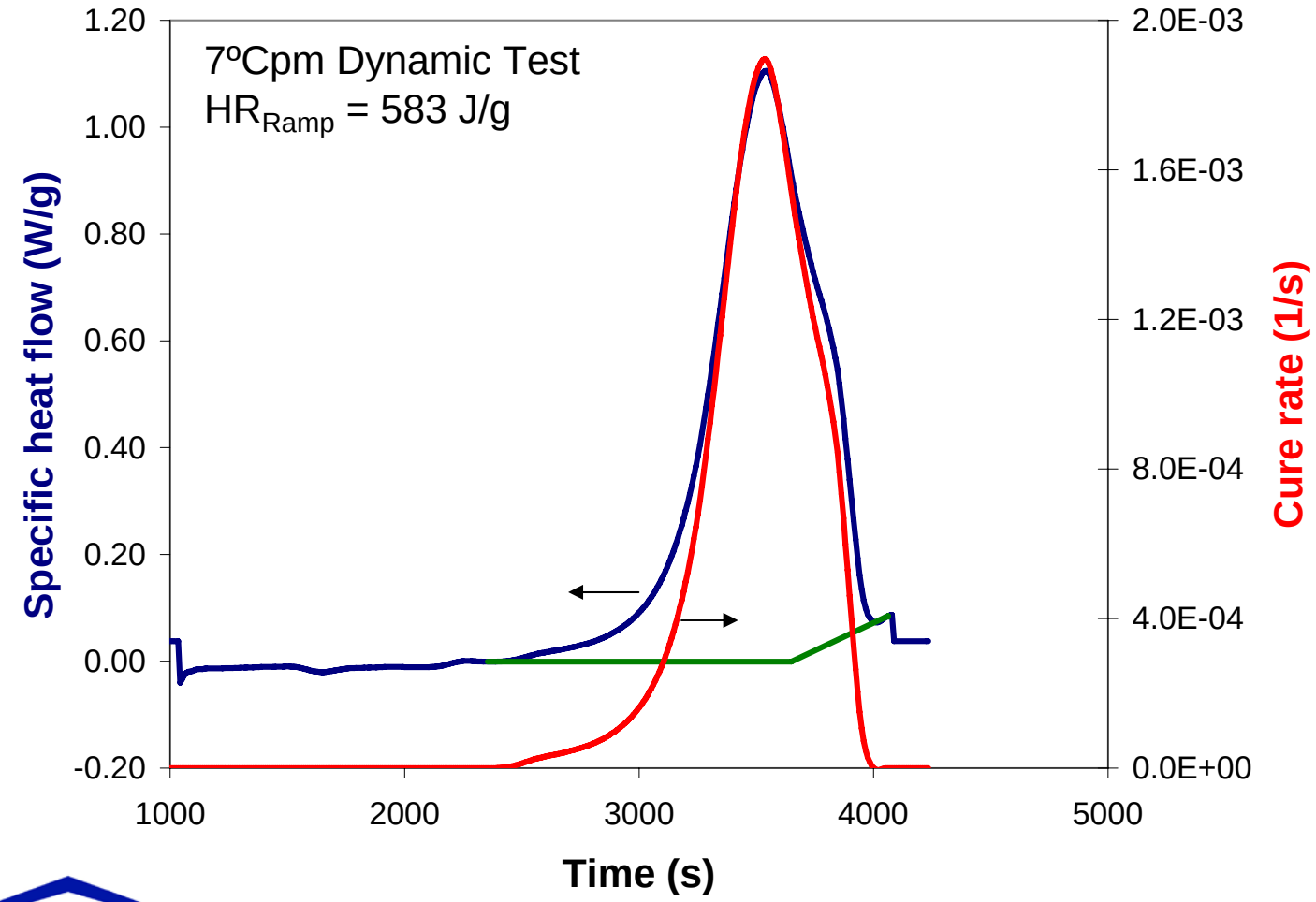
Heat Flow Response – 6cpm



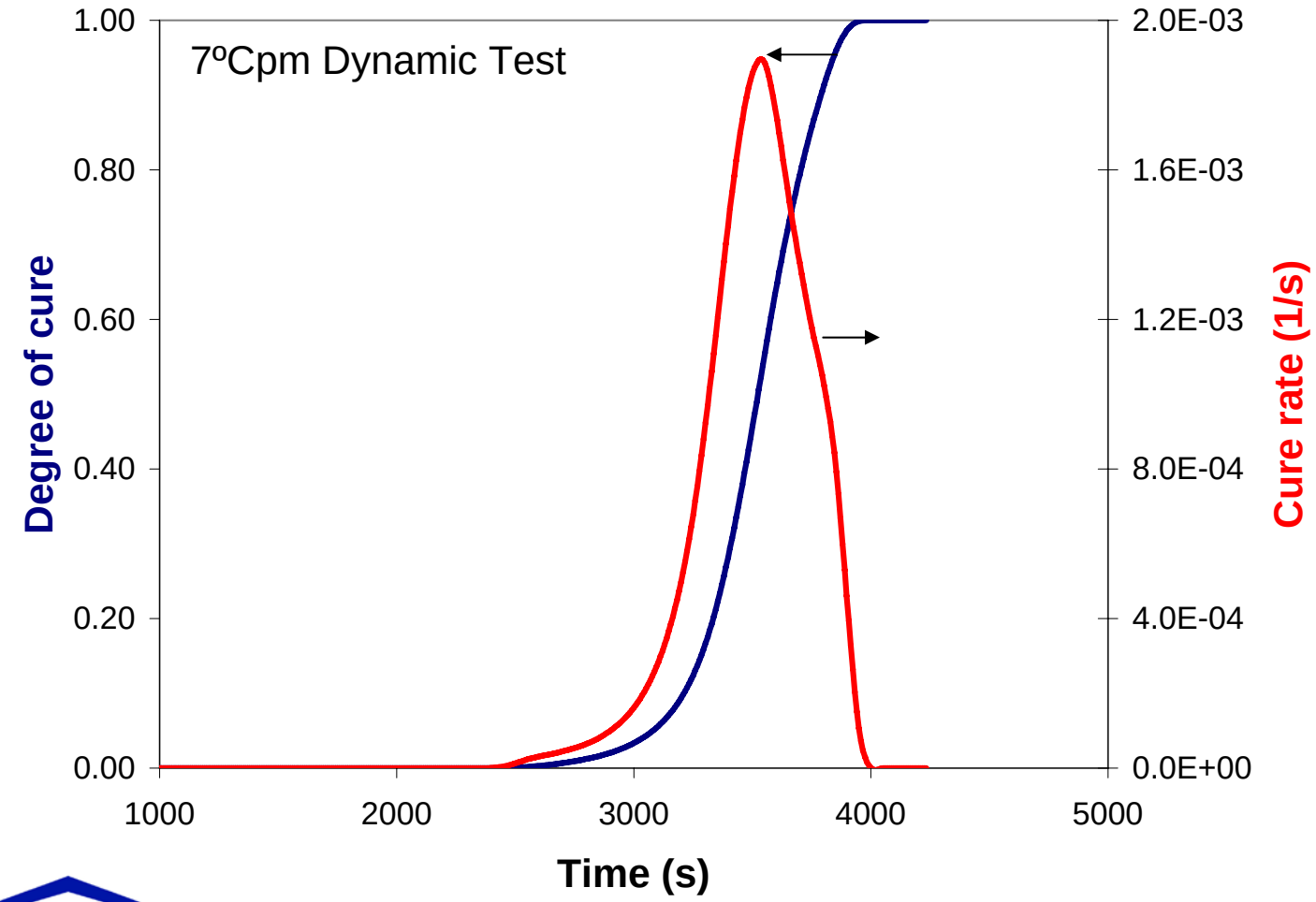
DoC and Cure Rate – 6cpm



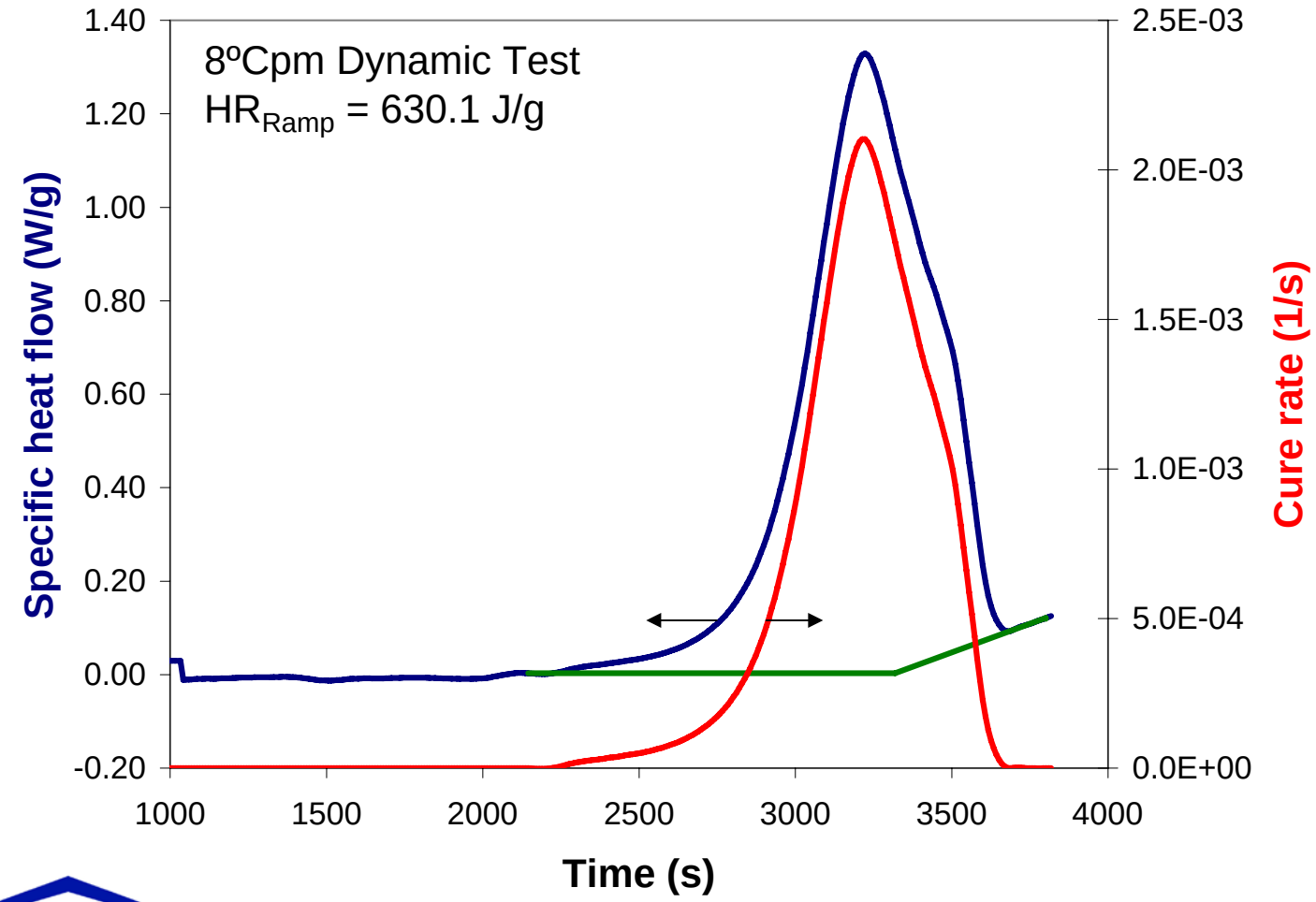
Heat Flow Response – 7cpm



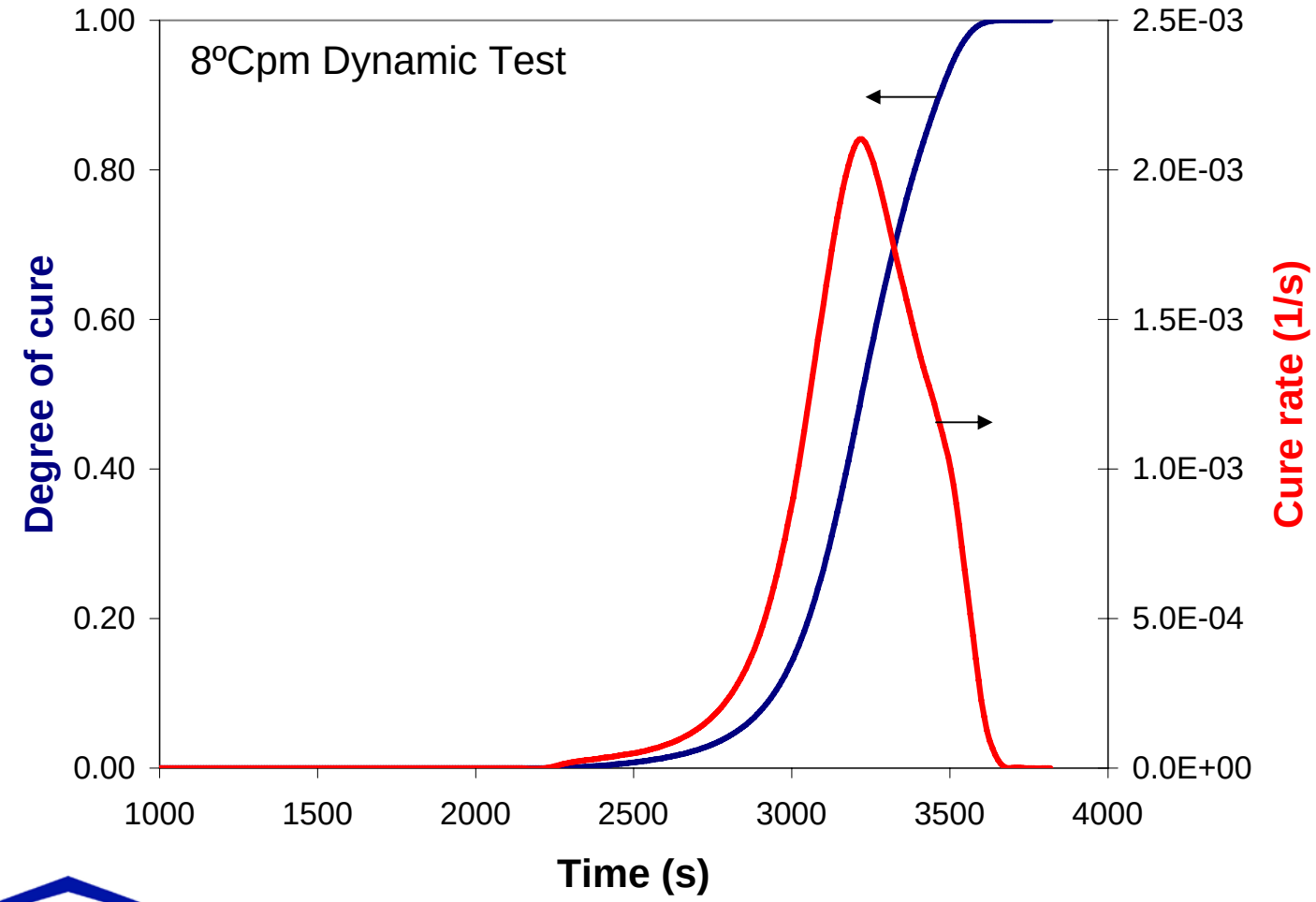
DoC and Cure Rate – 7cpm



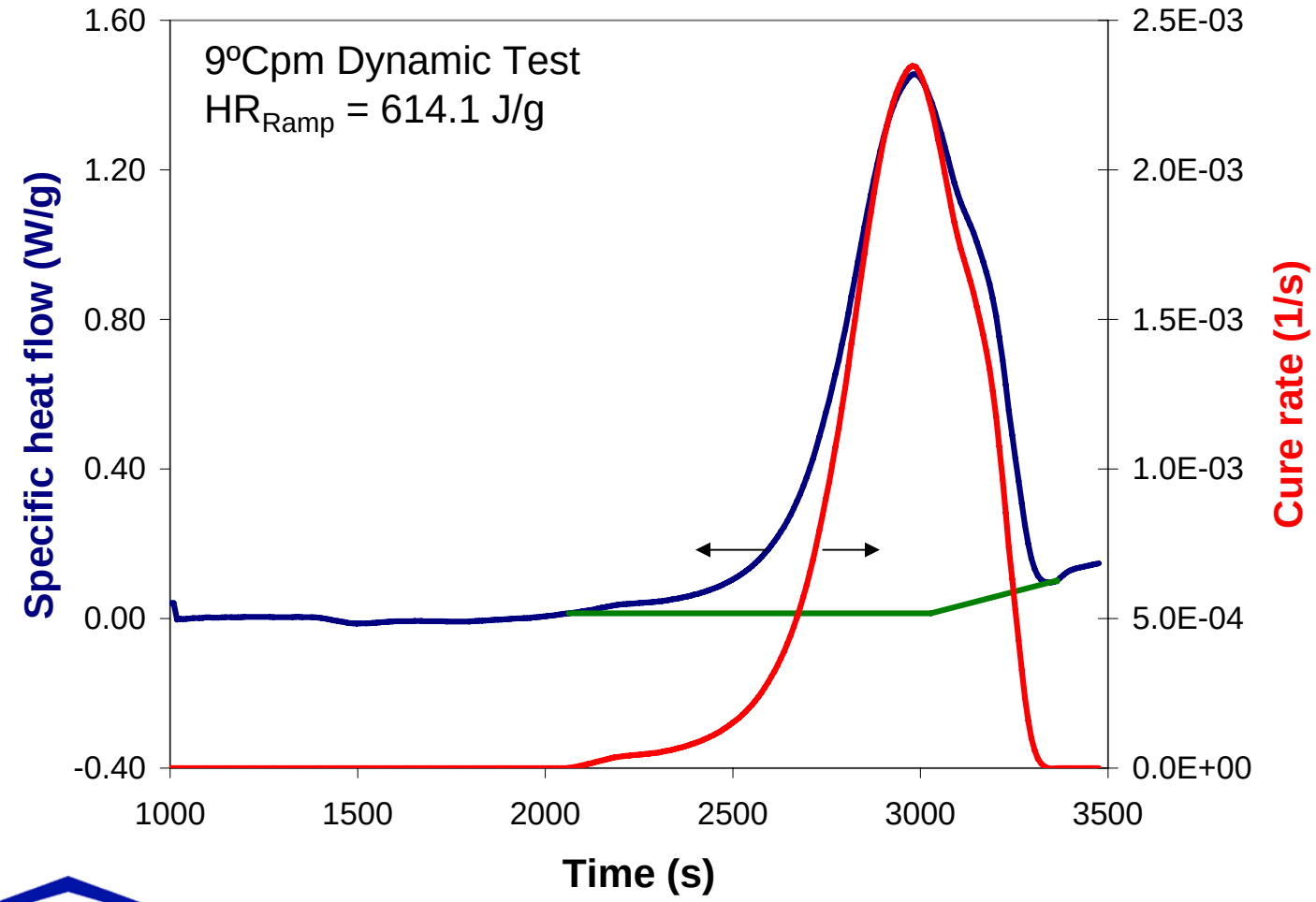
Heat Flow Response – 8cpm



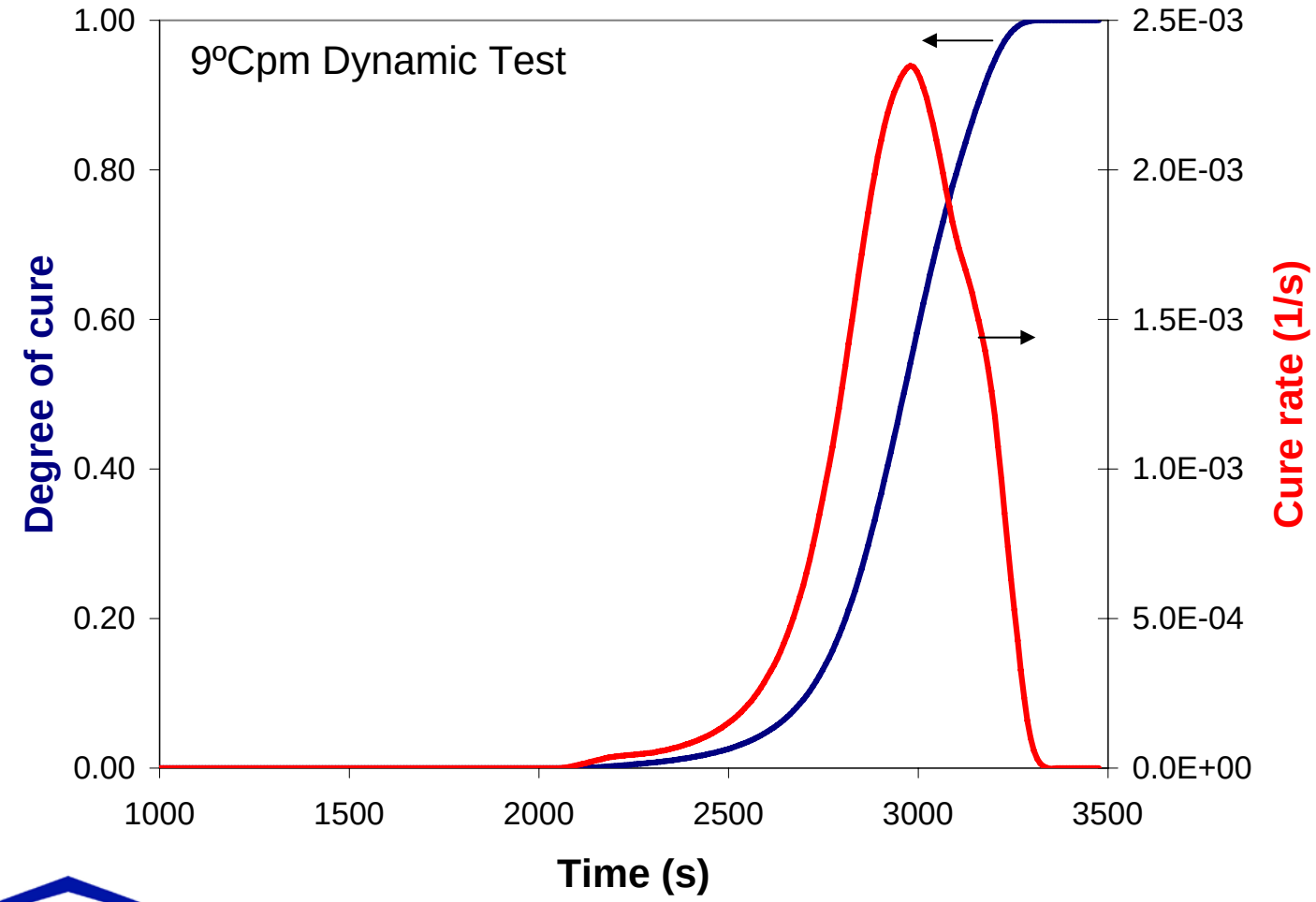
DoC and Cure Rate – 8cpm



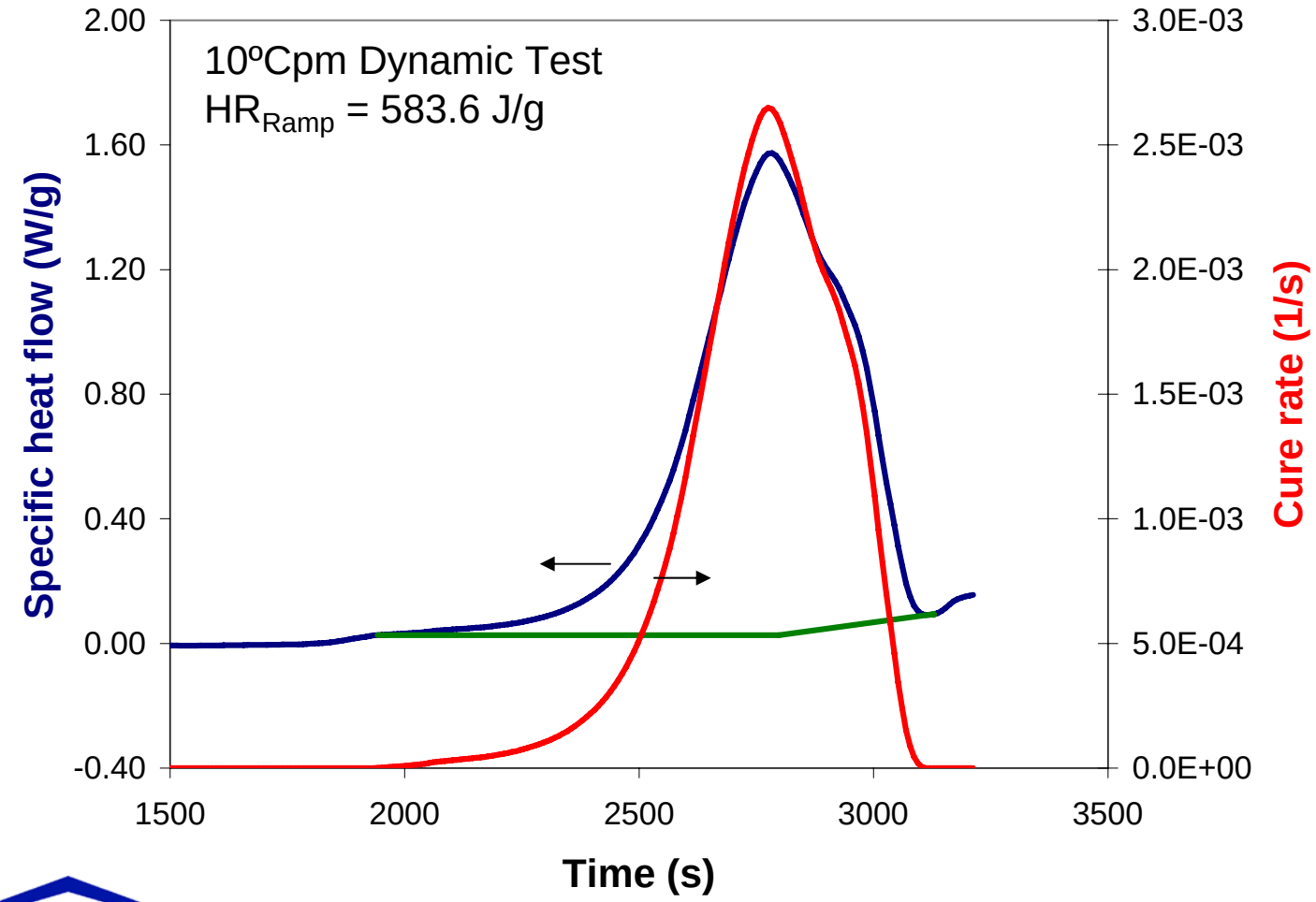
Heat Flow Response – 9cpm



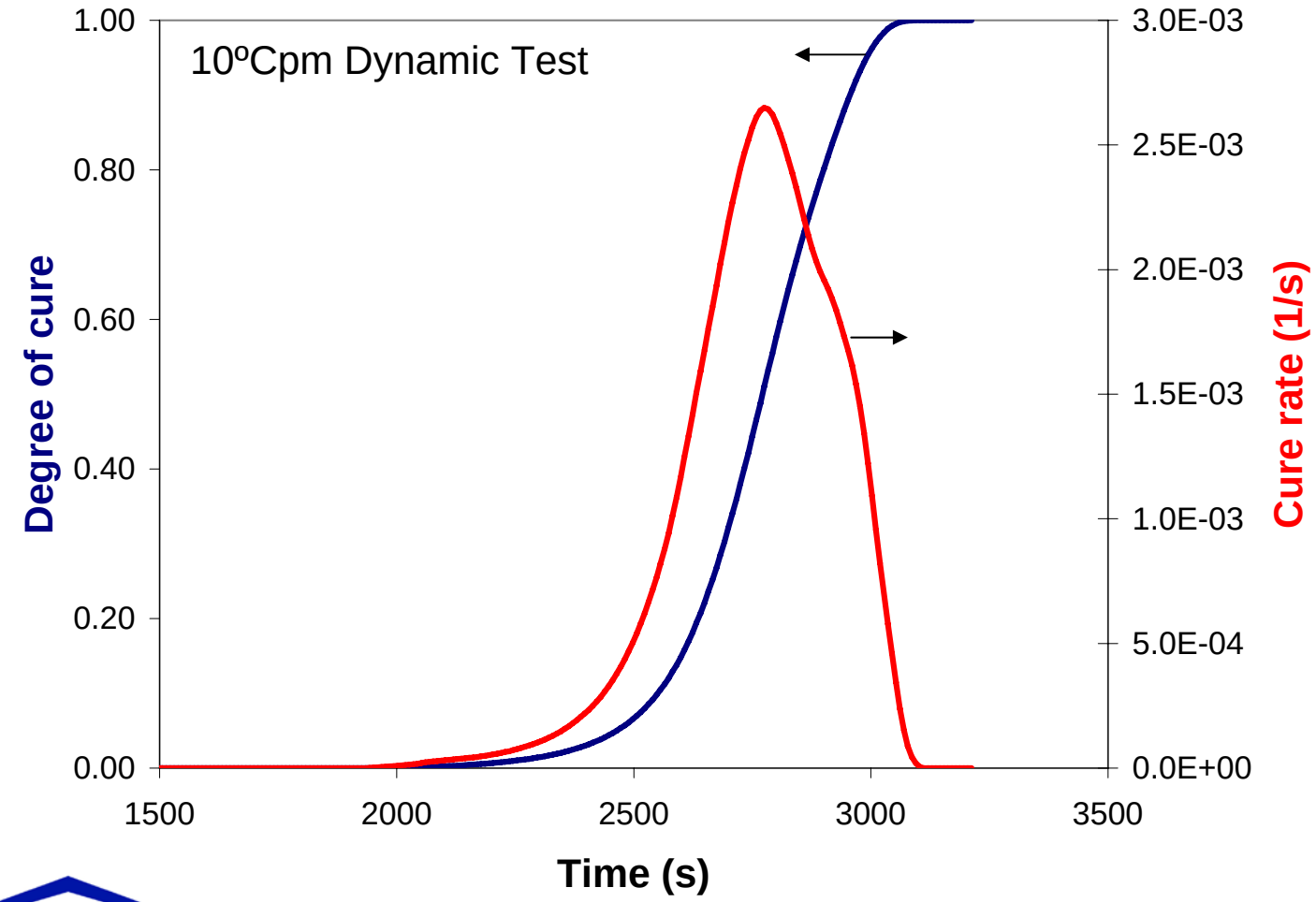
DoC and Cure Rate – 9cpm



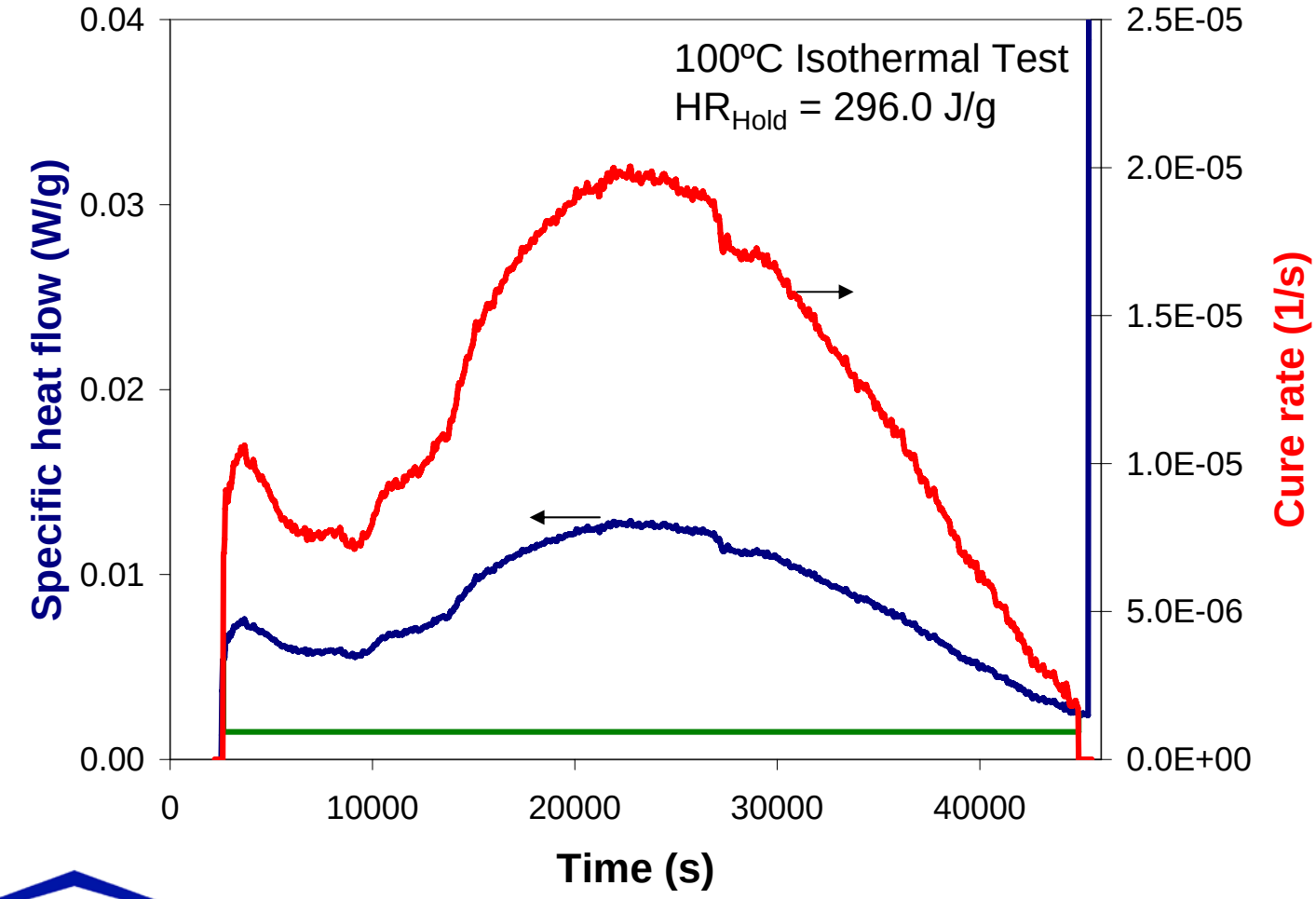
Heat Flow Response – 10cpm



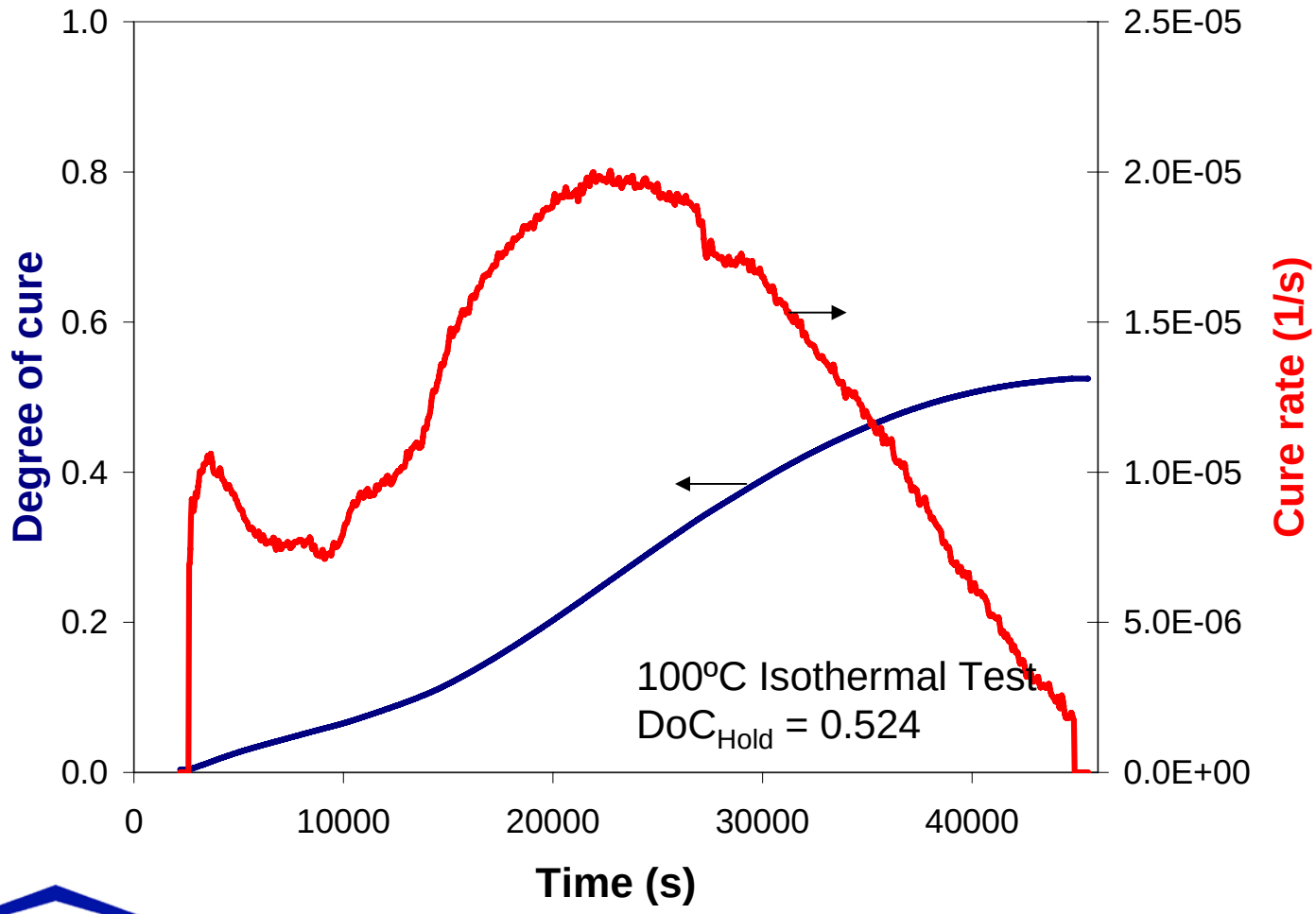
DoC and Cure Rate – 10cpm



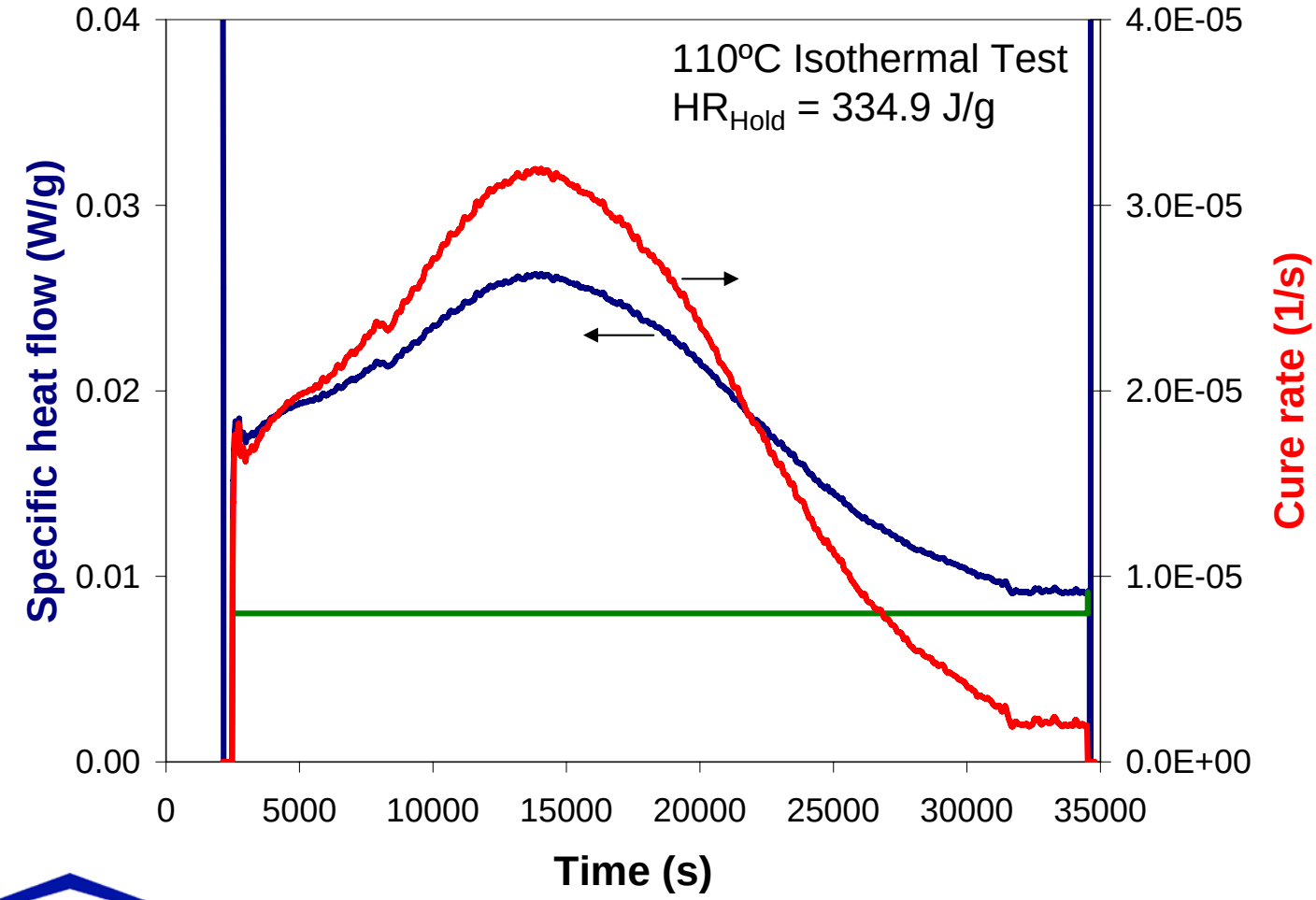
Heat Flow Response – 100ºC



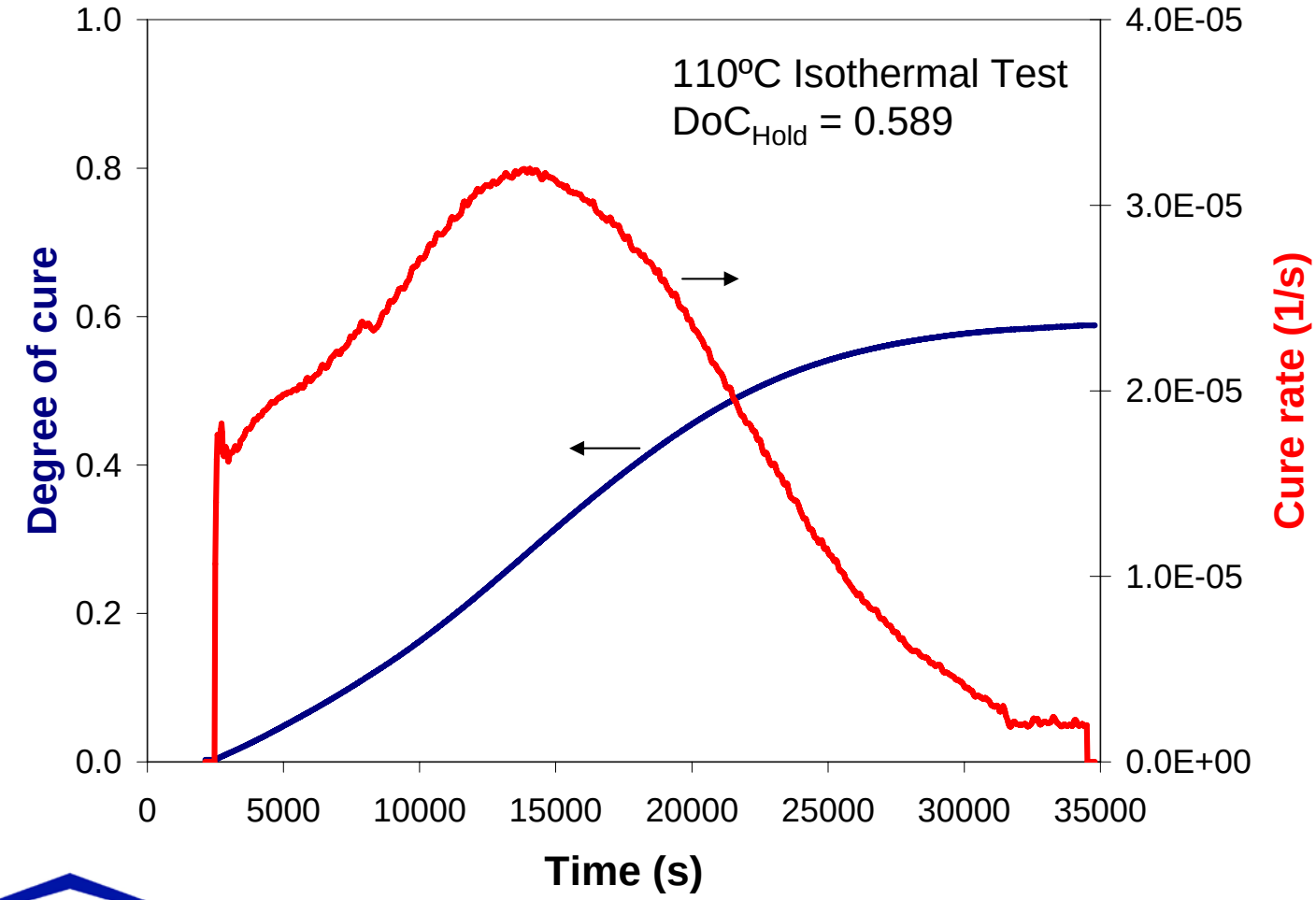
DoC and Cure Rate – 100ºC



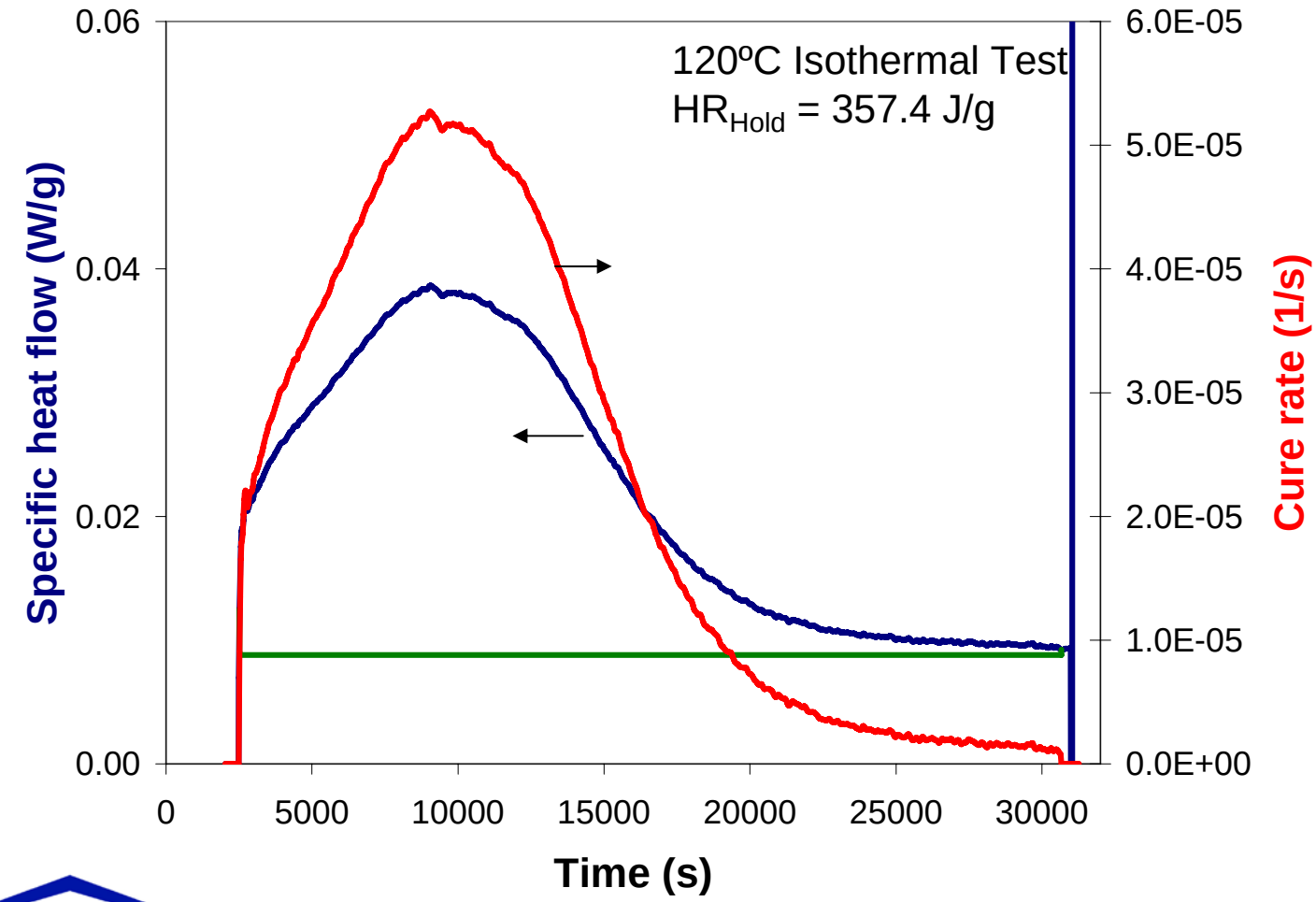
Heat Flow Response – 110ºC



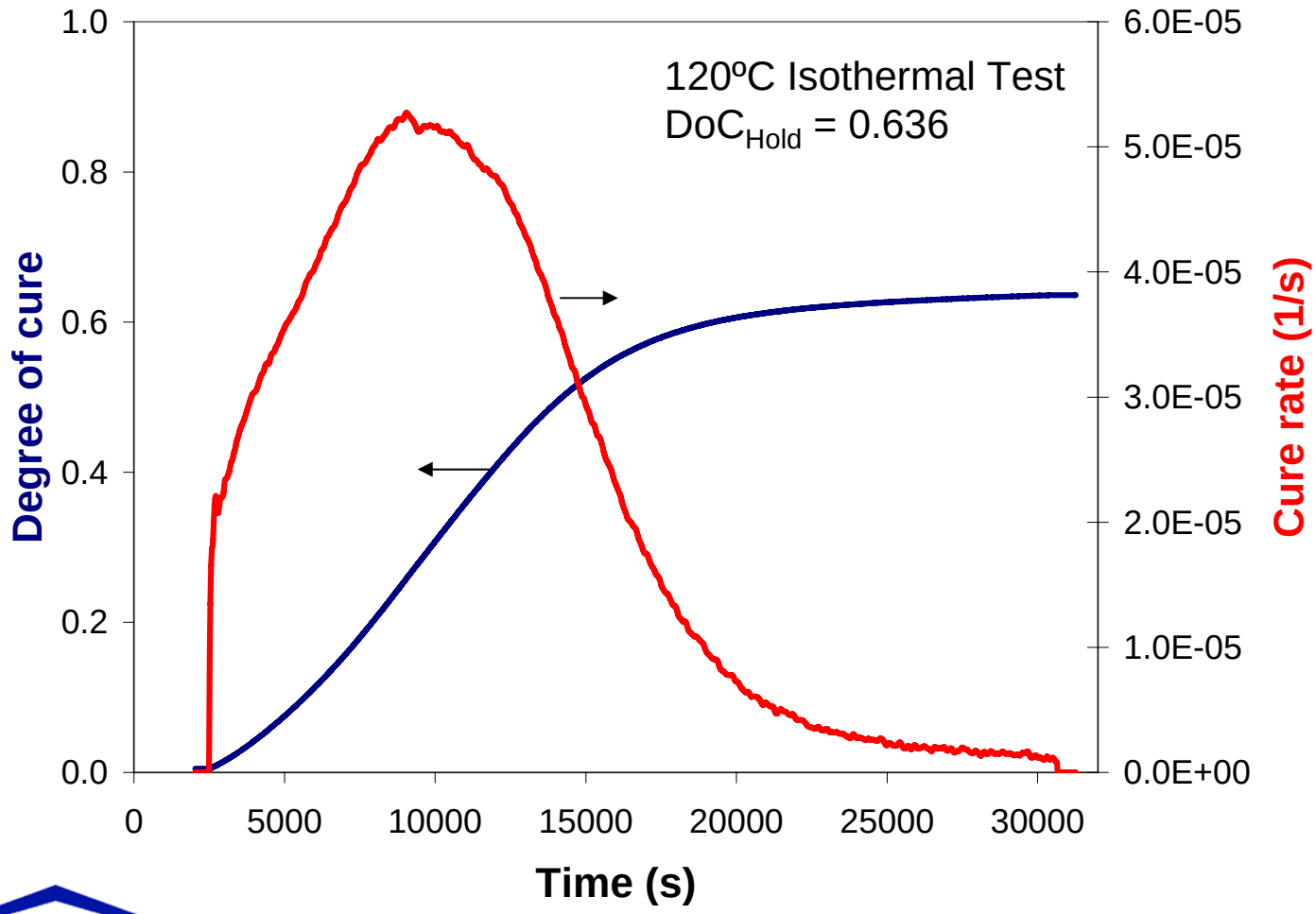
DoC and Cure Rate – 110ºC



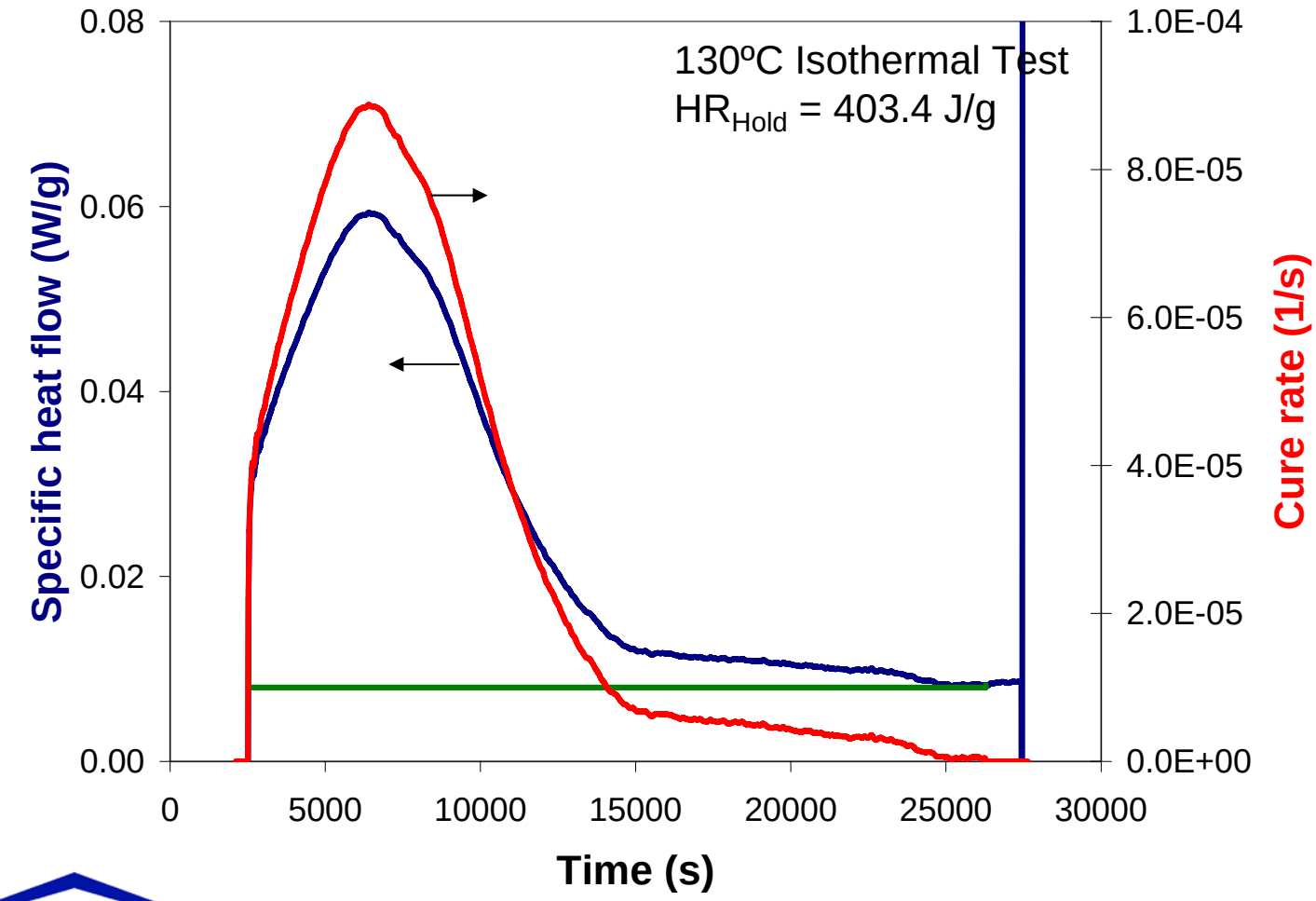
Heat Flow Response – 120ºC



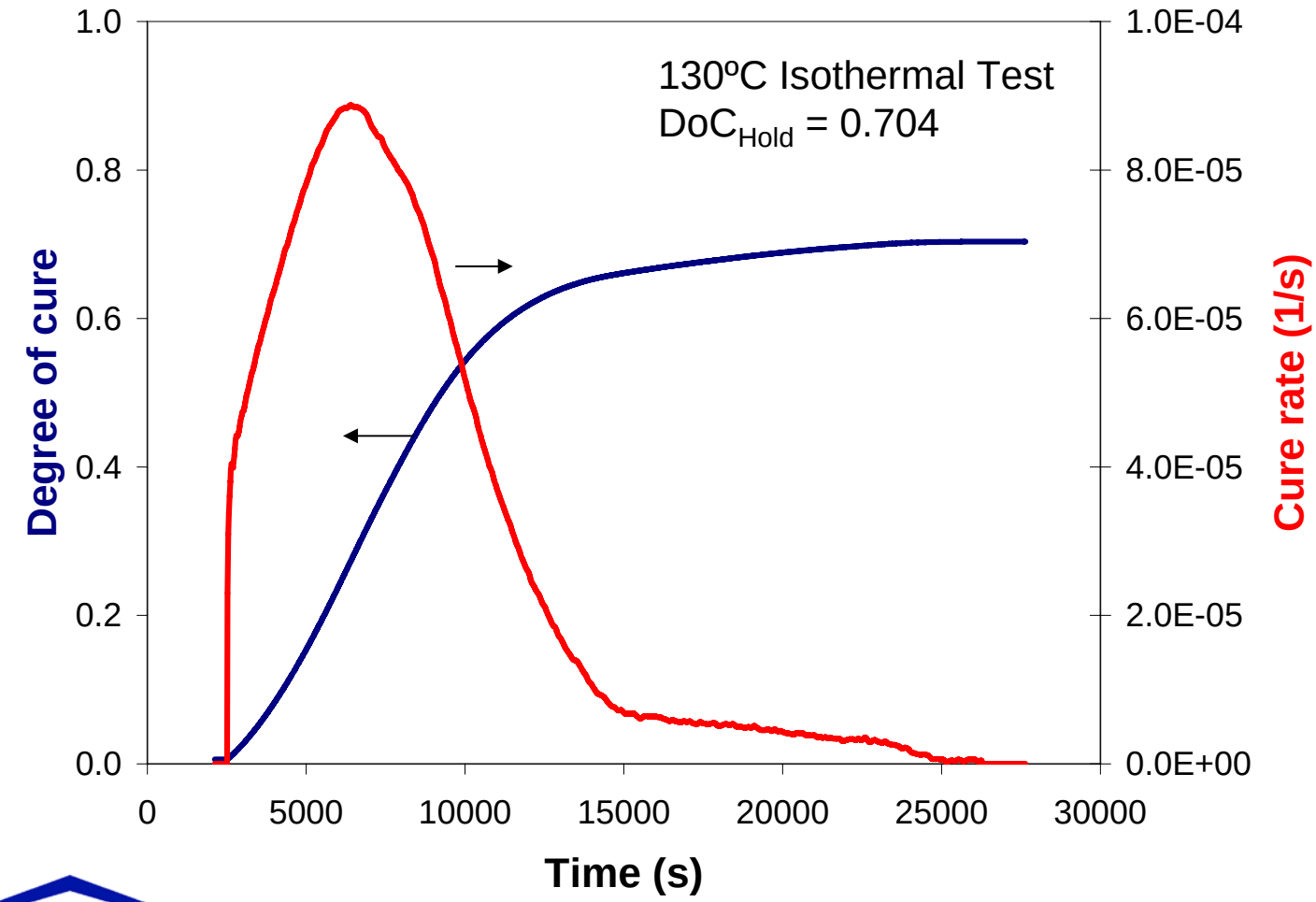
DoC and Cure Rate – 120ºC



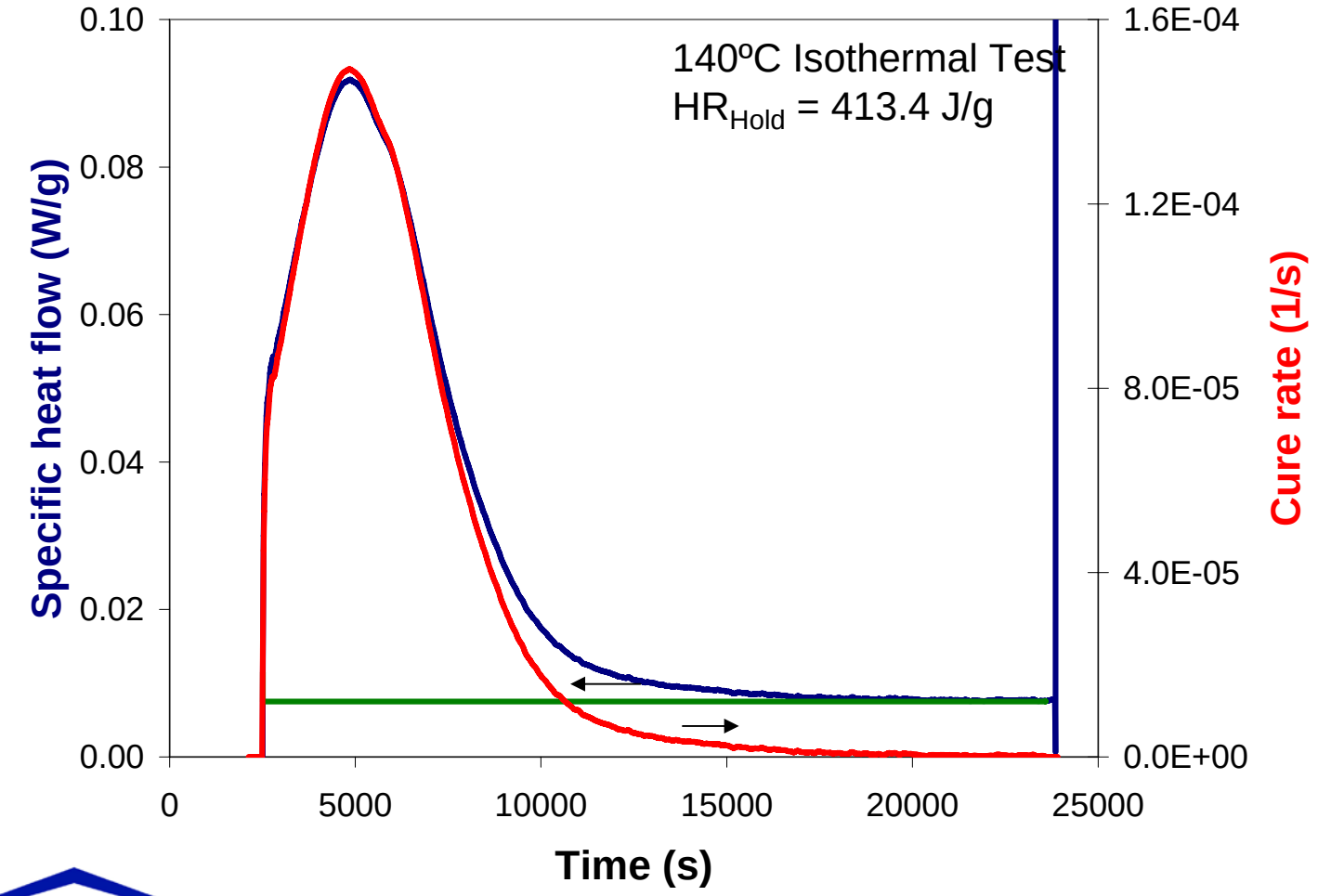
Heat Flow Response – 130ºC



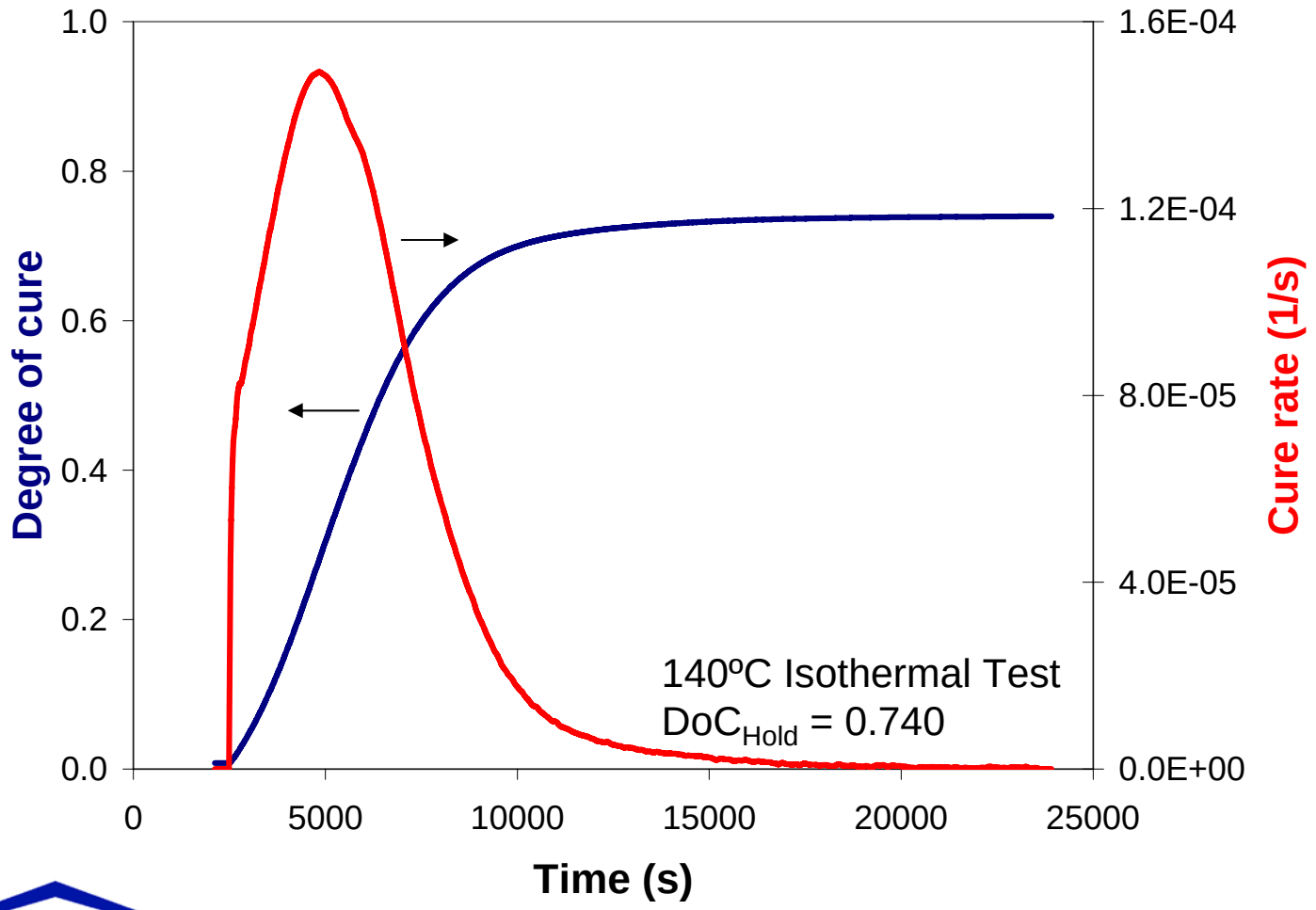
DoC and Cure Rate – 130ºC



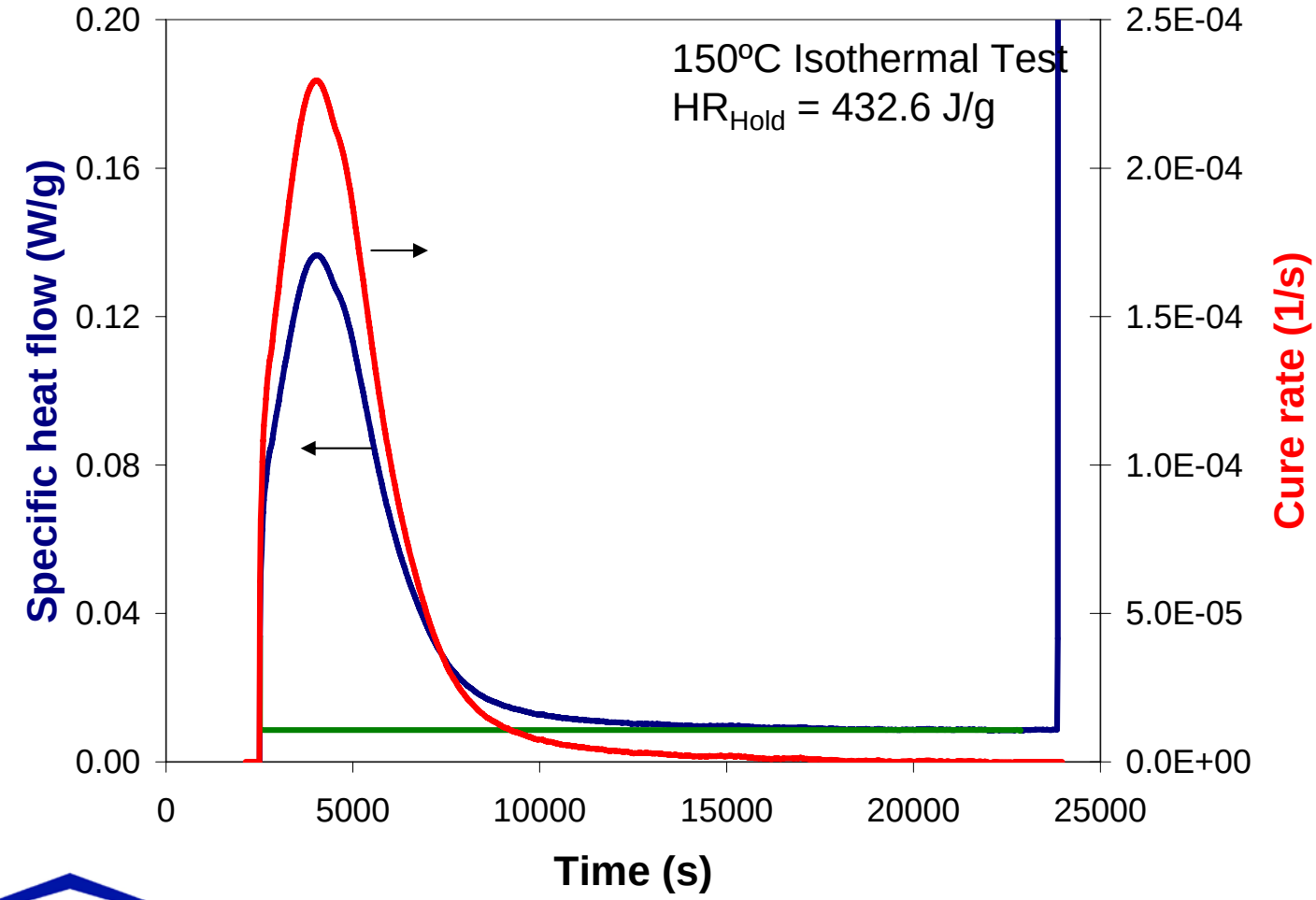
Heat Flow Response – 140ºC



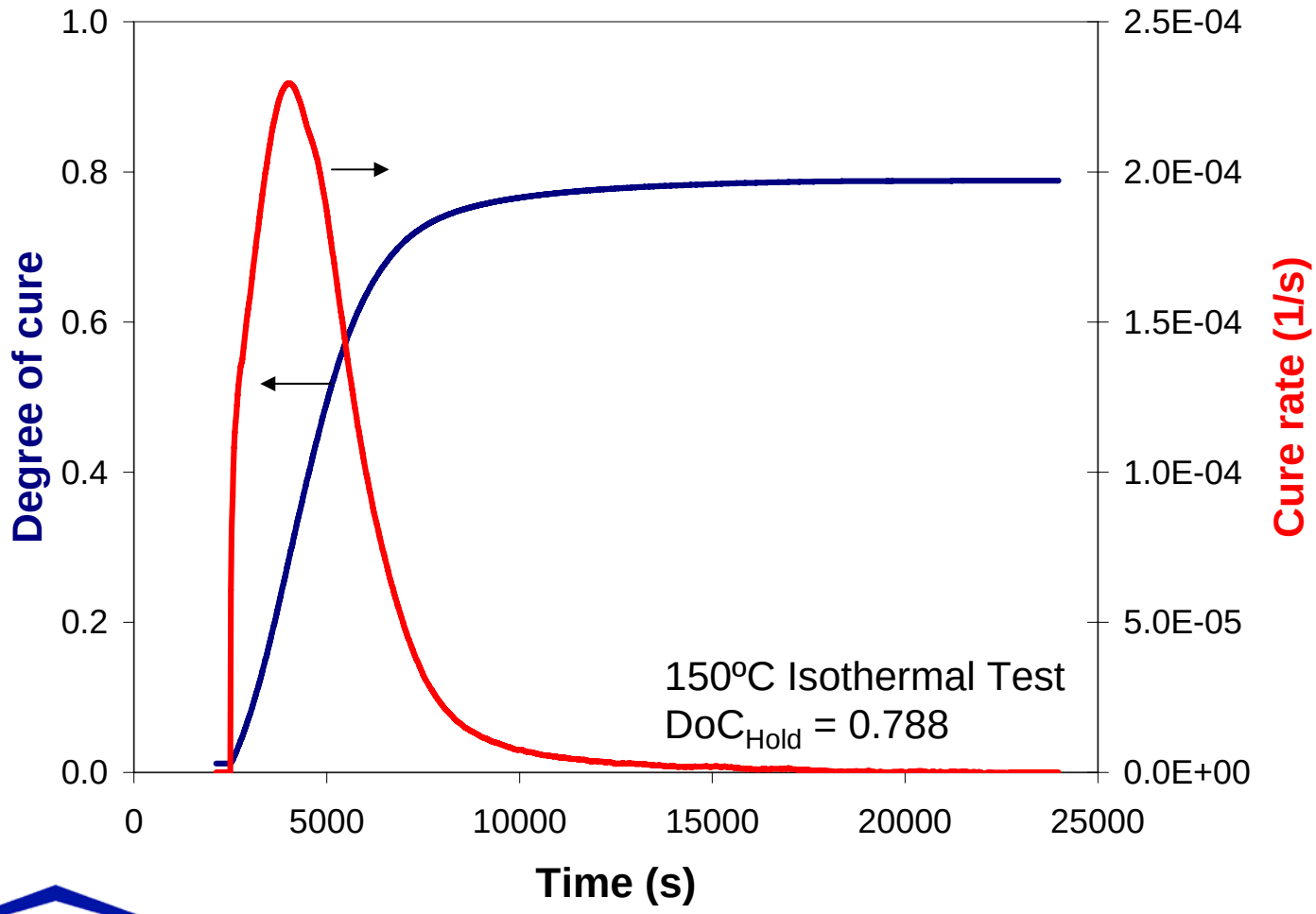
DoC and Cure Rate – 140ºC



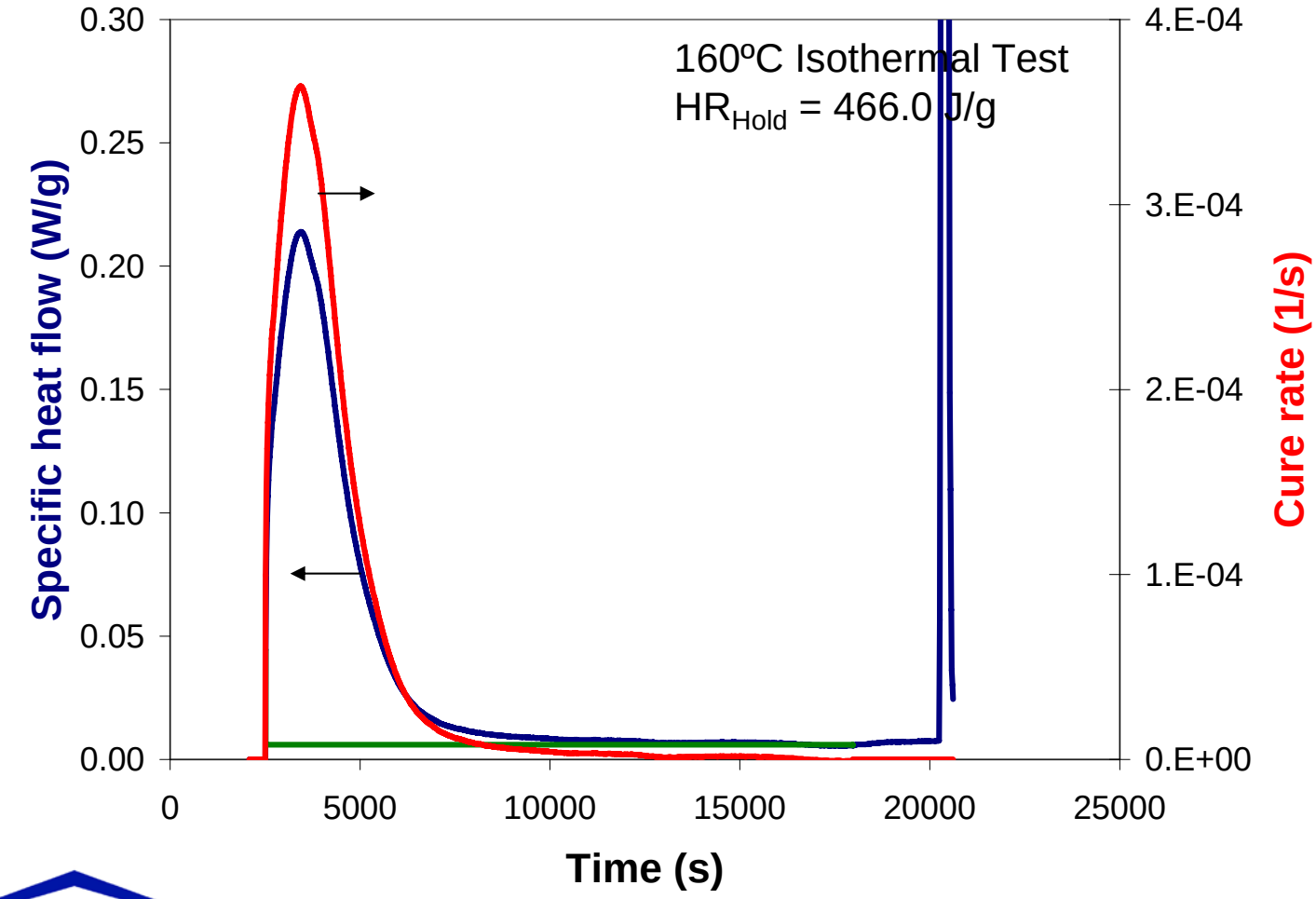
Heat Flow Response – 150ºC



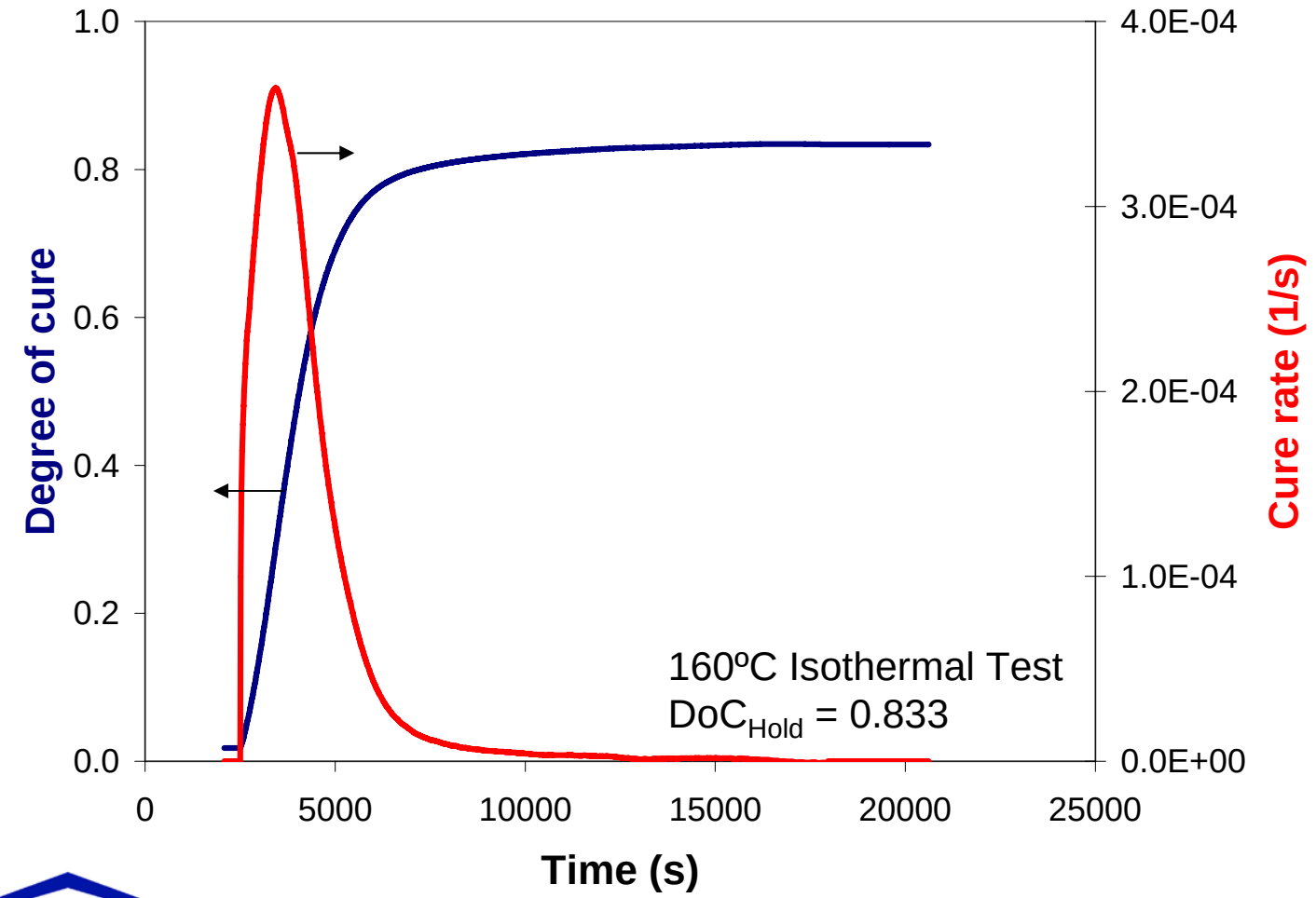
DoC and Cure Rate – 150ºC



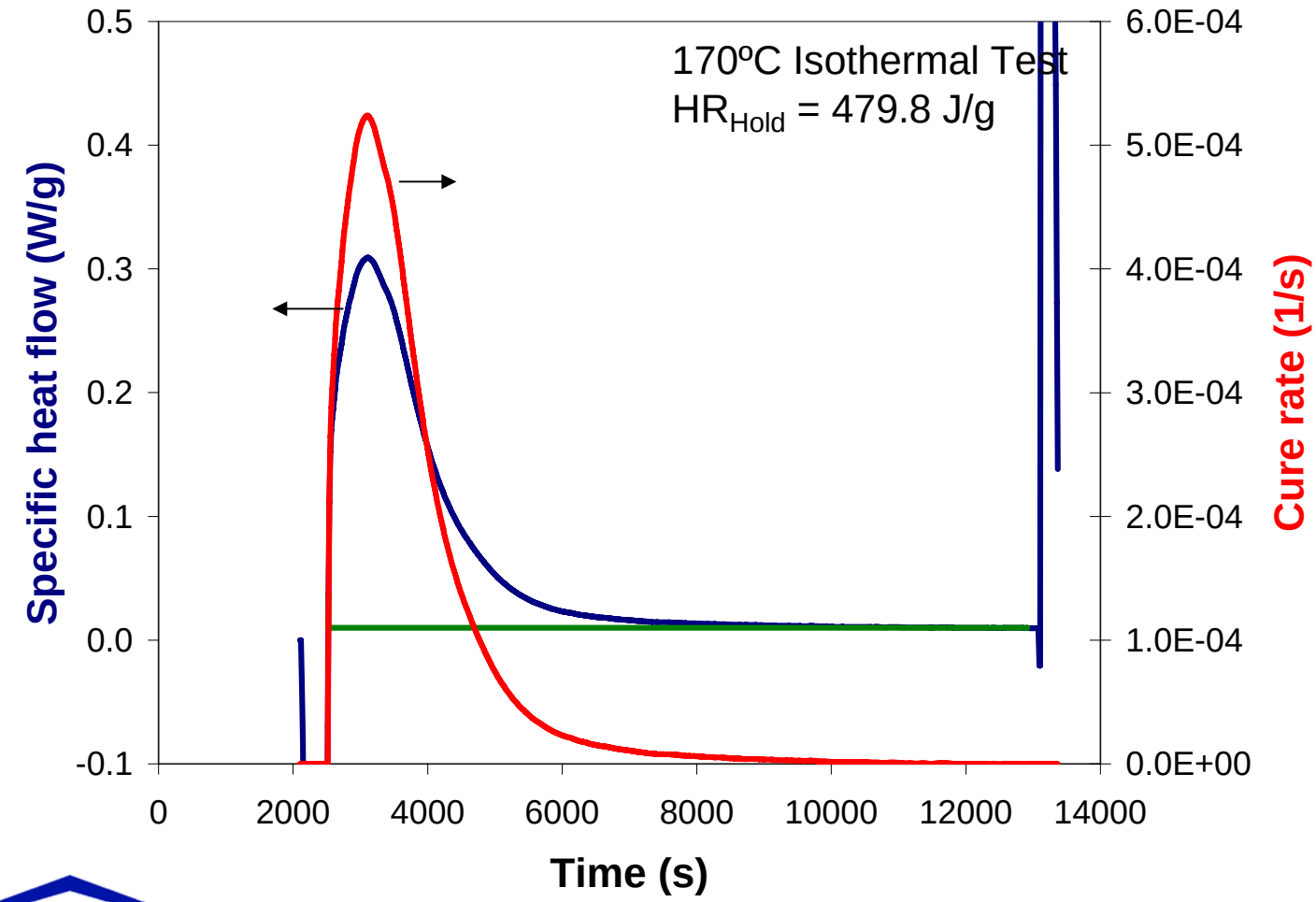
Heat Flow Response – 160ºC



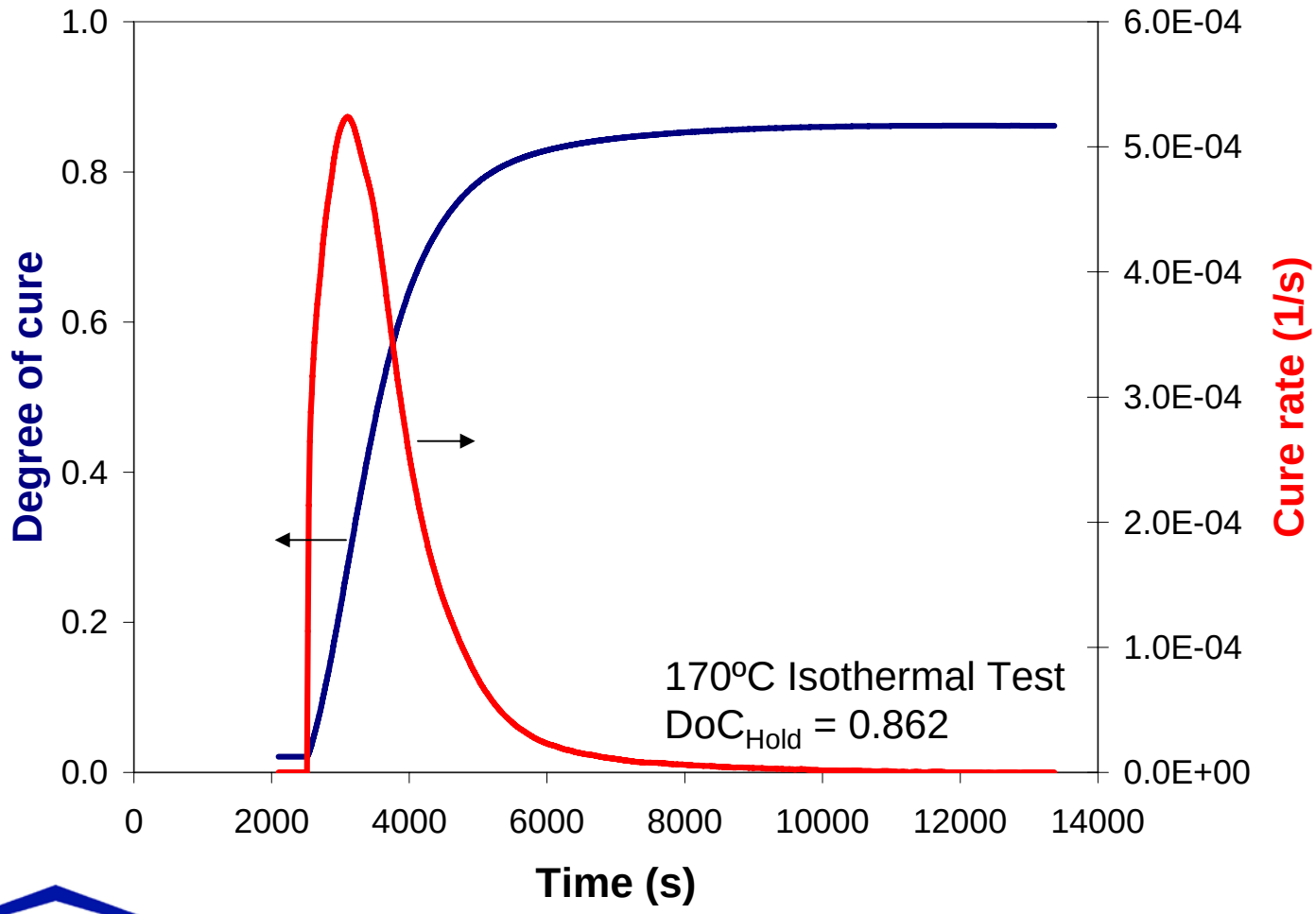
DoC and Cure Rate – 160ºC



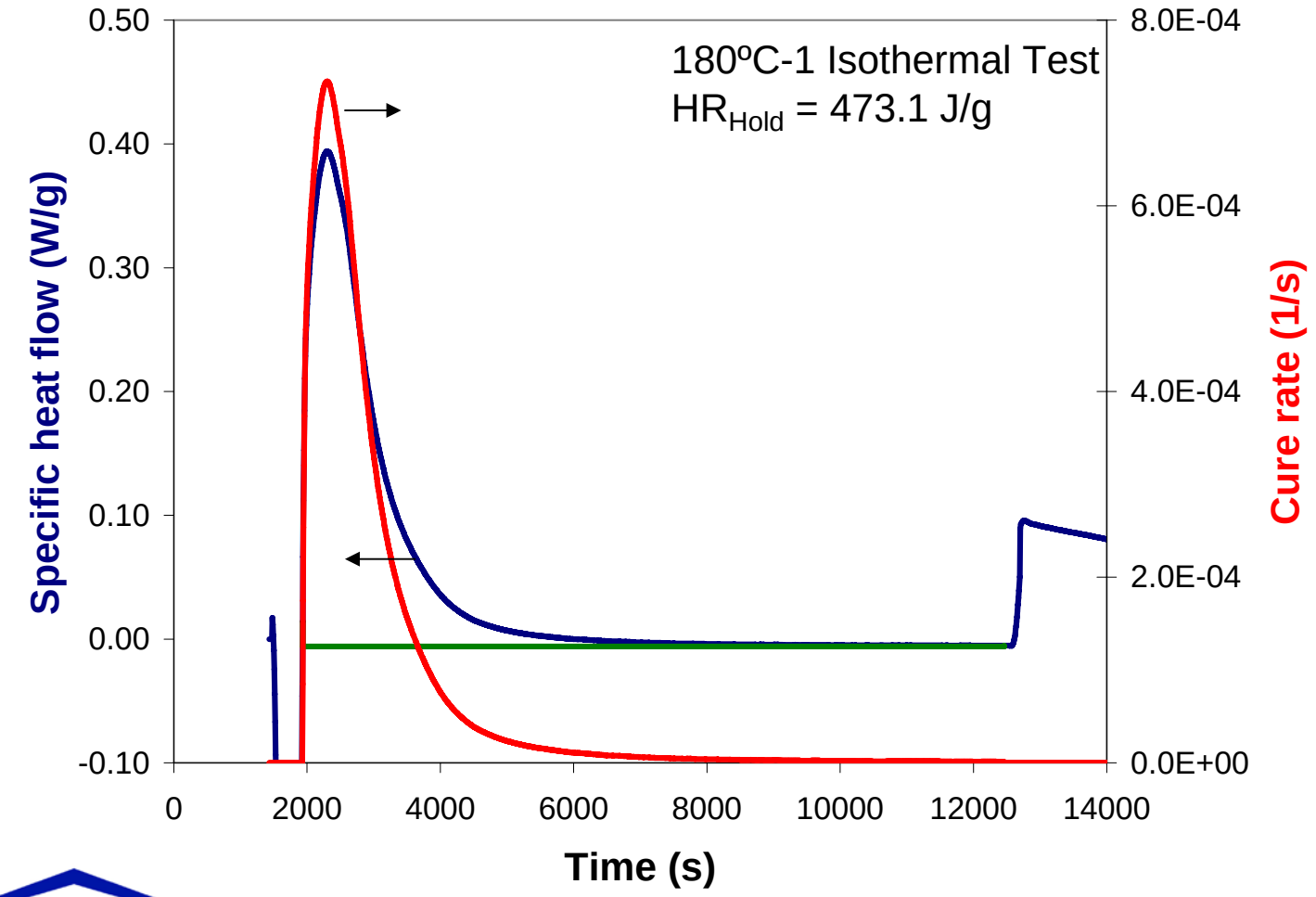
Heat Flow Response – 170ºC



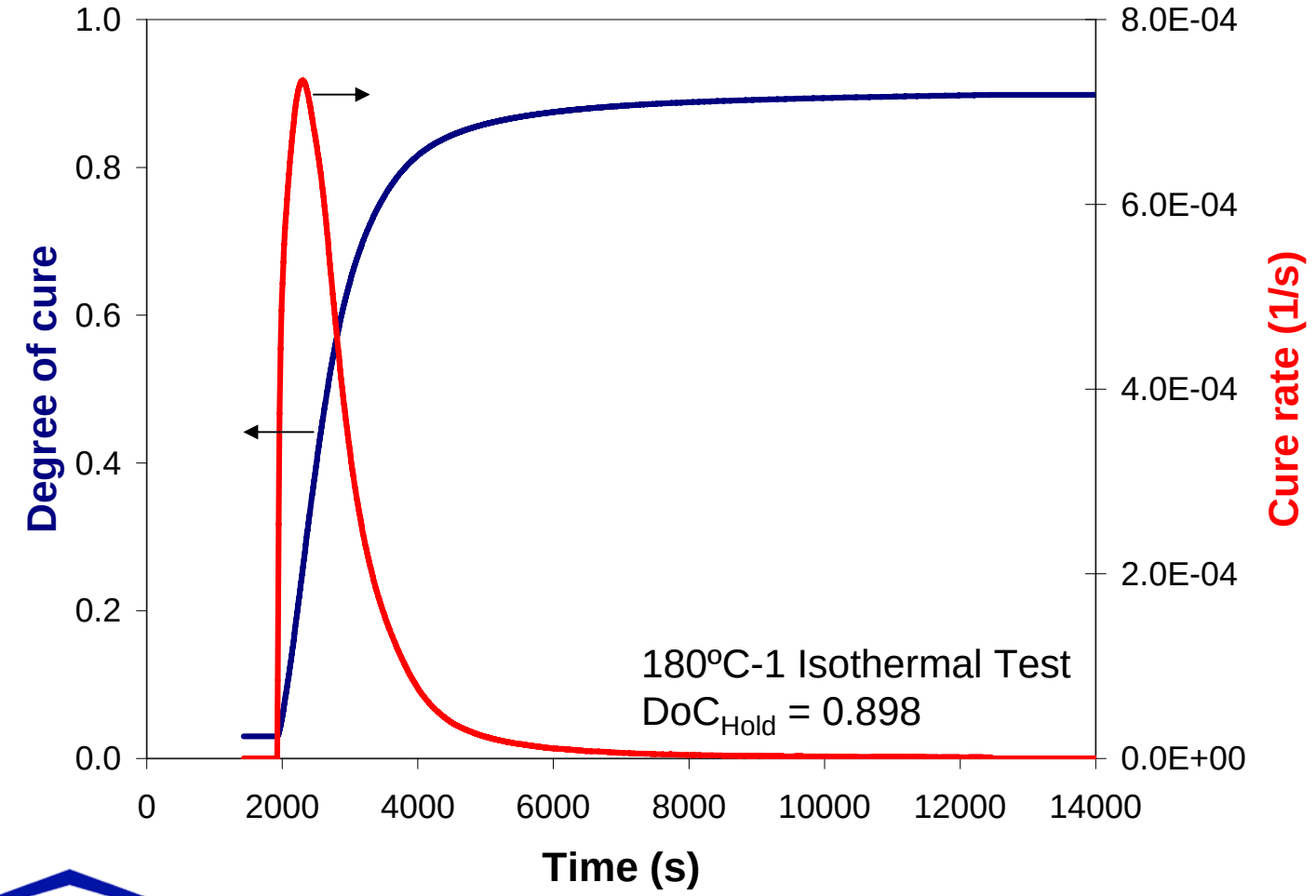
DoC and Cure Rate – 170ºC



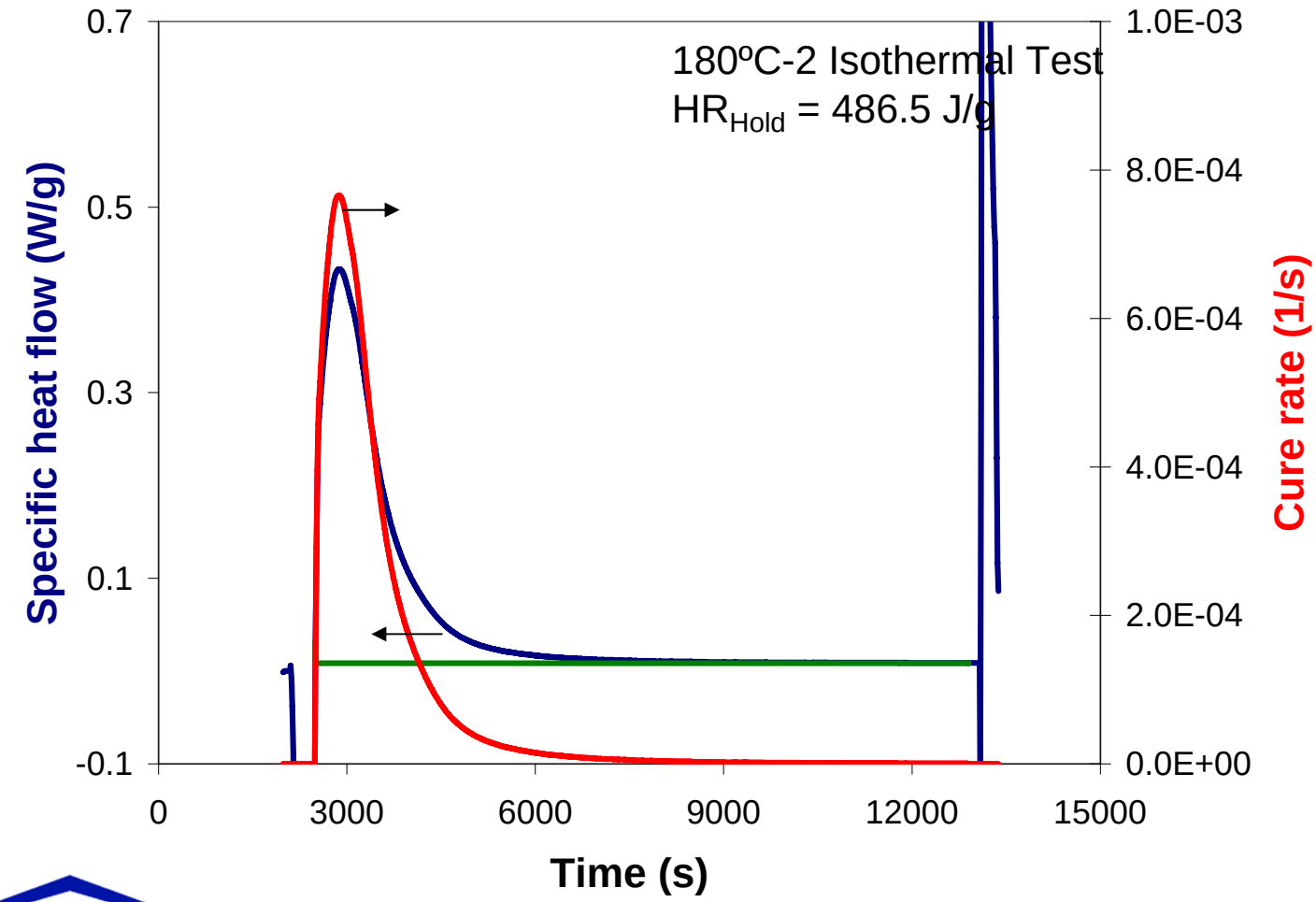
Heat Flow Response – 180ºC-1



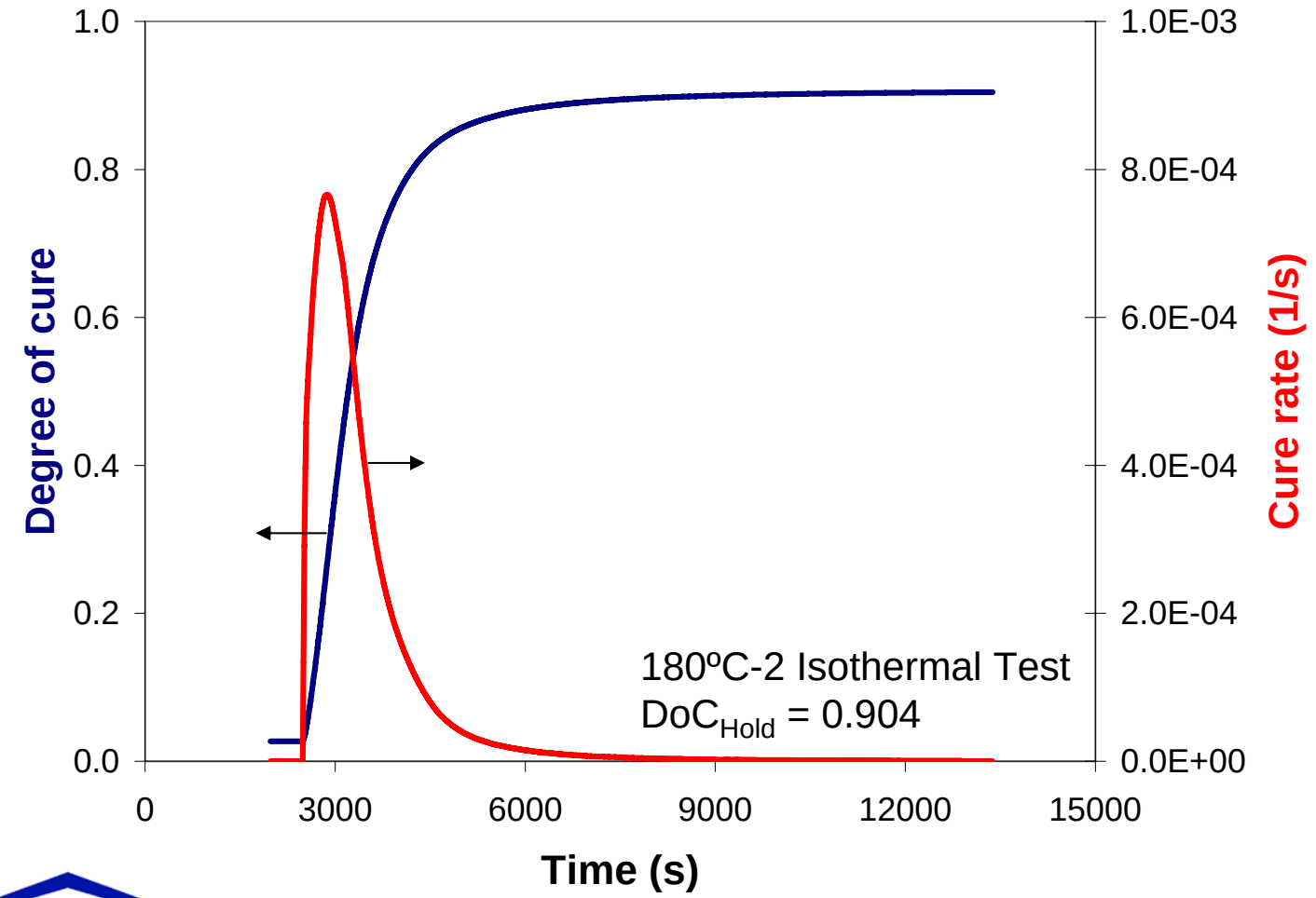
DoC and Cure Rate – 180ºC-1



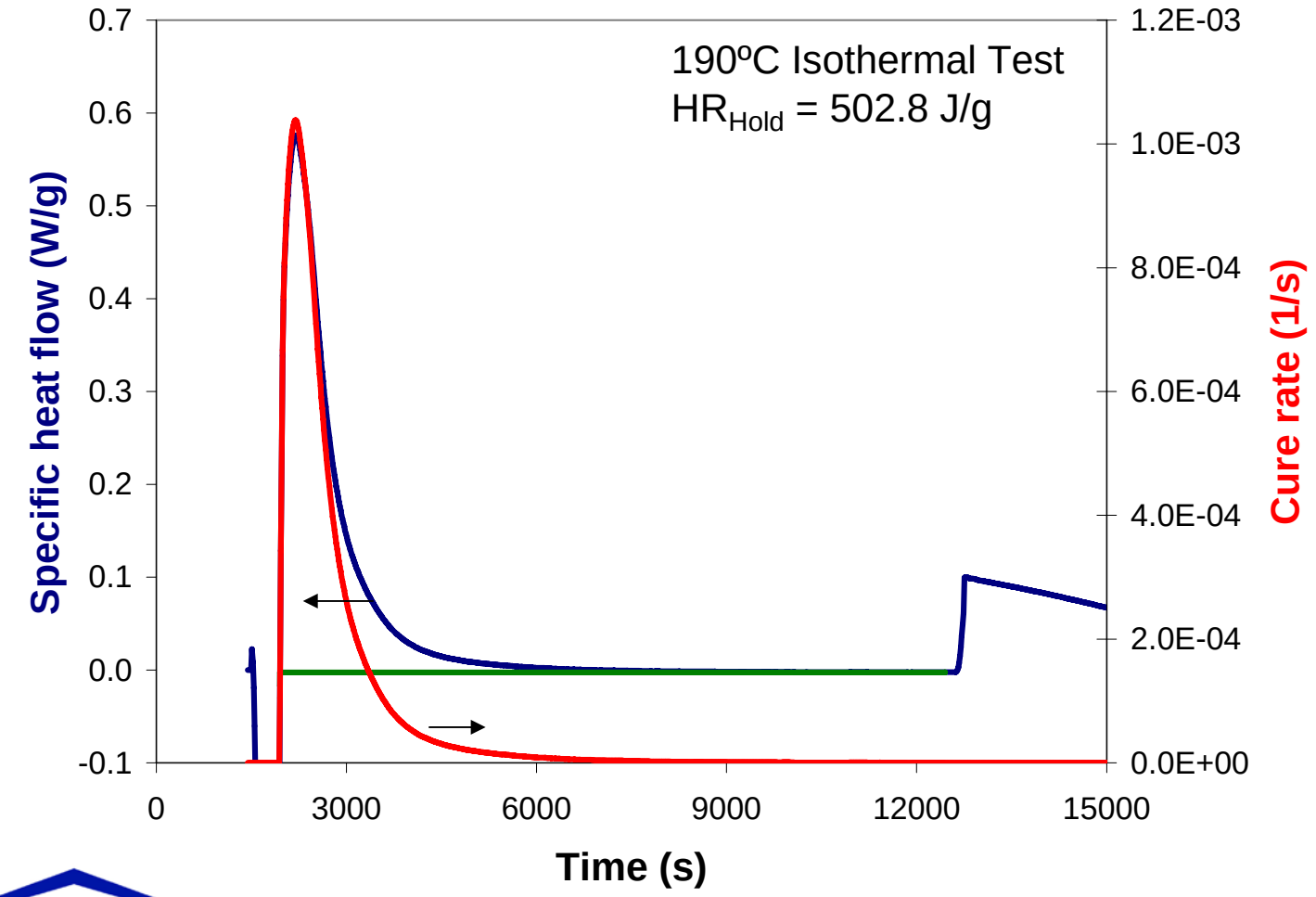
Heat Flow Response – 180ºC-2



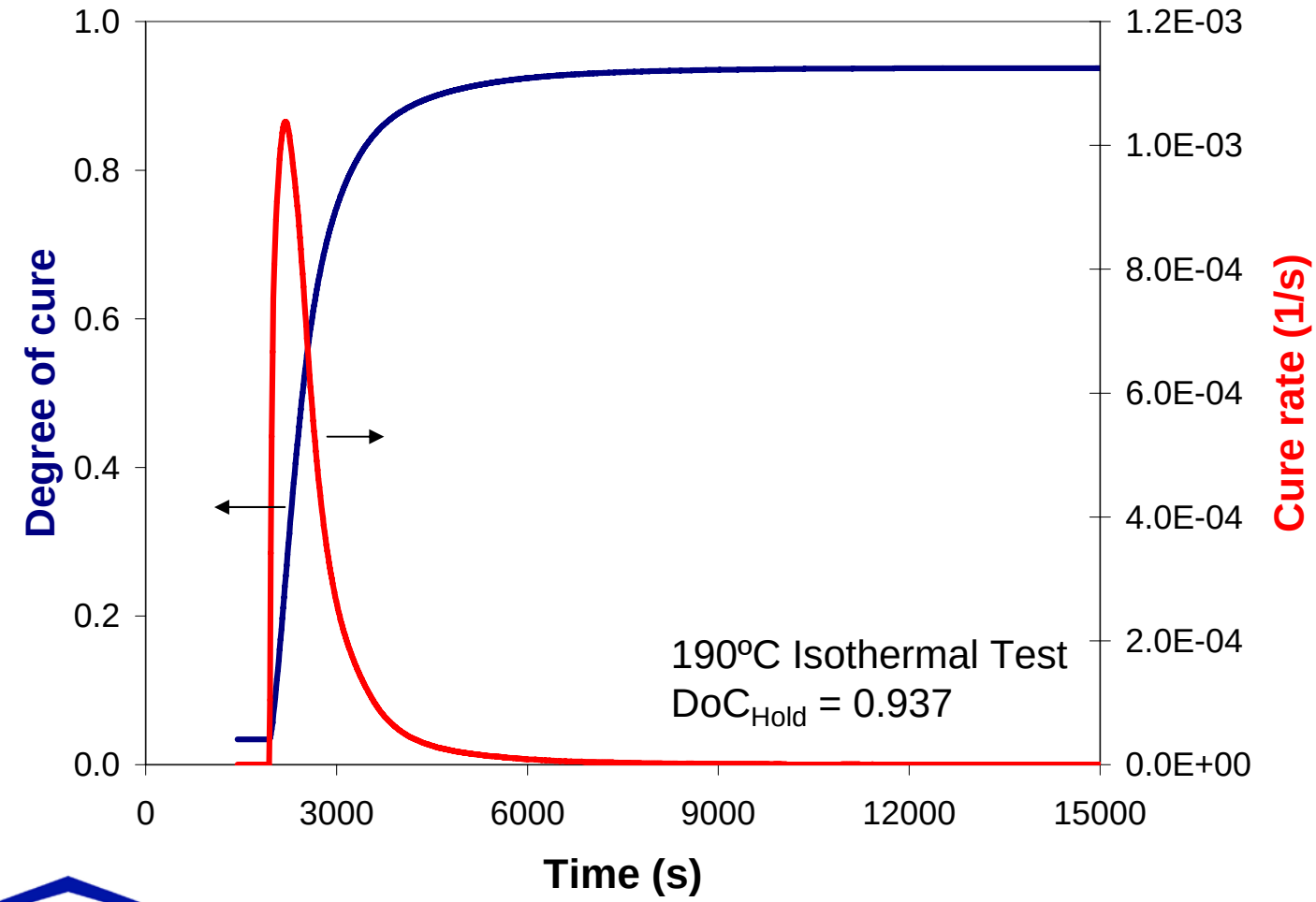
DoC and Cure Rate – 180ºC-2



Heat Flow Response – 190ºC



DoC and Cure Rate – 190ºC

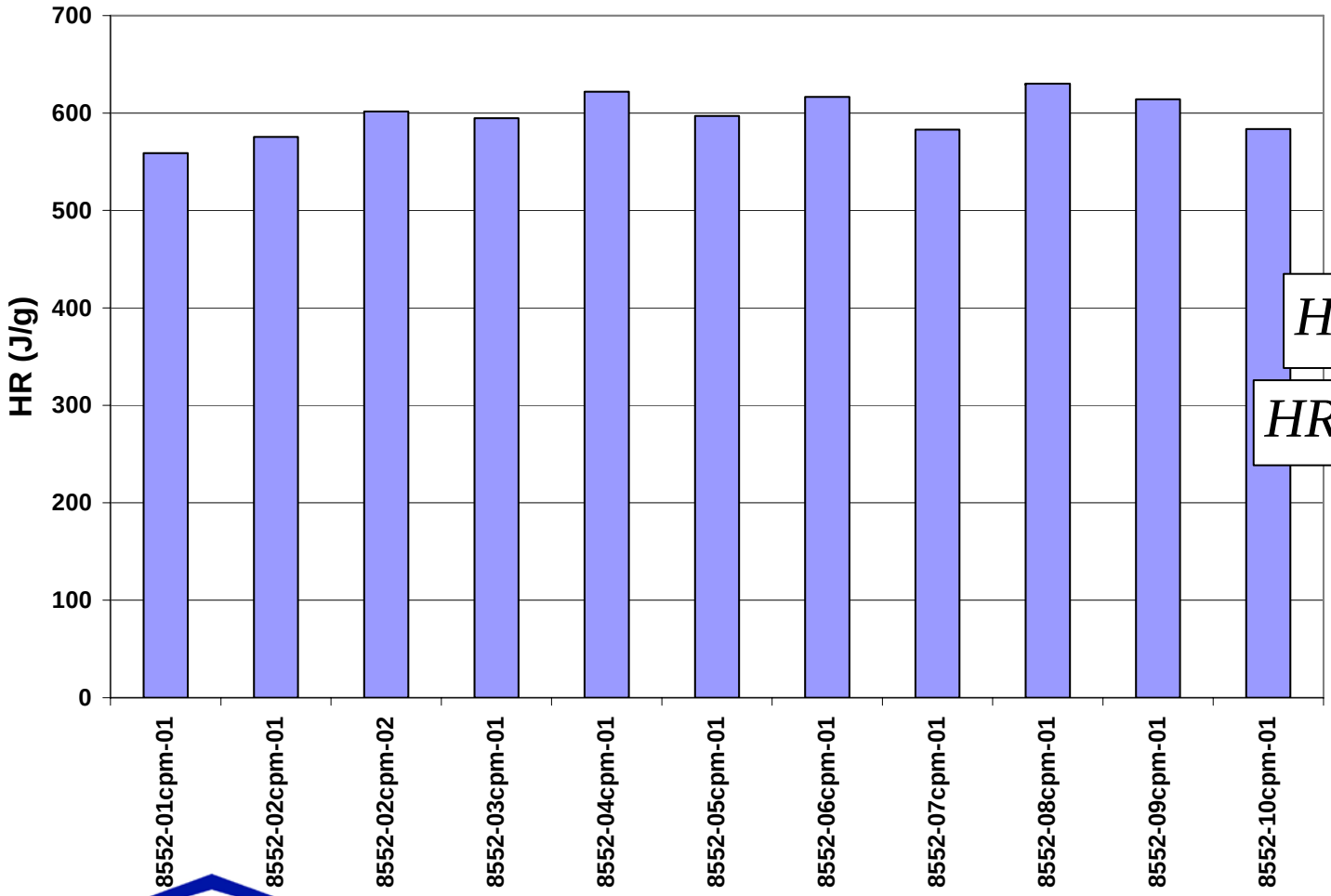


Heat of Reaction Tables

Test	Heat of Reaction (J/g)		
	Pre-Test	Ramp	Total
8552-MDYN-01cpm-01	0.00	558.91	558.91
8552-MDYN-02cpm-01	0.00	575.43	575.43
8552-MDYN-02cpm-02	0.00	601.65	601.65
8552-MDYN-03cpm-01	0.00	594.88	594.88
8552-MDYN-04cpm-01	0.00	621.82	621.82
8552-MDYN-05cpm-01	0.00	596.98	596.98
8552-MDYN-06cpm-01	0.00	616.41	616.41
8552-MDYN-07cpm-01	0.00	582.98	582.98
8552-MDYN-08cpm-01	0.00	630.07	630.07
8552-MDYN-09cpm-01	0.00	614.07	614.07
8552-MDYN-10cpm-01	0.00	583.62	583.62

Test	Heat of Reaction (J/g)			
	Iso-Ramp	Hold	Ramp	Total
8552-MISO-100C-01	2.00	295.95	270.47	568.42
8552-MISO-110C-01	2.00	334.88	235.30	572.18
8552-MISO-120C-01	3.00	357.40	206.01	566.41
8552-MISO-130C-01	3.50	403.40	171.32	578.21
8552-MISO-140C-01	4.50	413.43	147.17	565.10
8552-MISO-150C-01	6.50	432.63	118.29	557.42
8552-MISO-160C-01	10.00	466.01	95.26	571.27
8552-MISO-170C-01	12.00	479.84	79.00	570.84
8552-MISO-180C-01	16.50	473.10	55.43	545.03
8552-MISO-180C-02	15.00	486.49	53.05	554.54
8552-MISO-190C-01	19.00	502.82	34.88	556.70
8552-160C-INT15	8.50	99.40	445.77	553.66
8552-160C-INT30	8.50	274.09	275.24	557.83

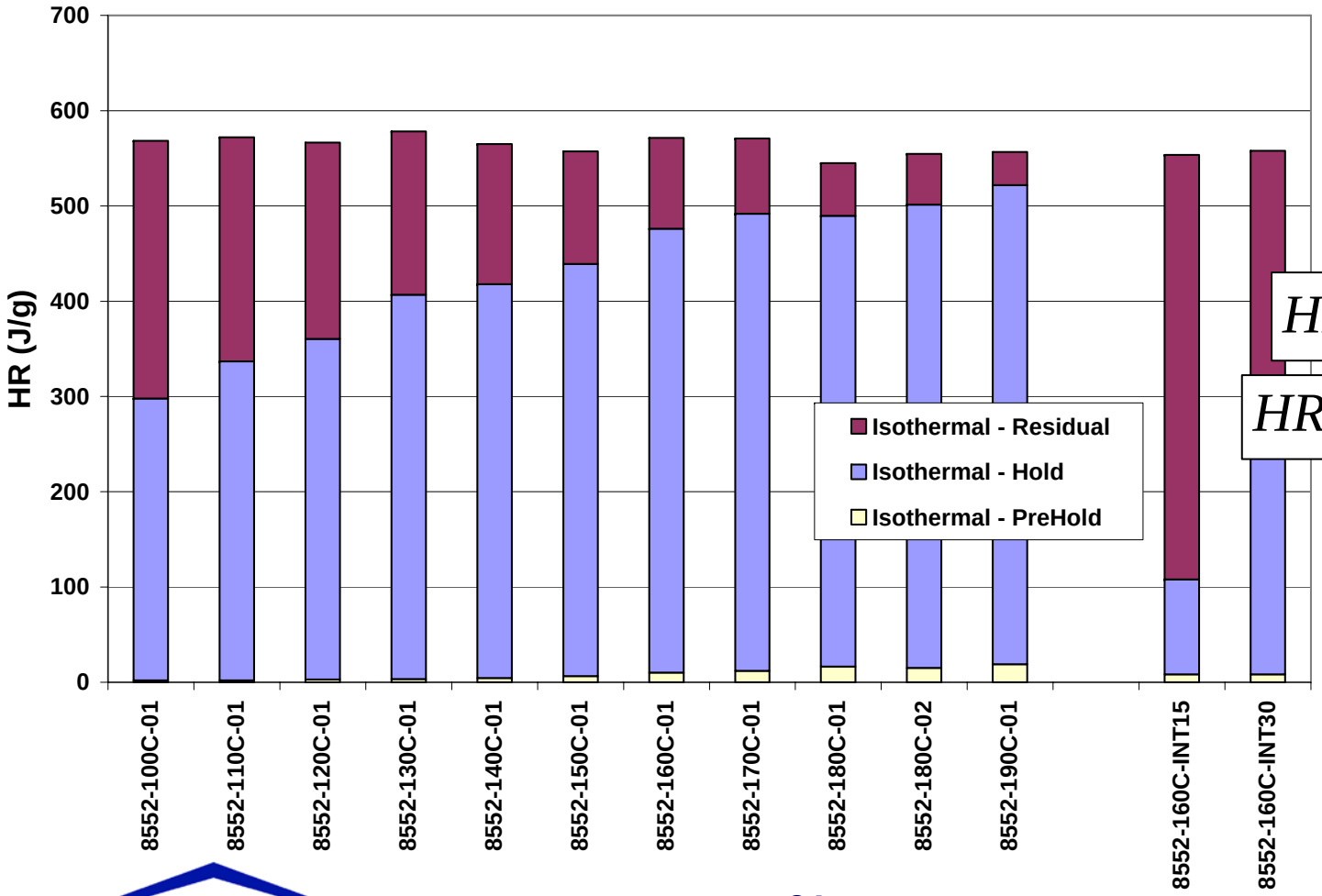
Total Heat of Reaction - Dyns



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

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

Total Heat of Reaction - Isos



$$HR_{Iso}^{avg} = 564 \quad \text{J/g}$$

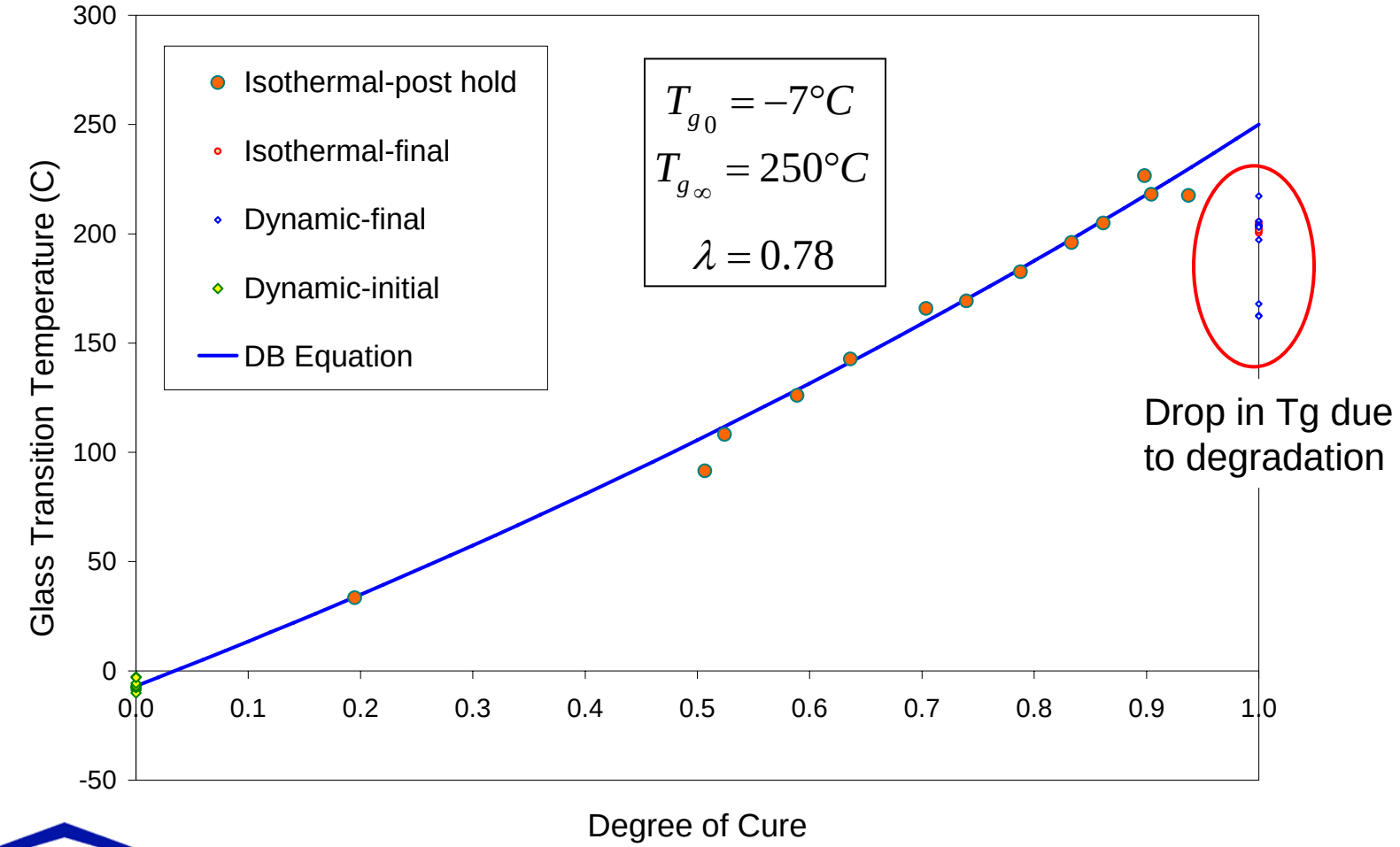
$$HR_{Model} = 600 \quad \text{J/g}$$

Tg Tables

Test	Initial		Final	
	DoC	Tg (ºC)	DoC	Tg (ºC)
8552-MDYN-01cpm-01	0.00	-8.84	1.00	197.26
8552-MDYN-02cpm-01	0.00	-8.15	1.00	205.79
8552-MDYN-02cpm-02	0.00	-10.09	1.00	217.28
8552-MDYN-03cpm-01	0.00	-7.36	1.00	204.15
8552-MDYN-04cpm-01	0.00	-6.66	1.00	203.67
8552-MDYN-05cpm-01	0.00	-7.68	1.00	203.58
8552-MDYN-06cpm-01	0.00	-7.28	1.00	203.48
8552-MDYN-07cpm-01	0.00	-2.70	1.00	203.05
8552-MDYN-08cpm-01	0.00	-7.08	1.00	162.63
8552-MDYN-09cpm-01	0.00	-5.78	1.00	162.28
8552-MDYN-10cpm-01	0.00	-2.99	1.00	167.96

Test	Initial		Post-Hold	
	DoC	Tg (ºC)	DoC	Tg (ºC)
8552-MISO-100C-01	0.00	-12.57	0.52	108.21
8552-MISO-110C-01	0.00	-14.36	0.59	126.05
8552-MISO-120C-01	0.00	-13.94	0.64	142.72
8552-MISO-130C-01	0.00	-13.48	0.70	165.85
8552-MISO-140C-01	0.00	-12.55	0.74	169.27
8552-MISO-150C-01	0.00	-12.71	0.79	182.65
8552-MISO-160C-01	0.00	-14.44	0.83	195.99
8552-MISO-170C-01	0.00	-14.31	0.86	204.91
8552-MISO-180C-01	0.00	-	0.90	226.65
8552-MISO-180C-02	0.00	-13.53	0.90	218.09
8552-MISO-190C-01	0.00	-	0.94	217.58
8552-160C-INT15	0.00	-14.28	0.19	33.47
8552-160C-INT30	0.00	-14.58	0.51	91.53

Glass Transition Temperature



Cure Kinetics Model

Chemical Reaction:

$$\dot{x}_k = \dot{x}_{k1} + \dot{x}_{k2}$$

where

$$\dot{x}_{k1} = 153{,}900.5 \times e^{\frac{-64{,}929.5}{RT}} (1-x)^{2.347} (x + 0.1594)^{1.413}$$

$$\dot{x}_{k2} = 3.963 \times 10^{11} \times e^{\frac{-133{,}168.3}{RT}} (1-x)^{1.029} x^{5.586}$$

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] If f<small then f=small (=1E-30 see CMT_vars2.f90)

Cure Kinetics Model

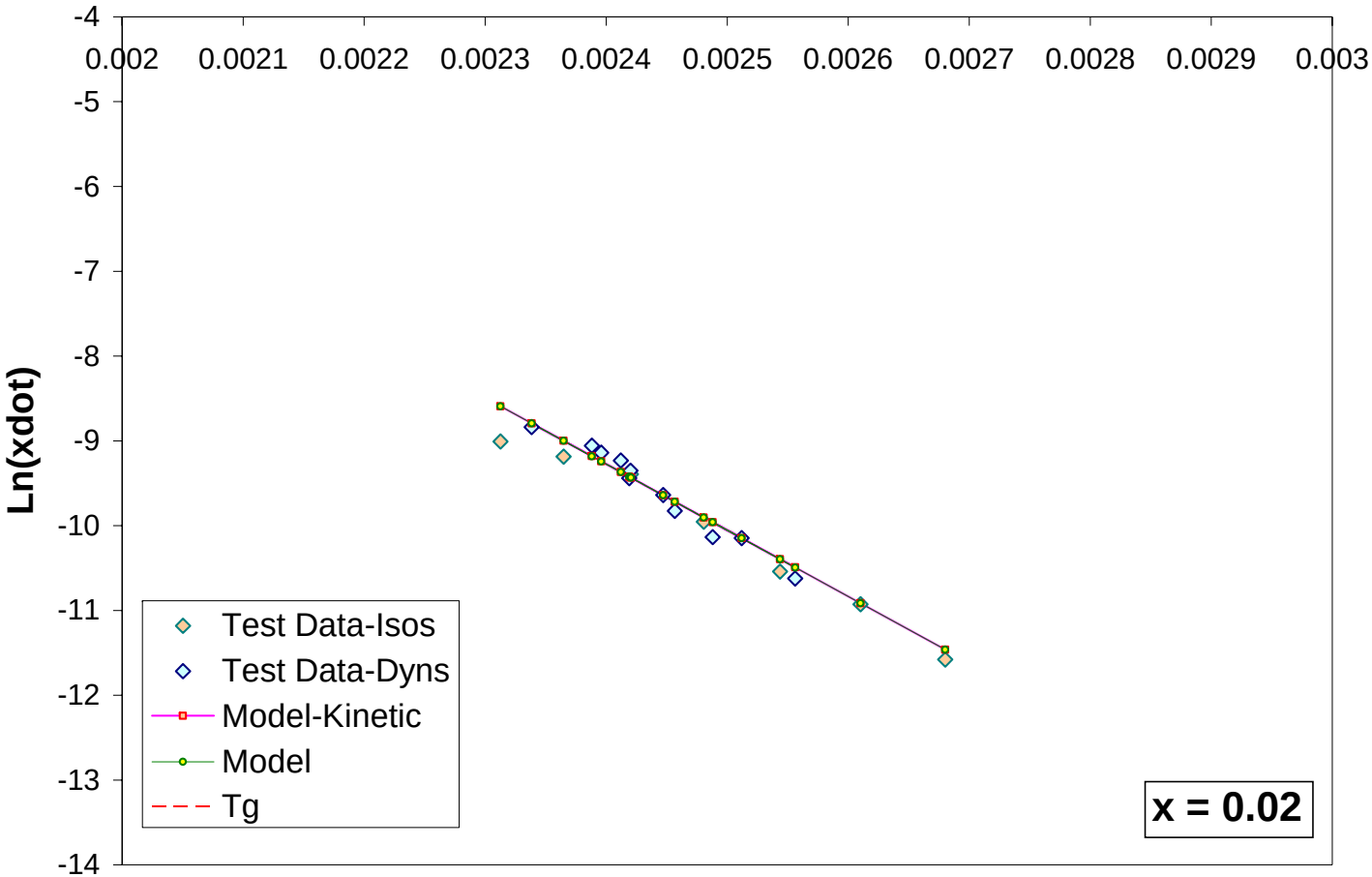
$$\dot{x} = \left(\frac{1}{\dot{x}_k} + \frac{1}{\dot{x}_d} \right)^{-1}$$

Total Heat of Reaction: $HR = 600 \quad \text{J/g}$

Glass Transition (Tg) Model (DeBenedetto):

$T_{g0} = \text{-}7°\text{C}$ $T_{g\infty} = 250°\text{C}$ $\lambda = 0.78$

Ln(xdot)-1/T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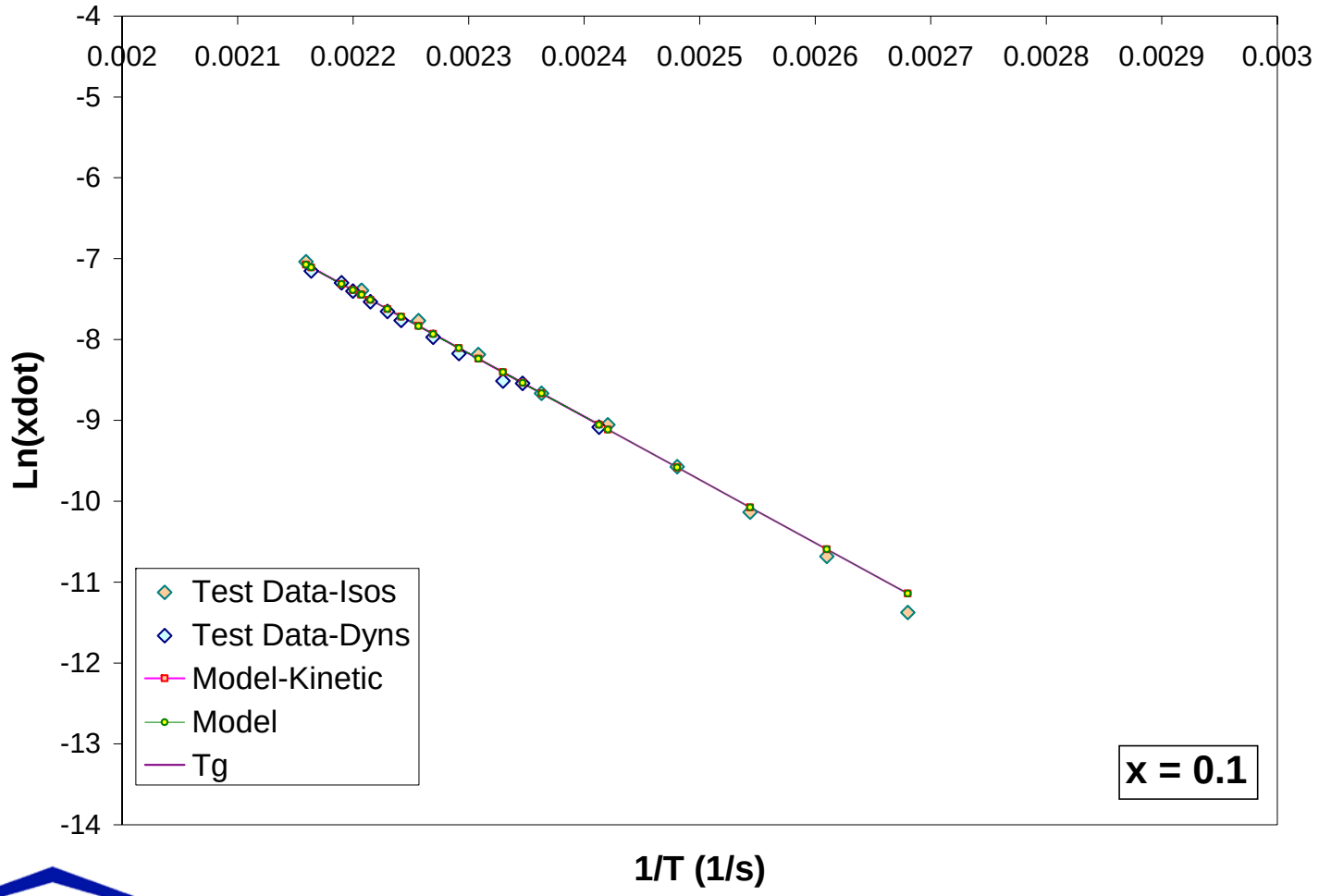
0.003
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.02

Ln(xdot)-1/T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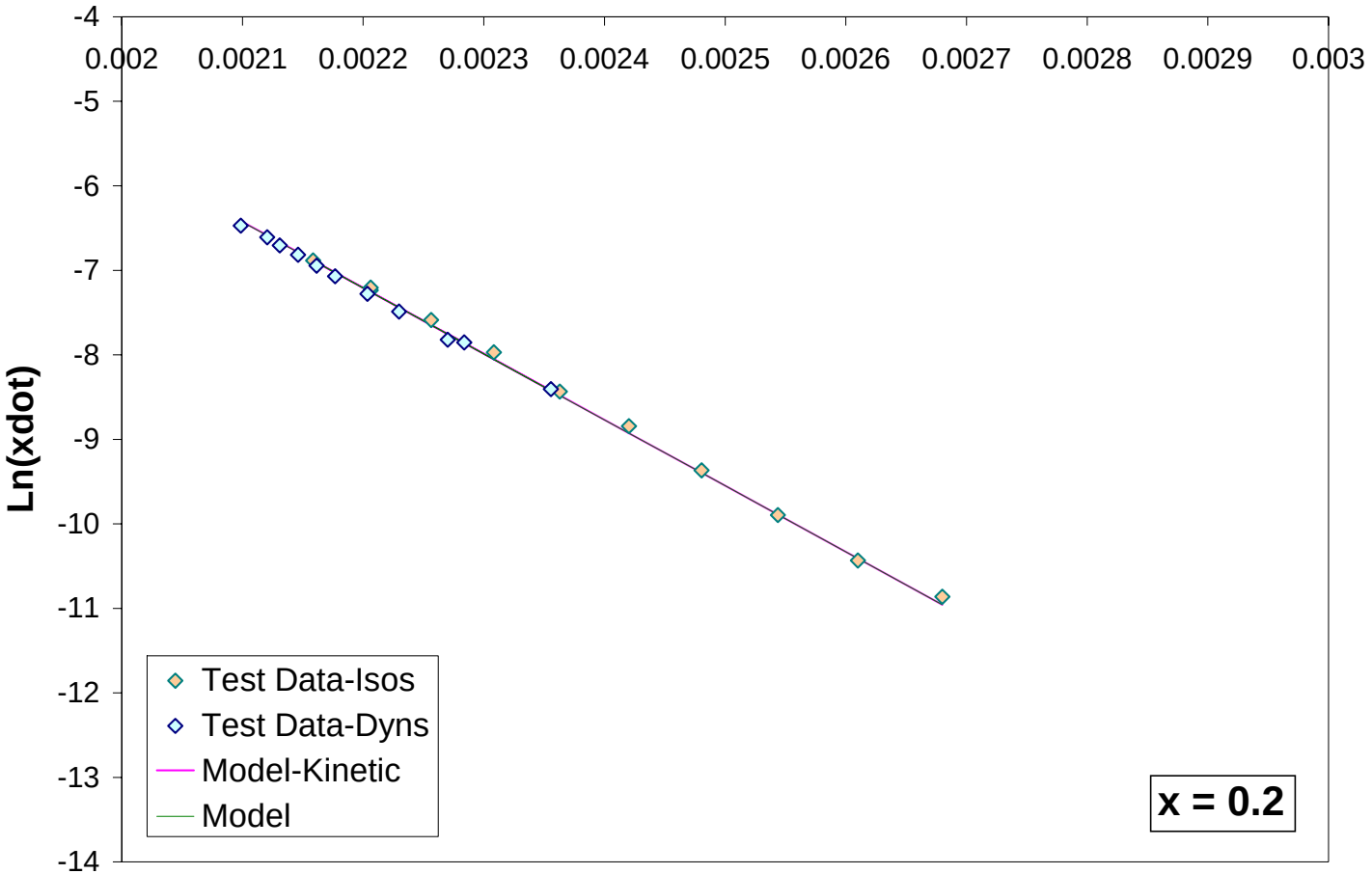
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.1

Ln(xdot)-1/T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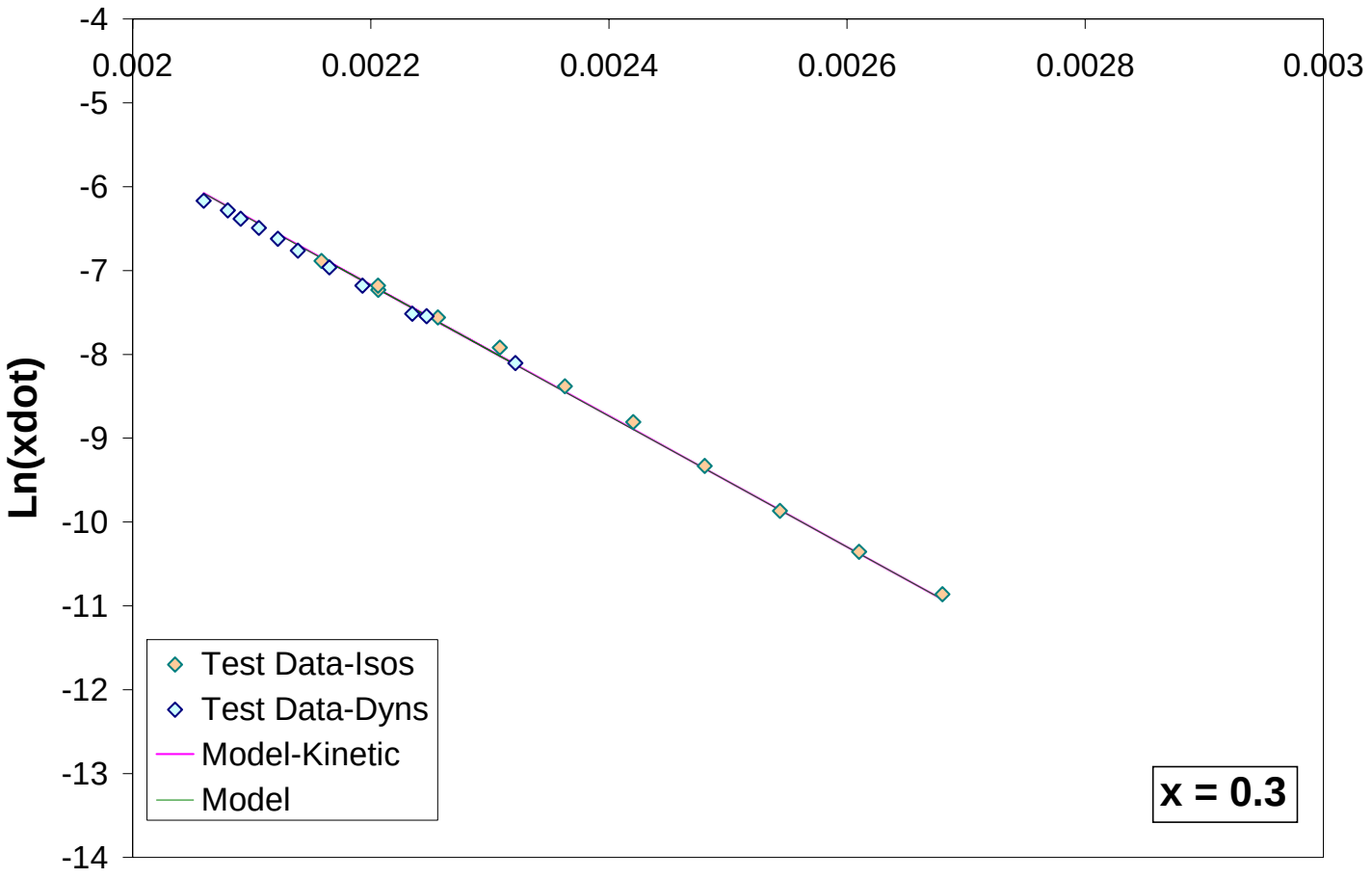
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
x = 0.2

Ln(xdot)-1/T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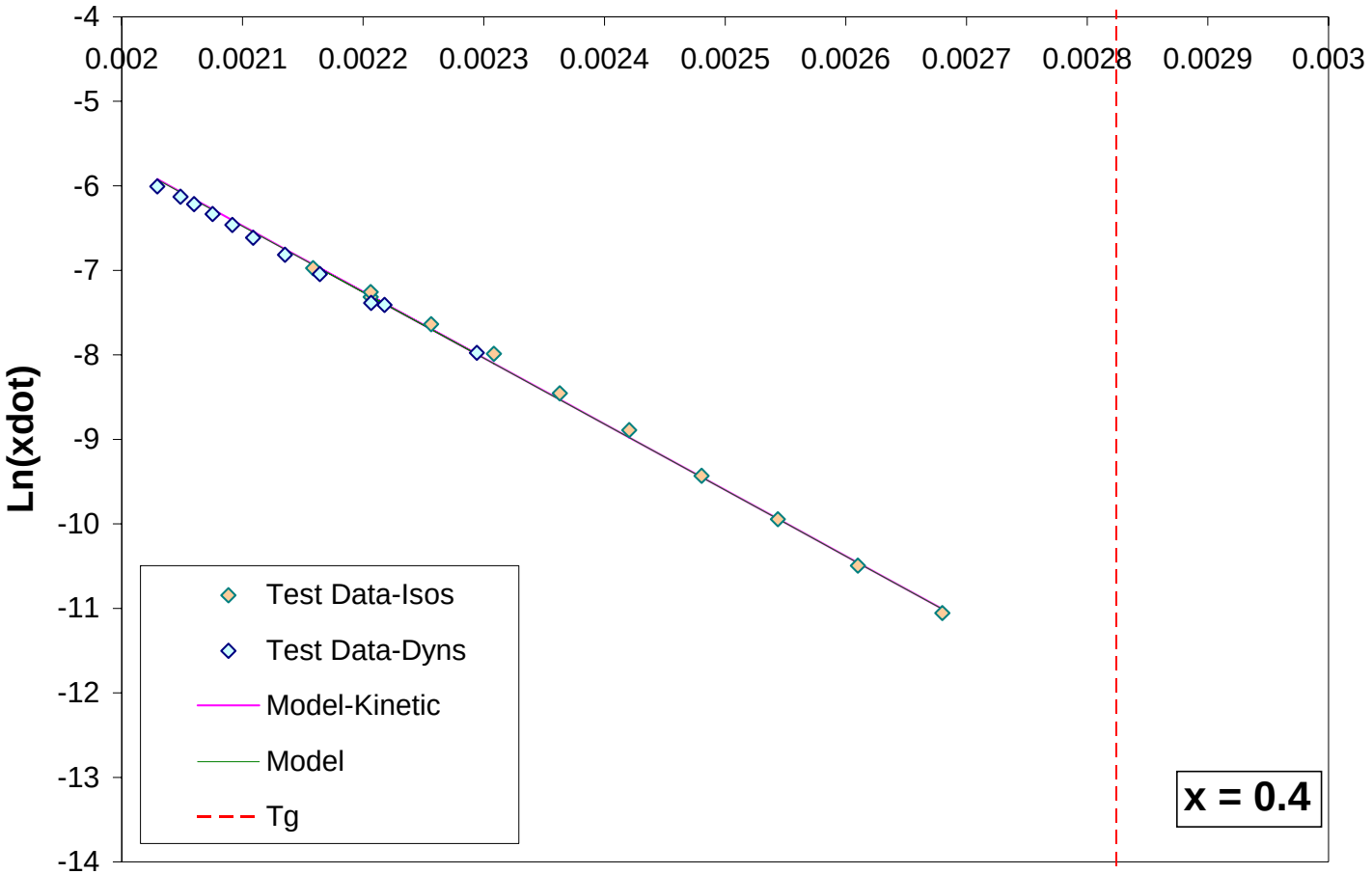
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
x = 0.3

Ln(xdot)-1/T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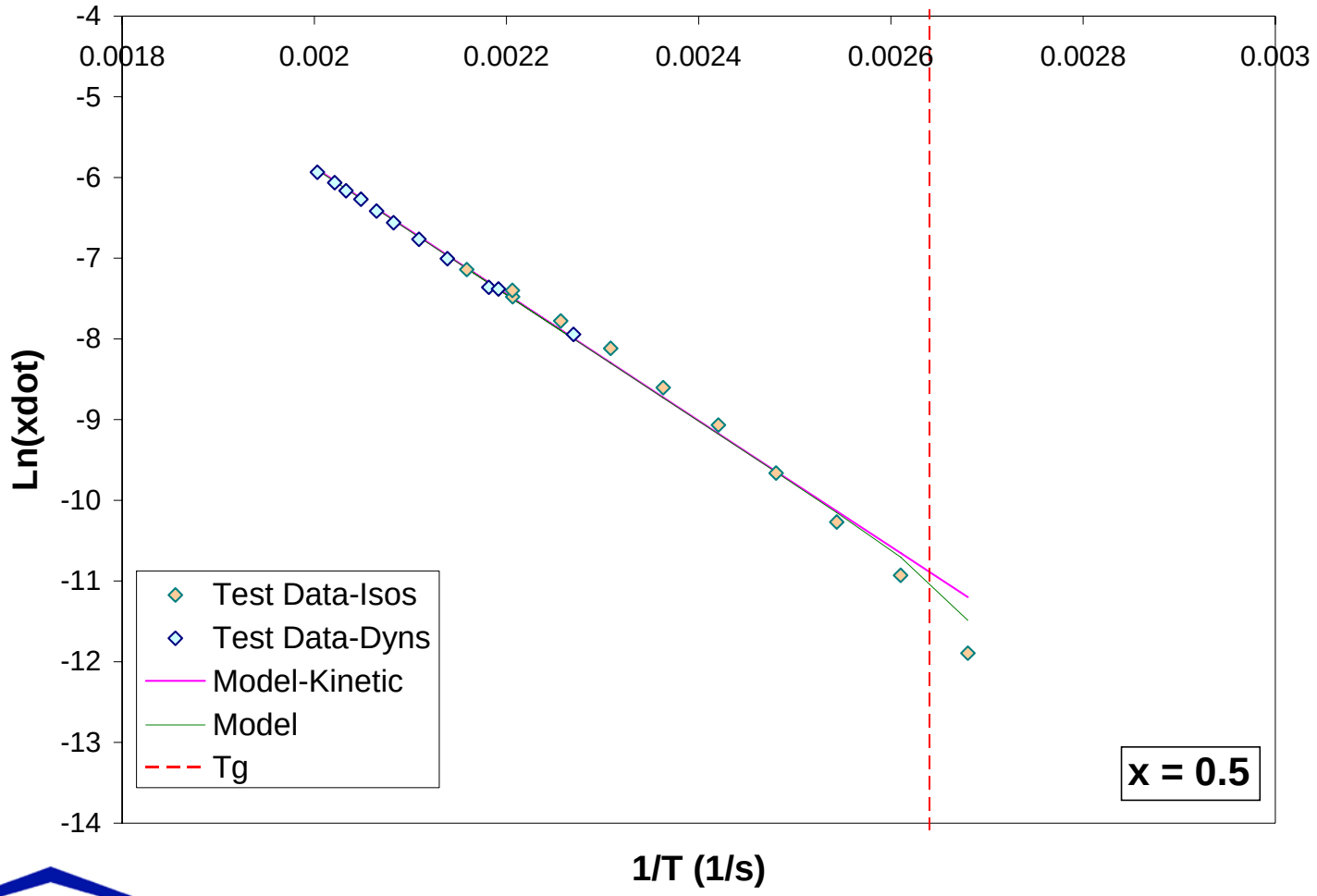
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.4

Ln(xdot)-1/T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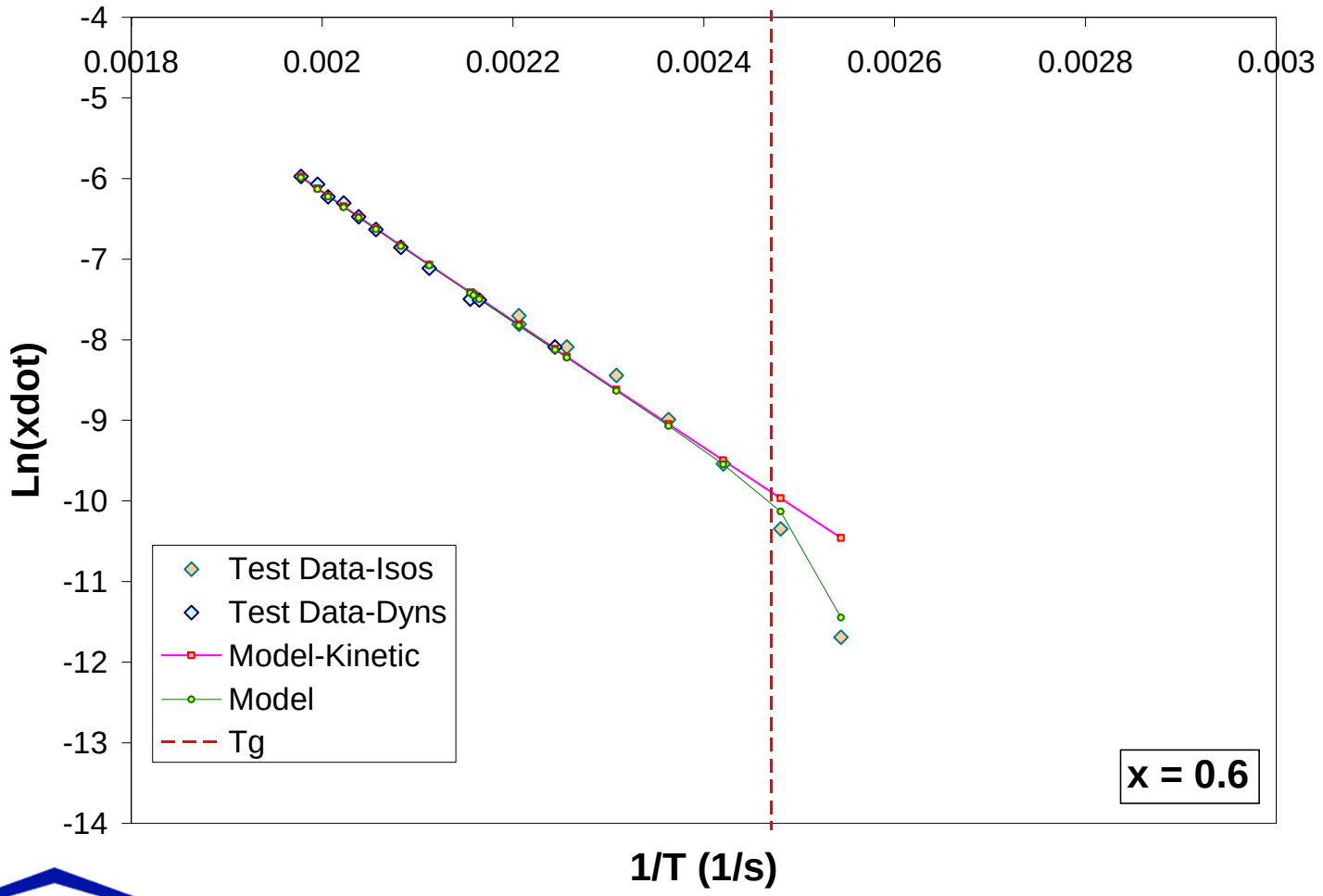
0.003
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.5

Ln(xdot)-1/T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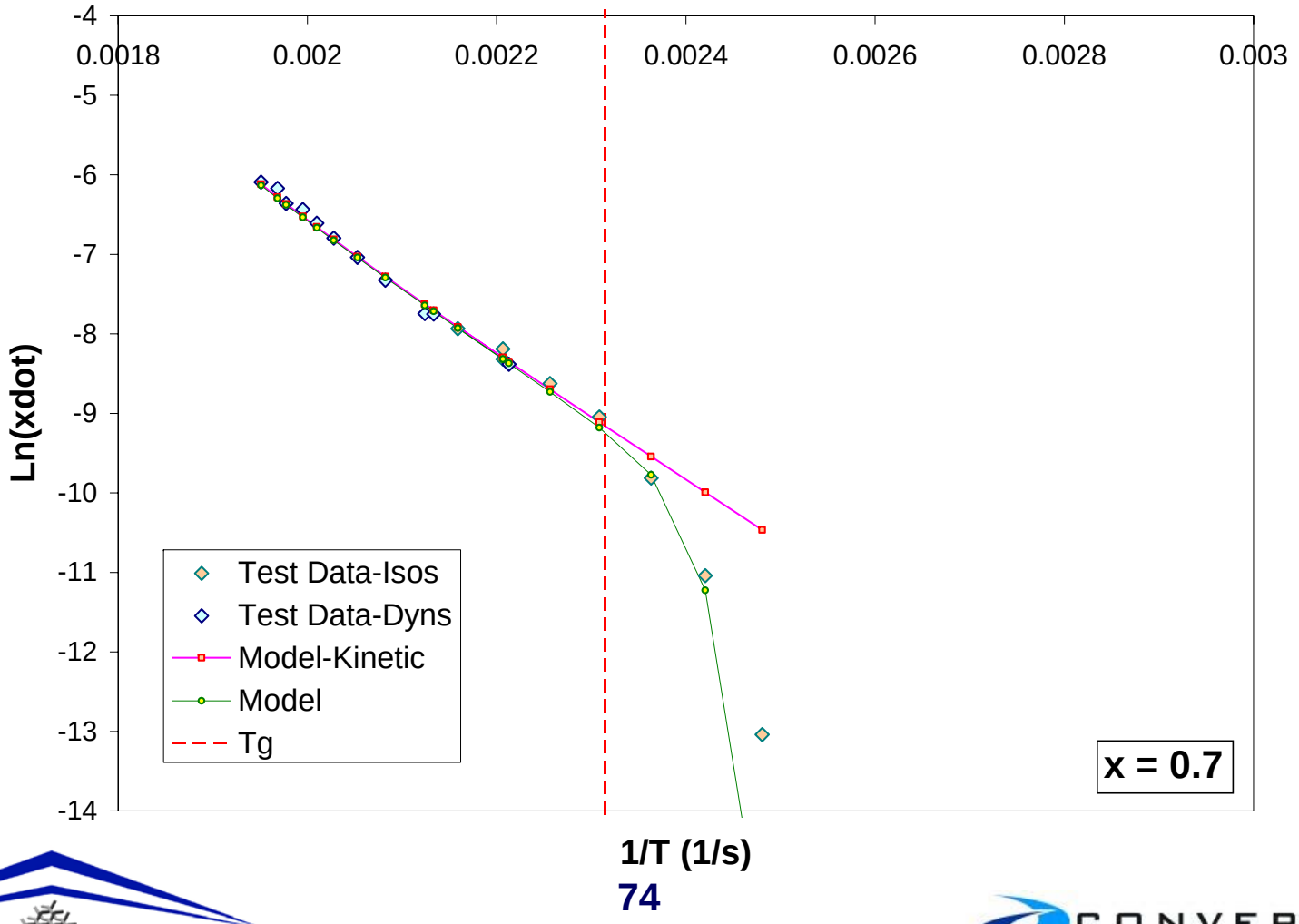
0.003
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.6

Ln(xdot)-1/T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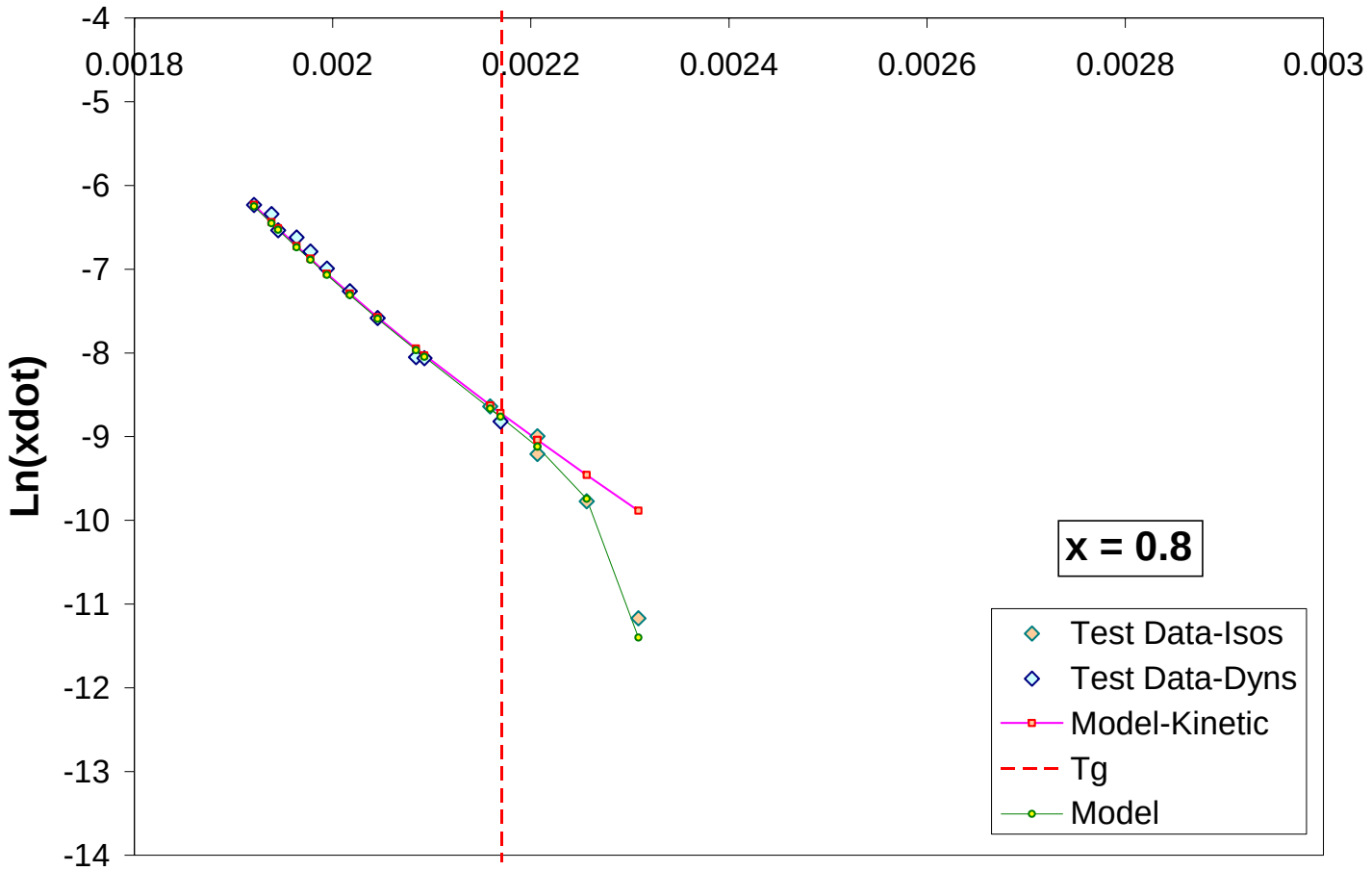
0.0028
0.003
Ln(xdot)
1/T (1/s)
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg
x = 0.7

Ln(xdot)-1/T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0.0024
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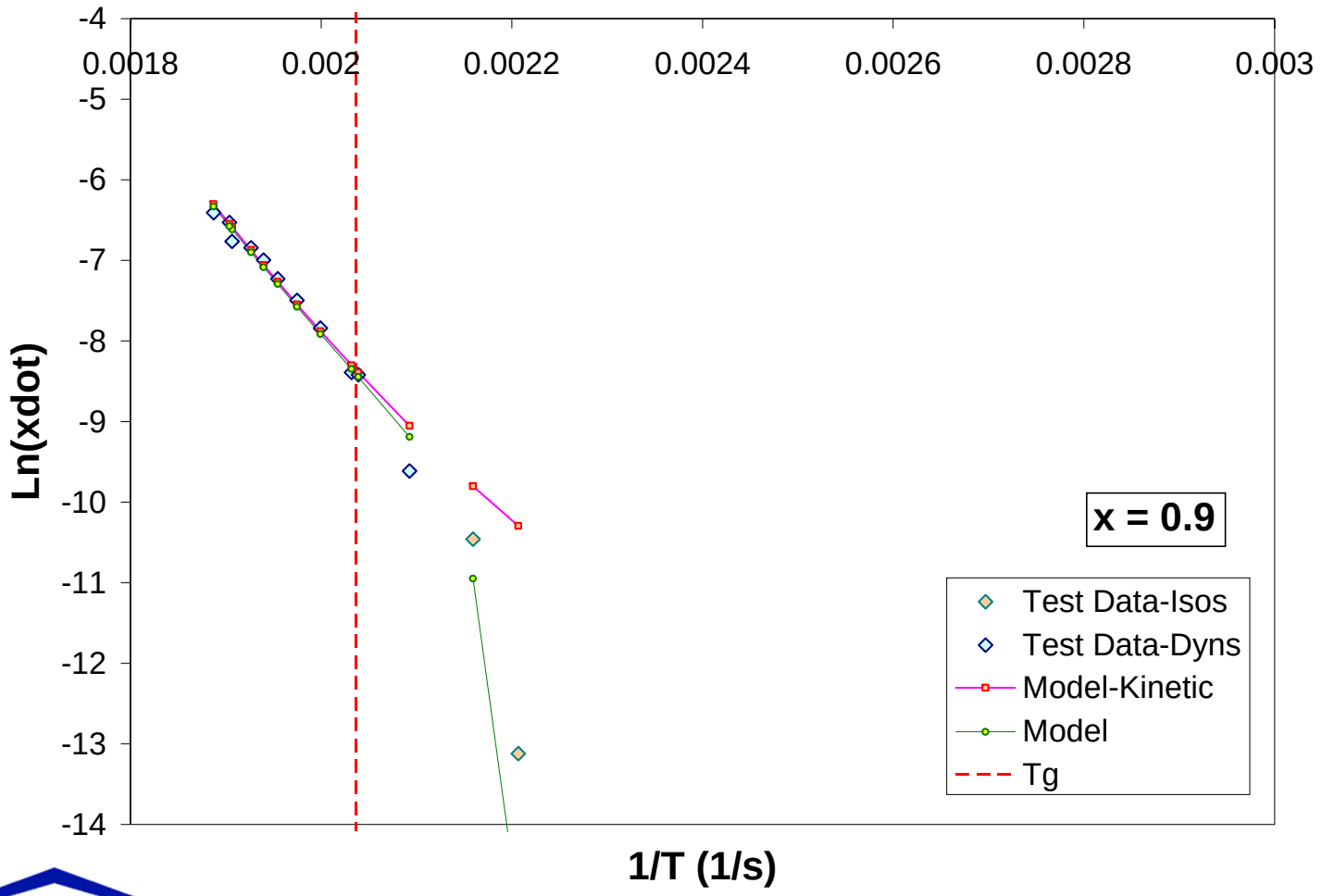
0.0028
0.003
Ln(xdot)
1/T (1/s)
x = 0.8
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Tg
Model

Ln(xdot)-1/T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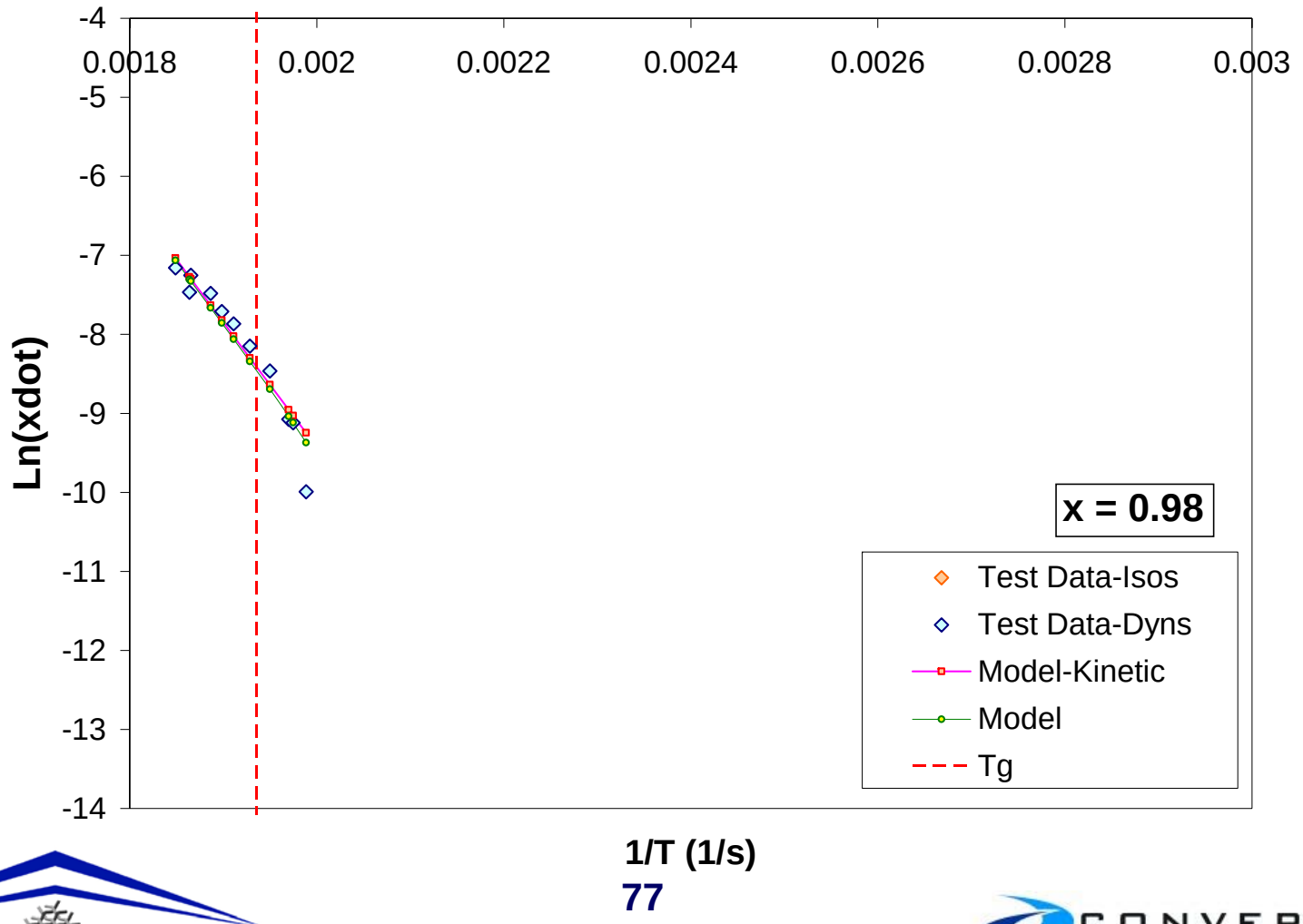
0.0028
0.003
Ln(xdot)
1/T (1/s)
x = 0.9
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg

Ln(xdot)-1/T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0.0018
0.002
0.0022
0.0024
0.0026
0.0028
0.003
Ln(xdot)
1/T (1/s)
x = 0.98
Test Data-Isos
Test Data-Dyns
Model-Kinetic
Model
Tg

Model Parameters

Parameter	Reaction 1	Reaction 2
k_0 (1/s)	153,900.5	3.963E+11
E_a (J/mol)	64,929.5	133,168.3
l	2.347	1.029
r	1.00	1.00
m	0.00	0.00
n_2	1.00	1.00
b	0.1594	0.00
n	1.413	5.586

Parameter	Value (unit)
T_{g_0}	-7 (ºC)
T_{g_∞}	250 (ºC)
λ	0.78

Parameter	Value (unit)
k_{d0}	4.0 (1/s)
B	0.21
a_1	4.8E-04 (1/ºC)
a_2	4.8E-04 (1/ºC)
$T_{g_{a1}}$	0 (ºC)
$T_{g_{a2}}$	100 (ºC)
b_2	0.021
b_1	0.031
$T_{g_{b1}}$	120 (ºC)
$T_{g_{b2}}$	195 (ºC)

Model's range of validity

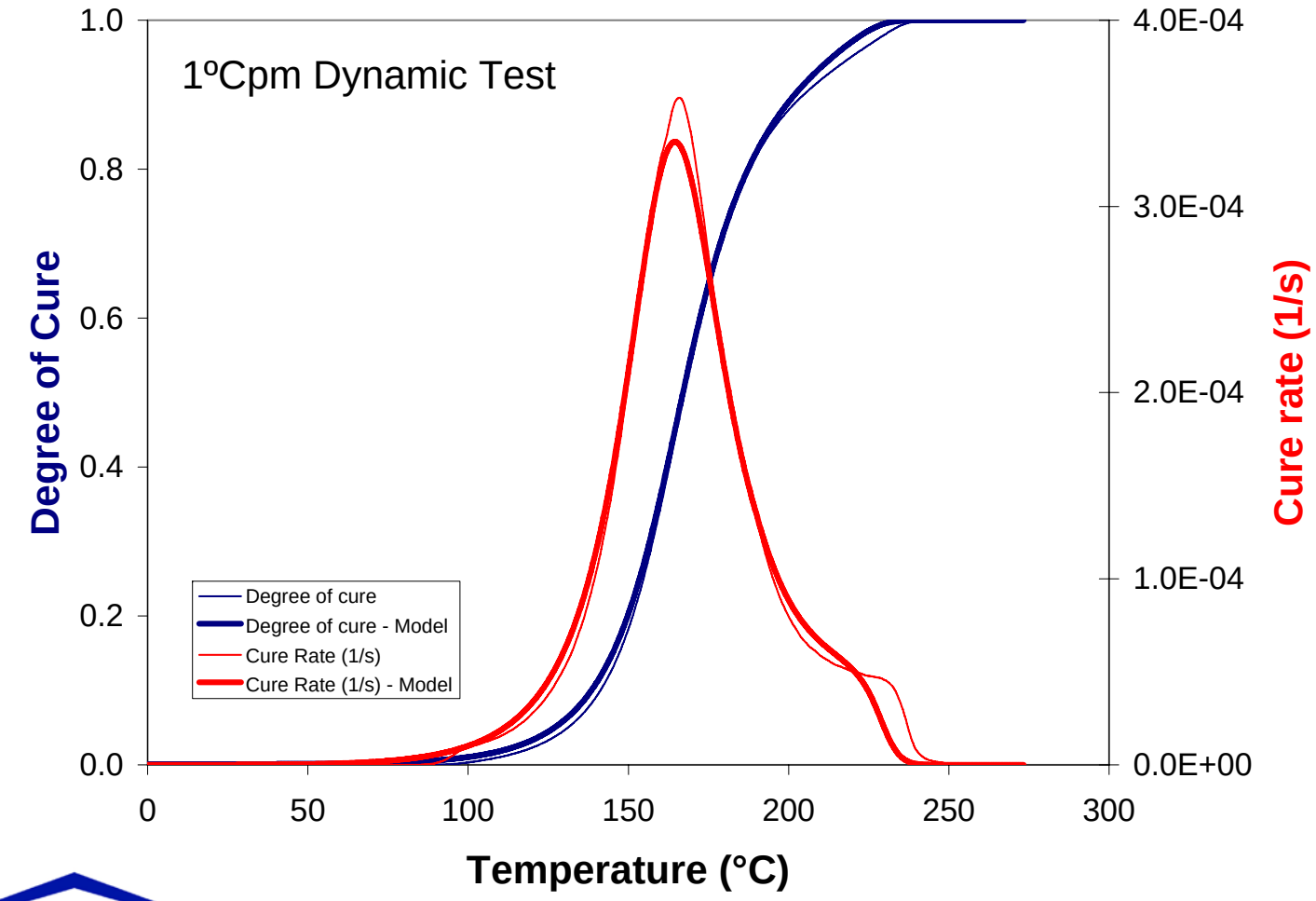
100ºC < Iso Temperature < 190ºC

-90ºC < Dyn Temperature < 275ºC

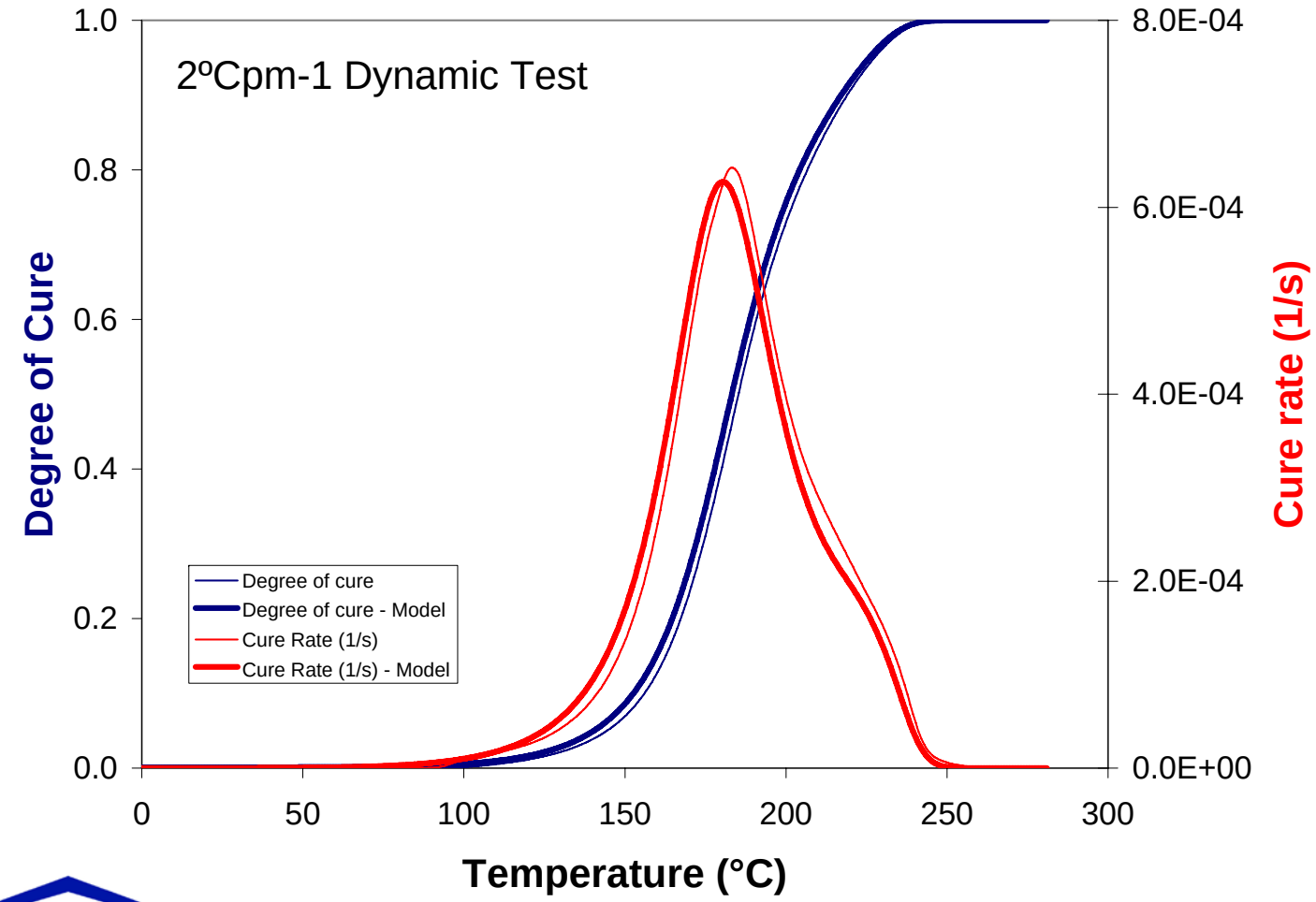
NCAMP

CONVERGENT MANUFACTURING TECHNOLOGIES

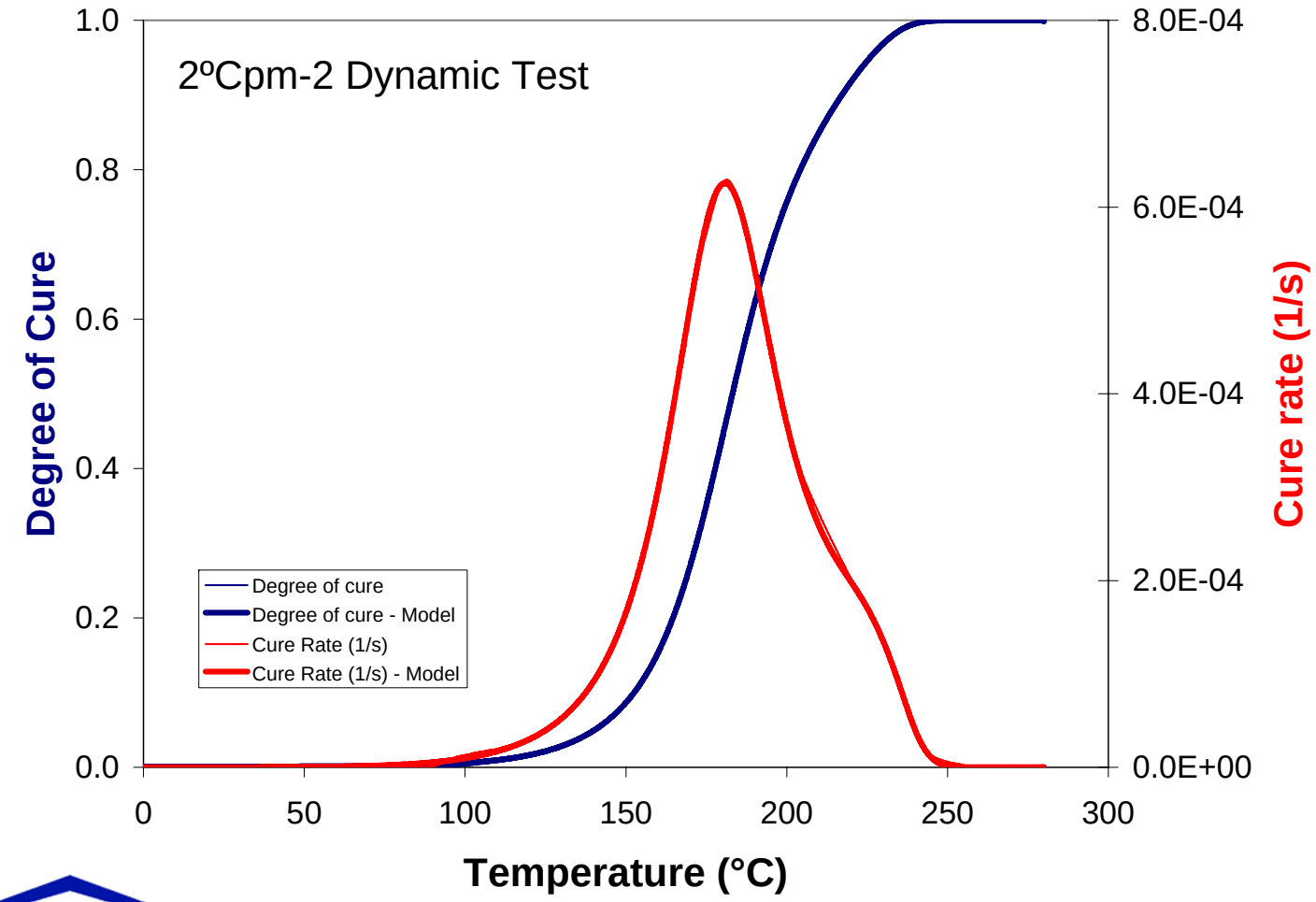
Dynamic Tests – 1cpm



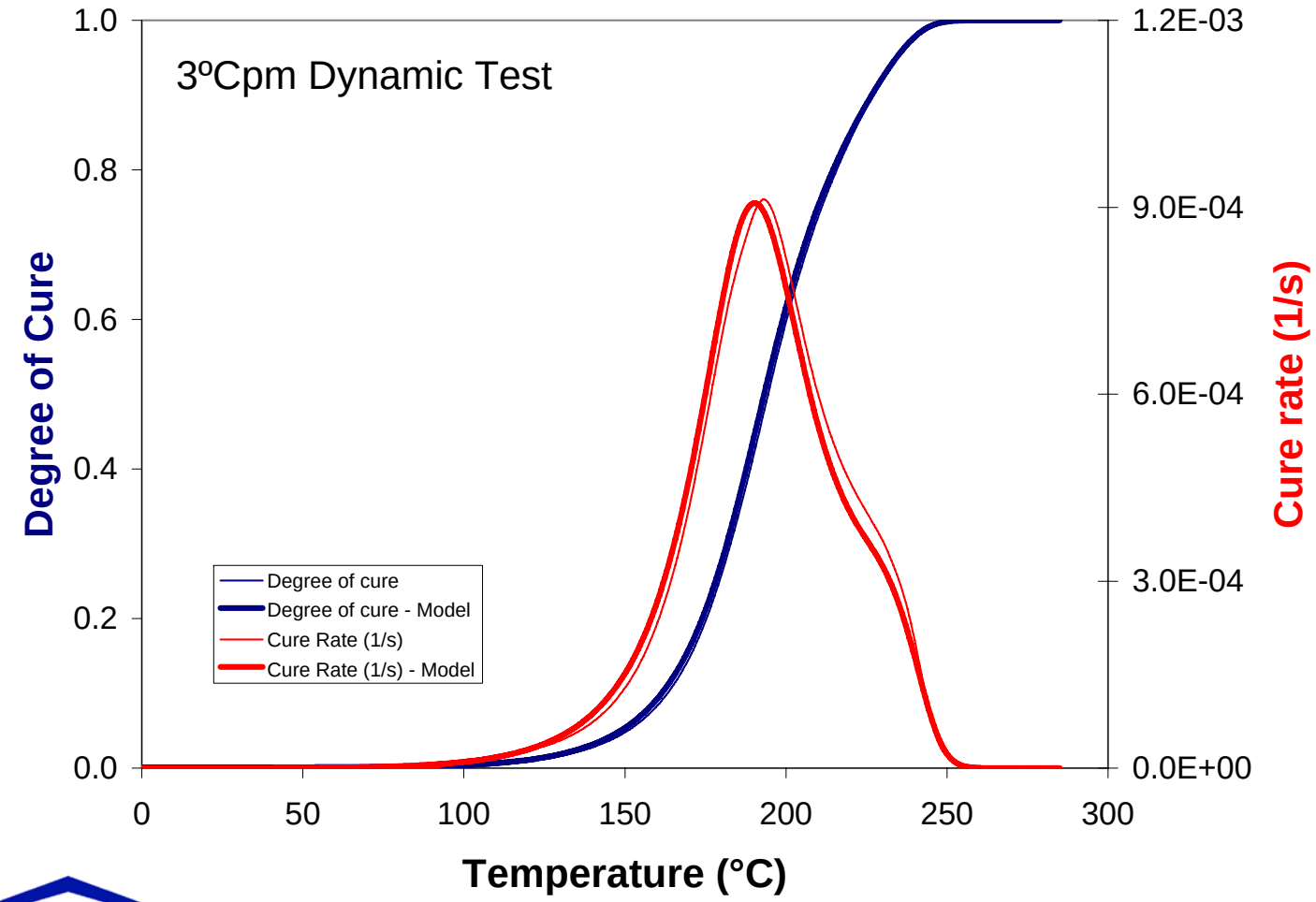
Dynamic Tests – 2cpm-1



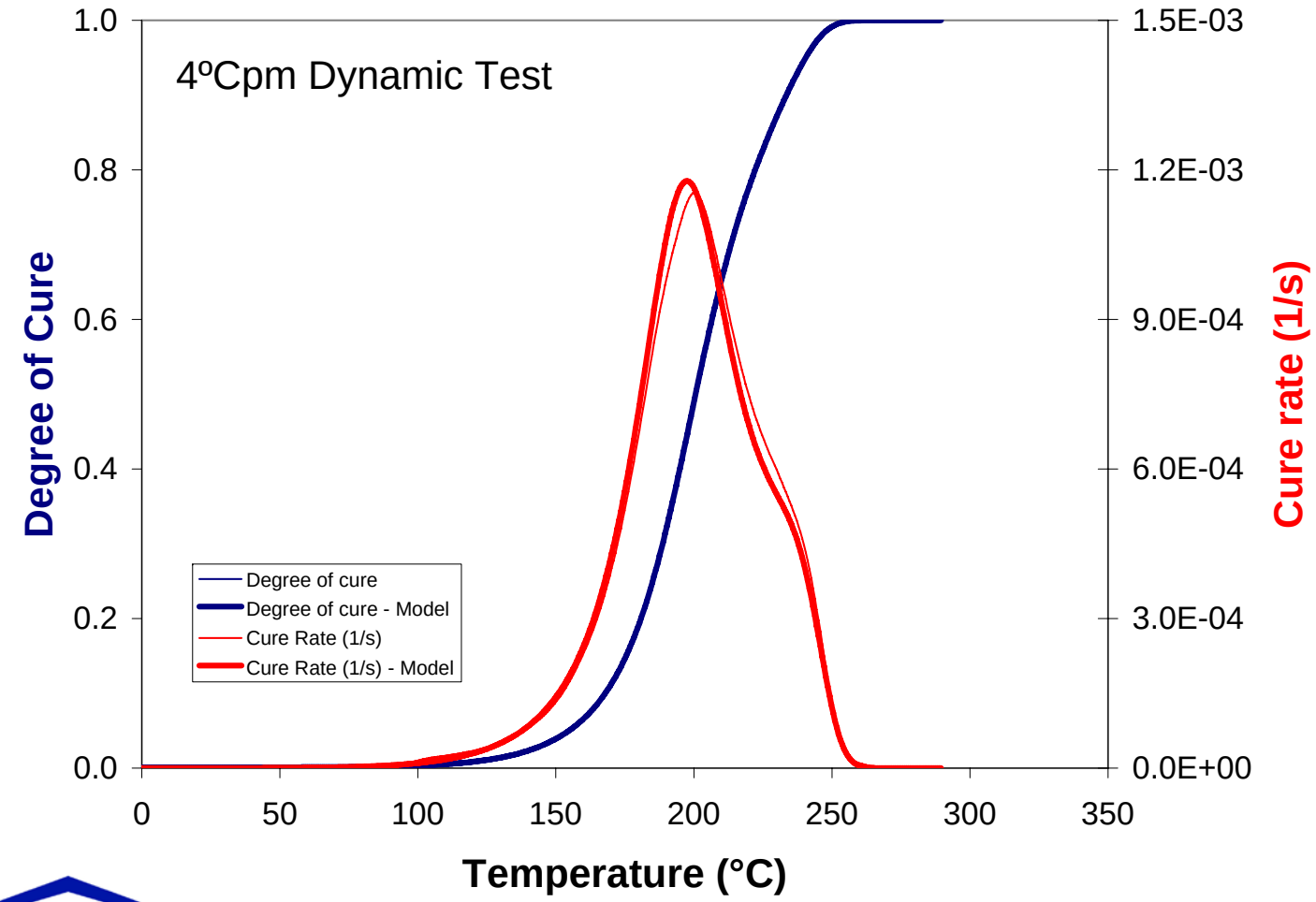
Dynamic Tests – 2cpm-2



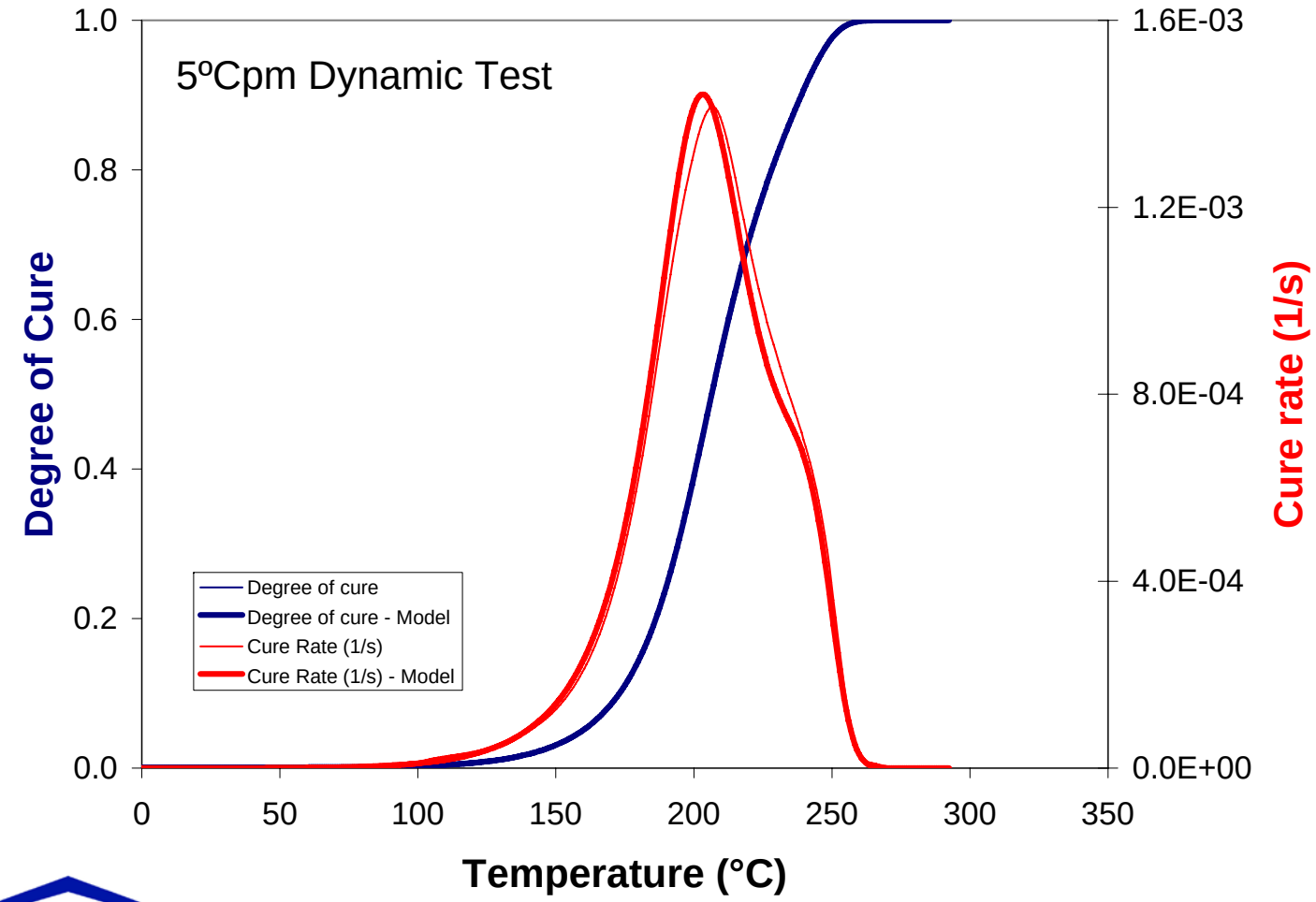
Dynamic Tests – 3cpm



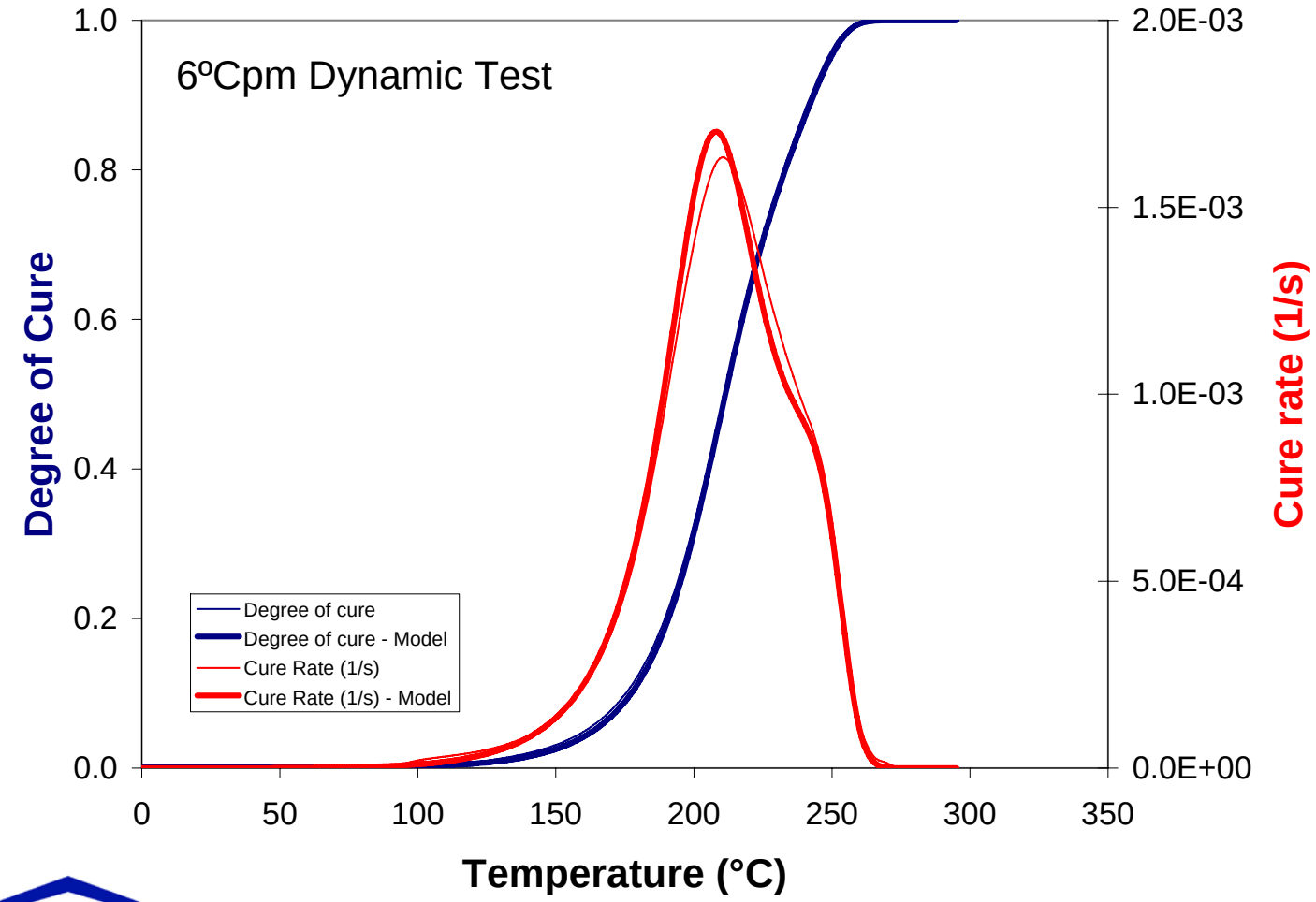
Dynamic Tests – 4cpm



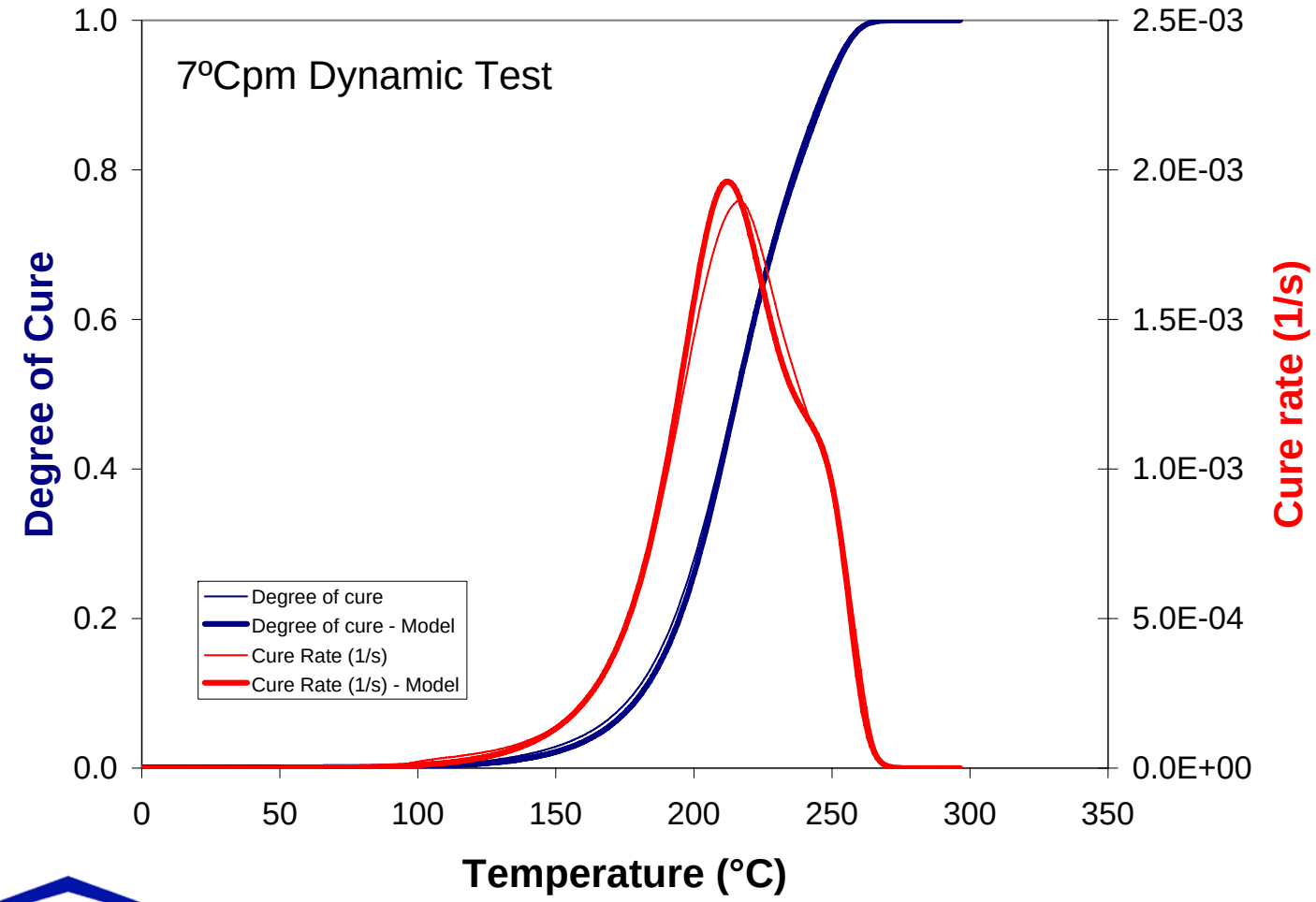
Dynamic Tests – 5cpm



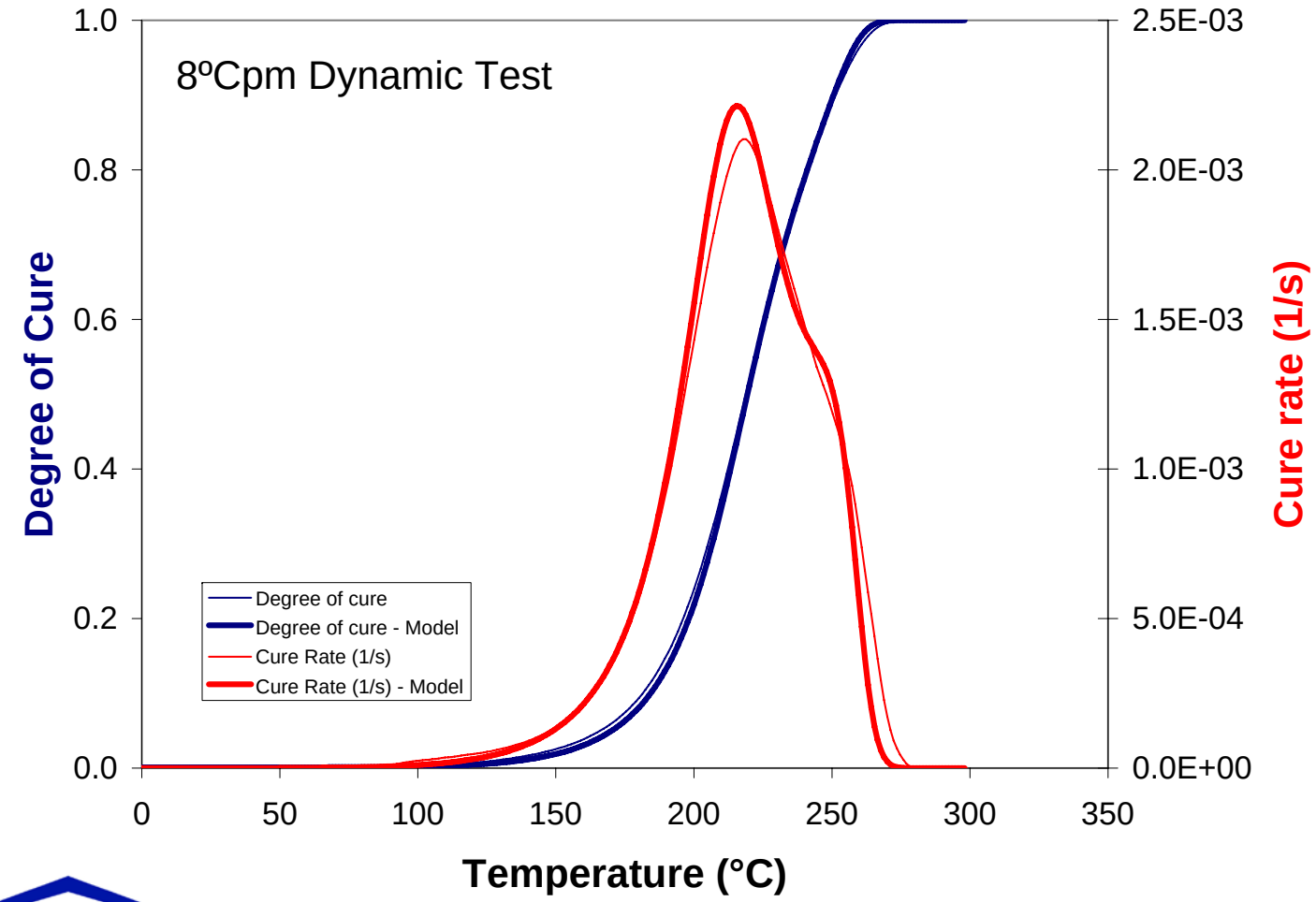
Dynamic Tests – 6cpm



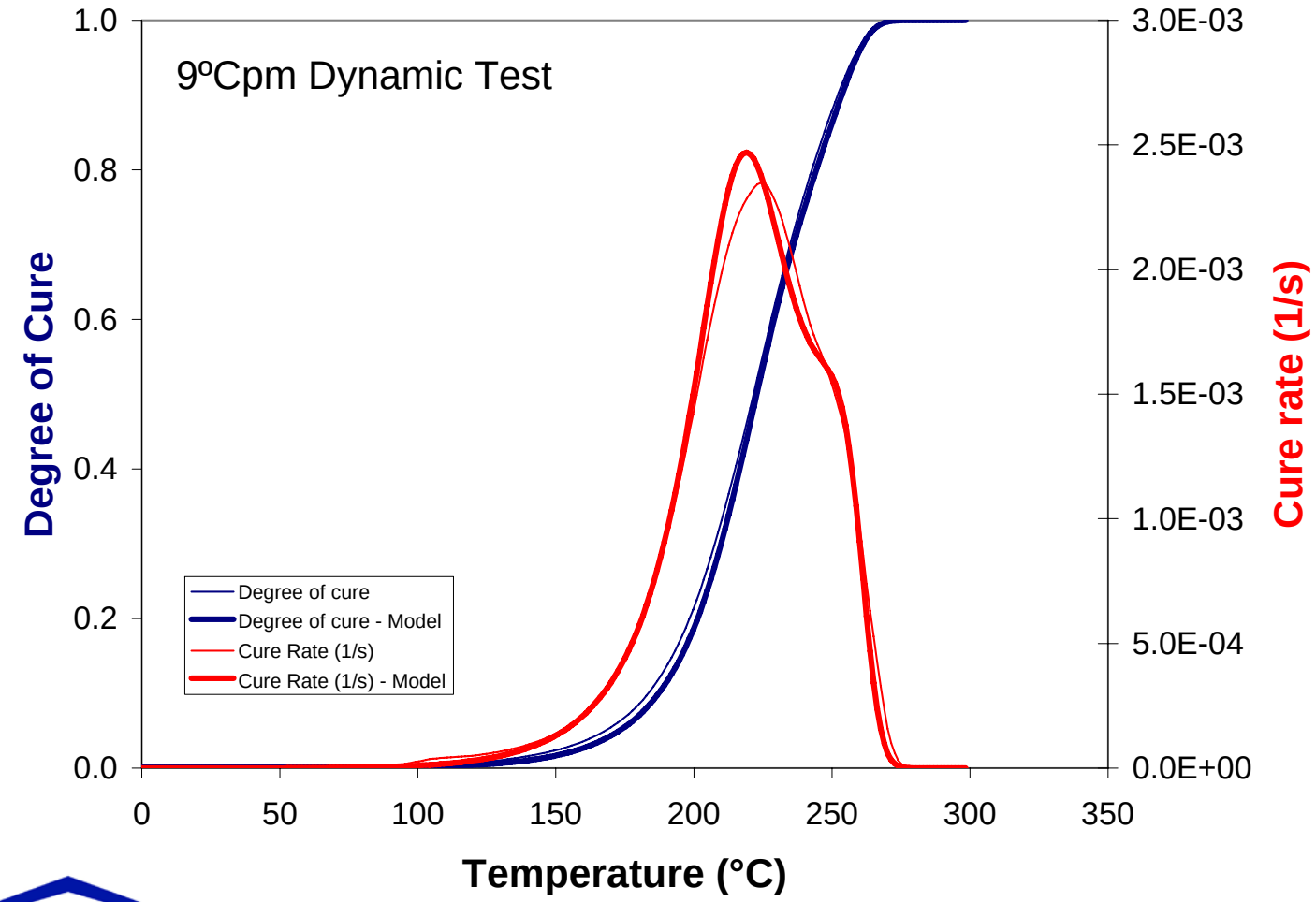
Dynamic Tests – 7cpm



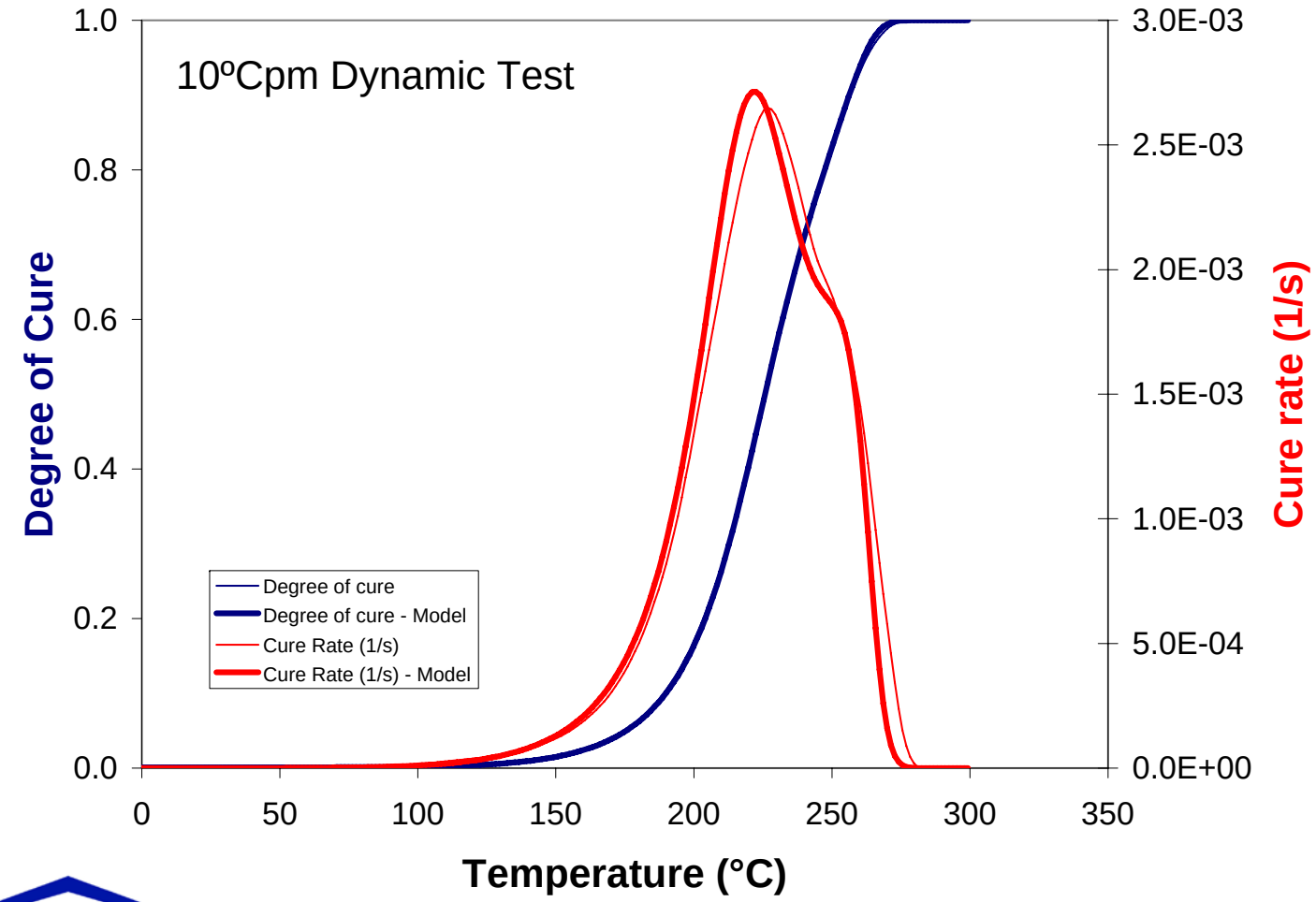
Dynamic Tests – 8cpm



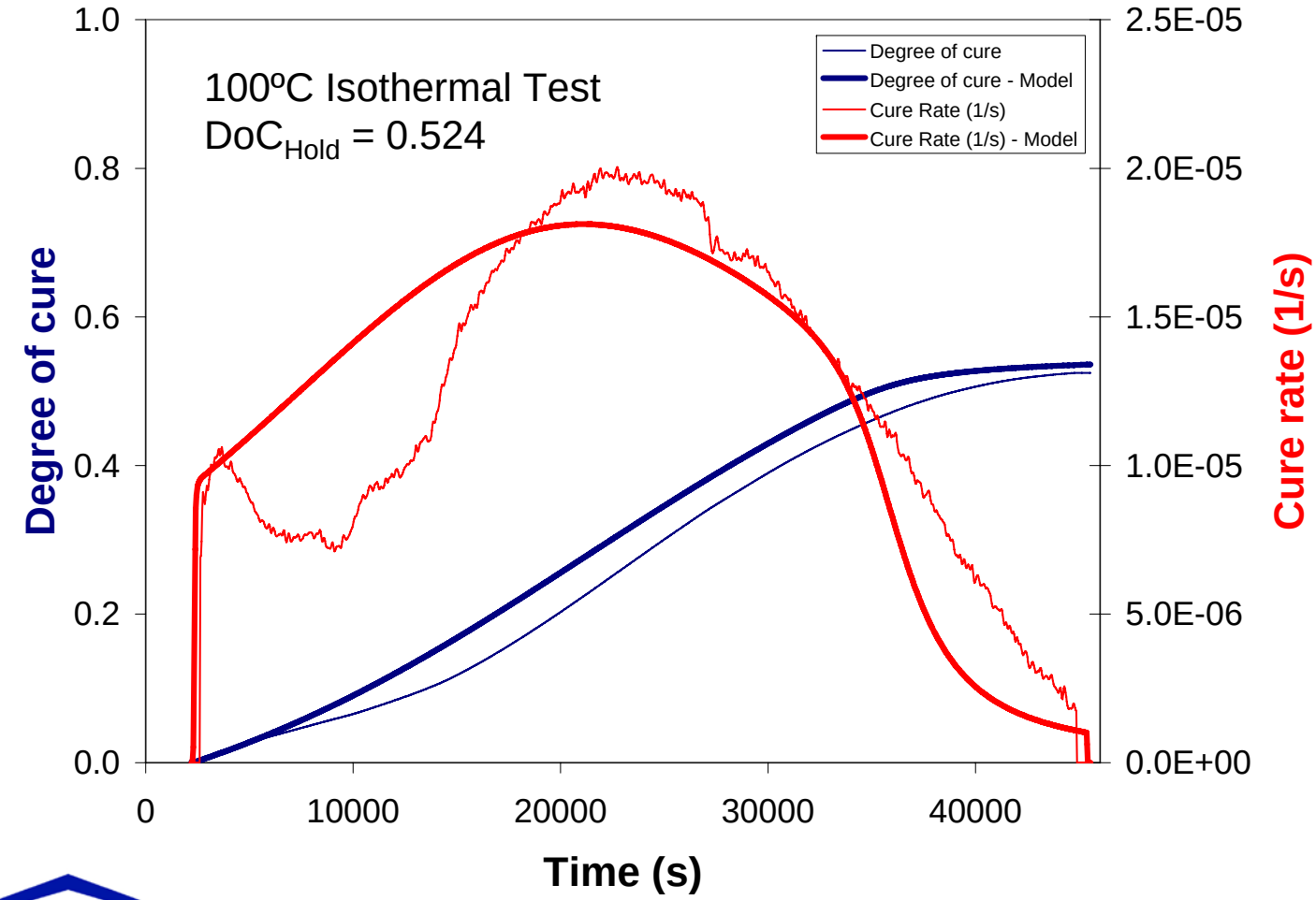
Dynamic Tests – 9cpm



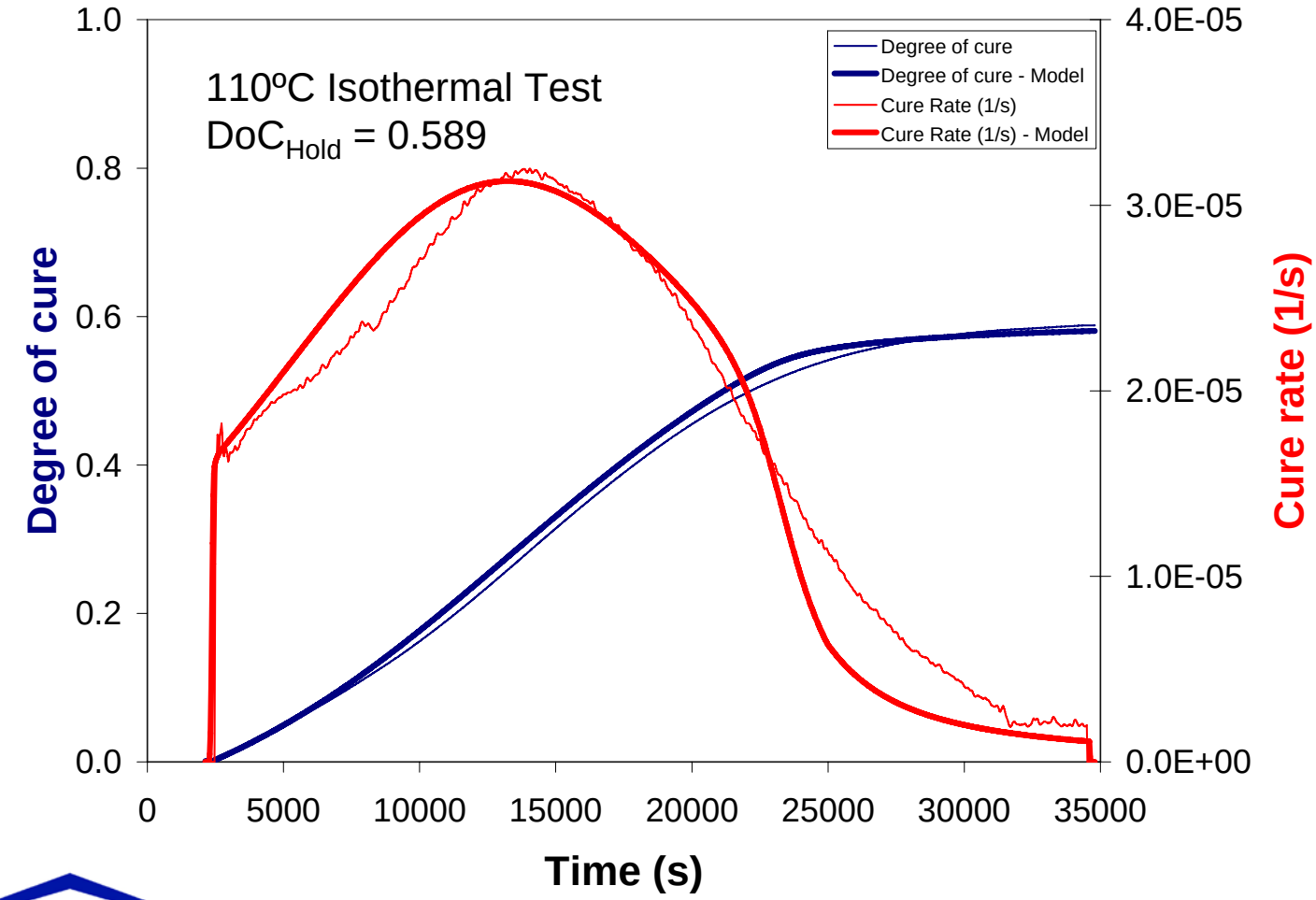
Dynamic Tests – 10cpm



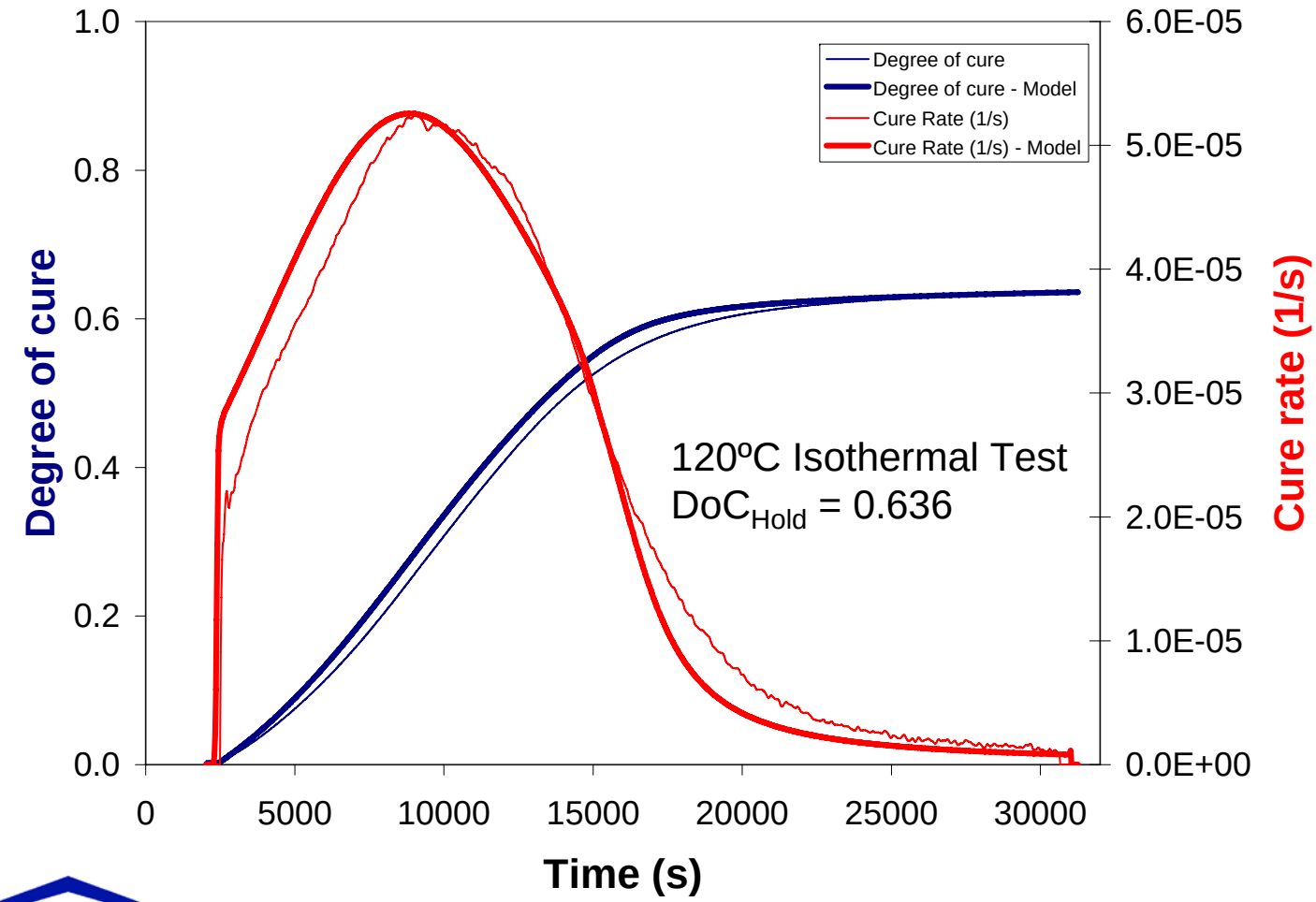
Isothermal Tests - 100ºC



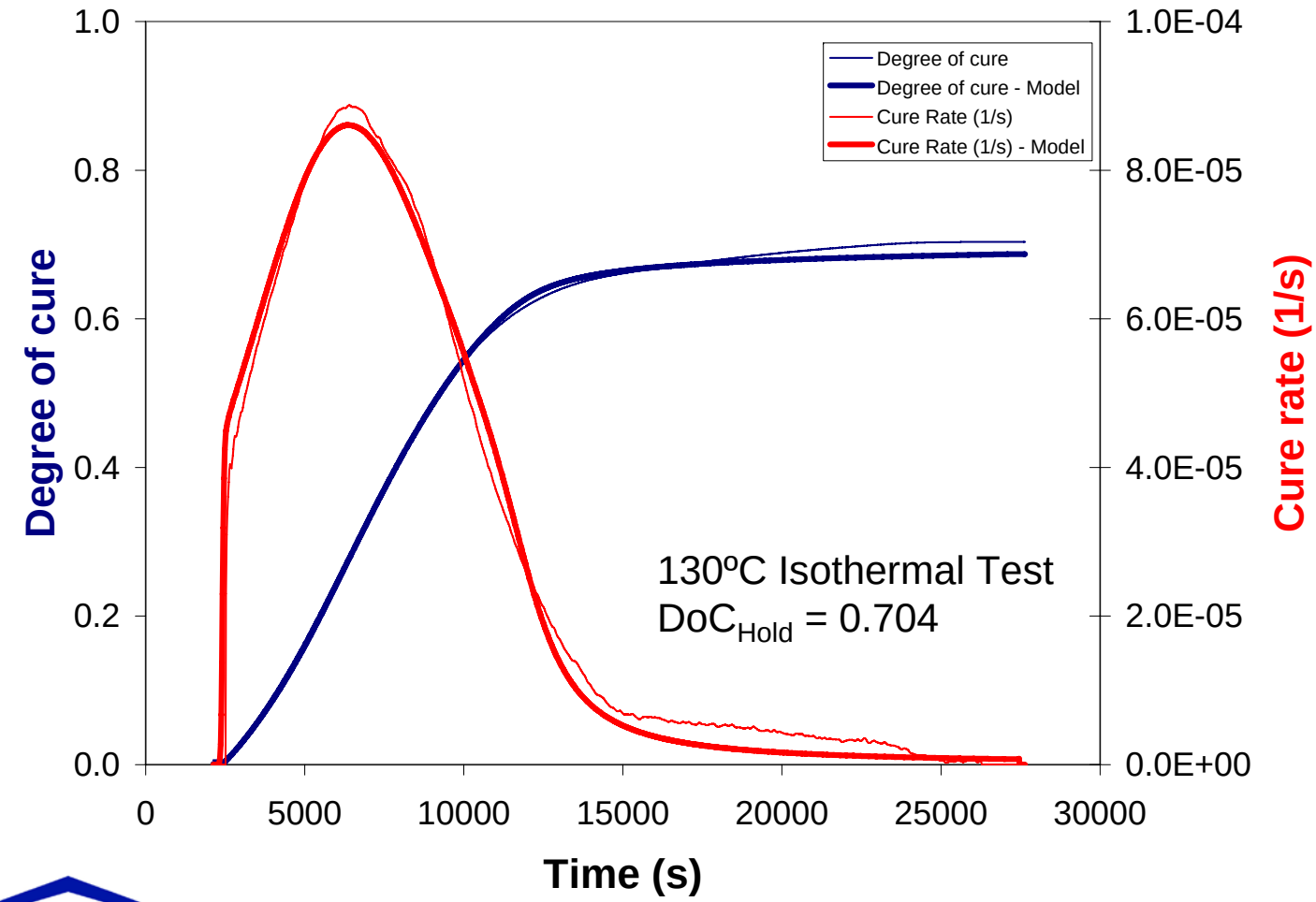
Isothermal Tests - 110ºC



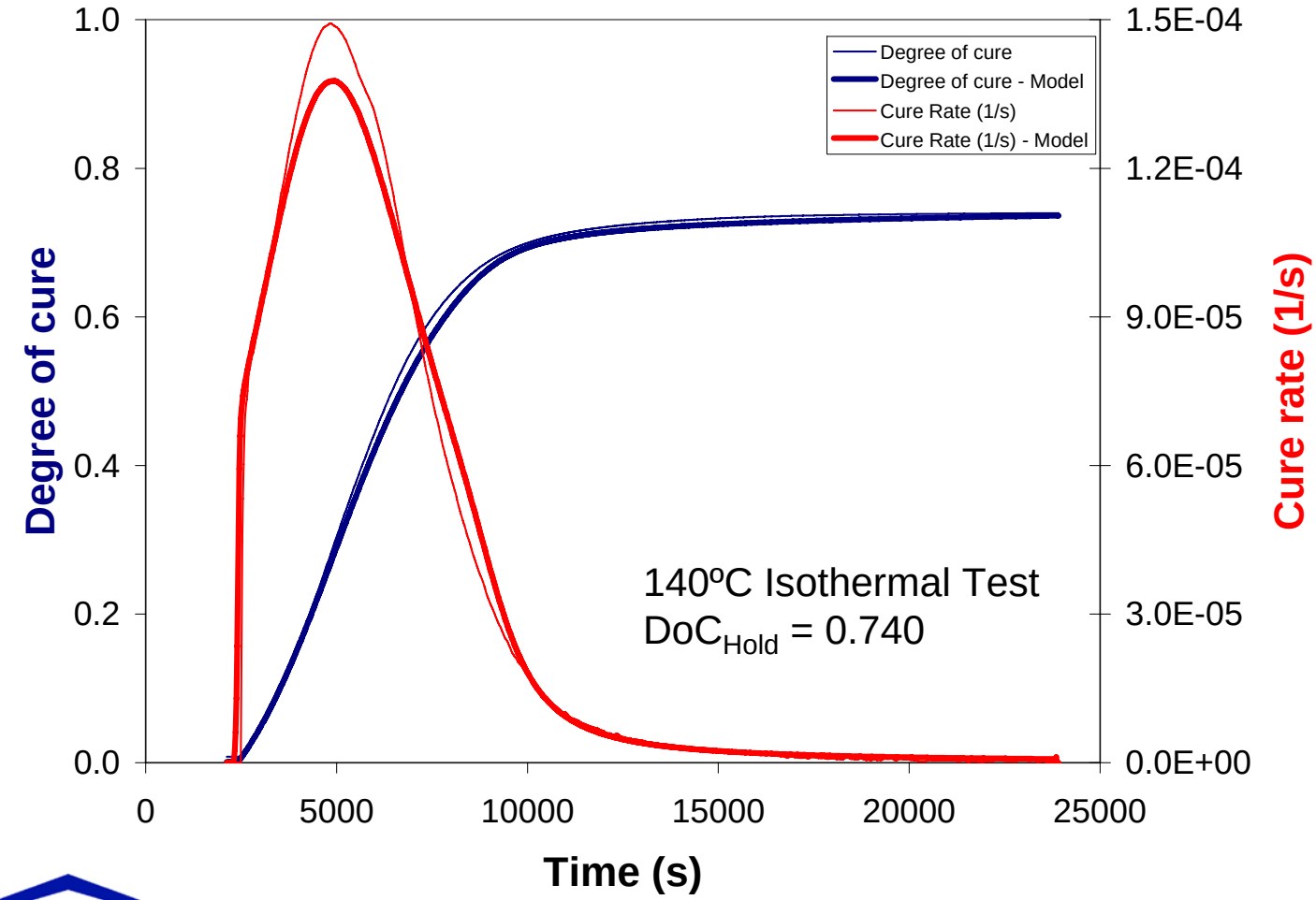
Isothermal Tests - 120ºC



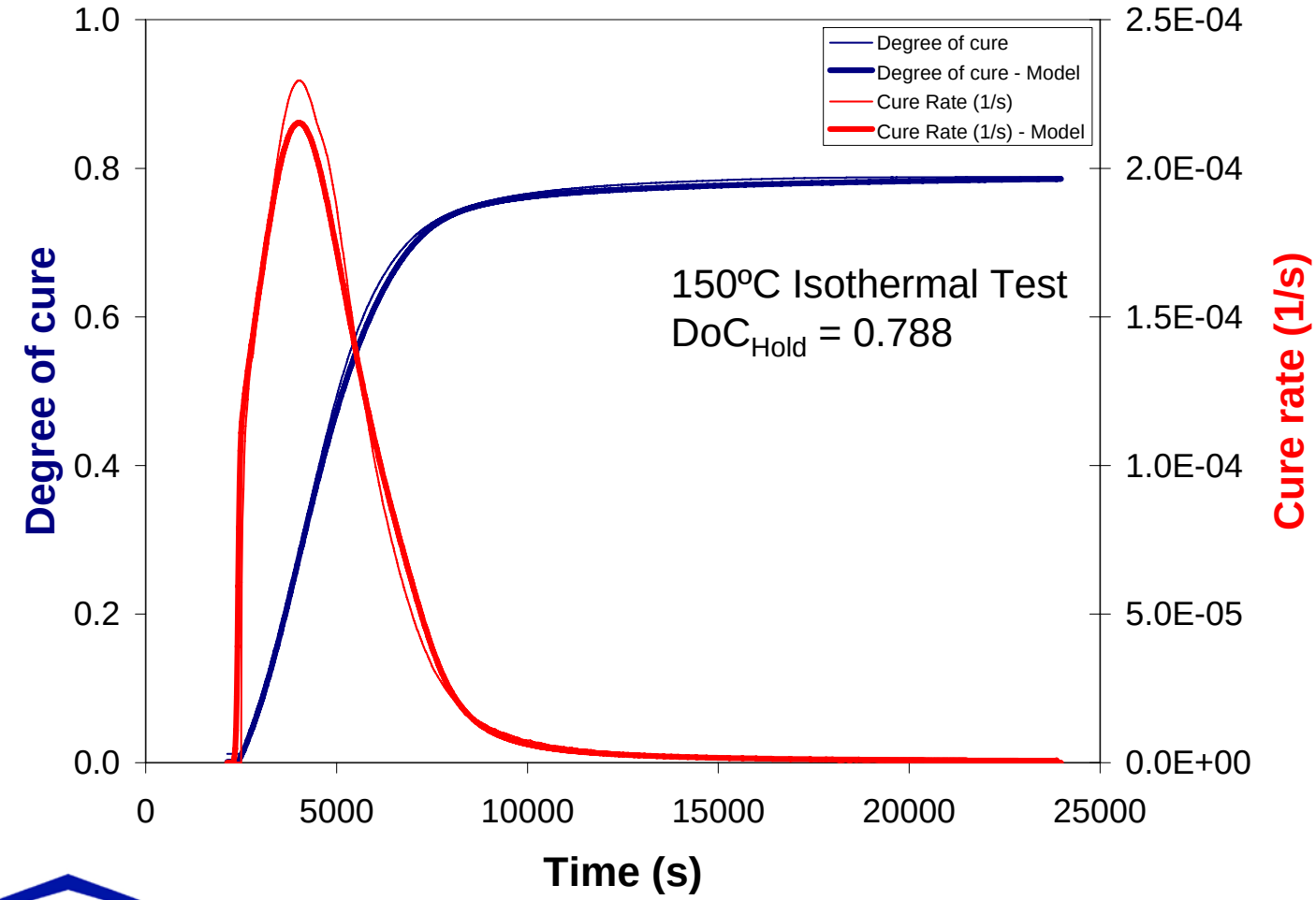
Isothermal Tests - 130ºC



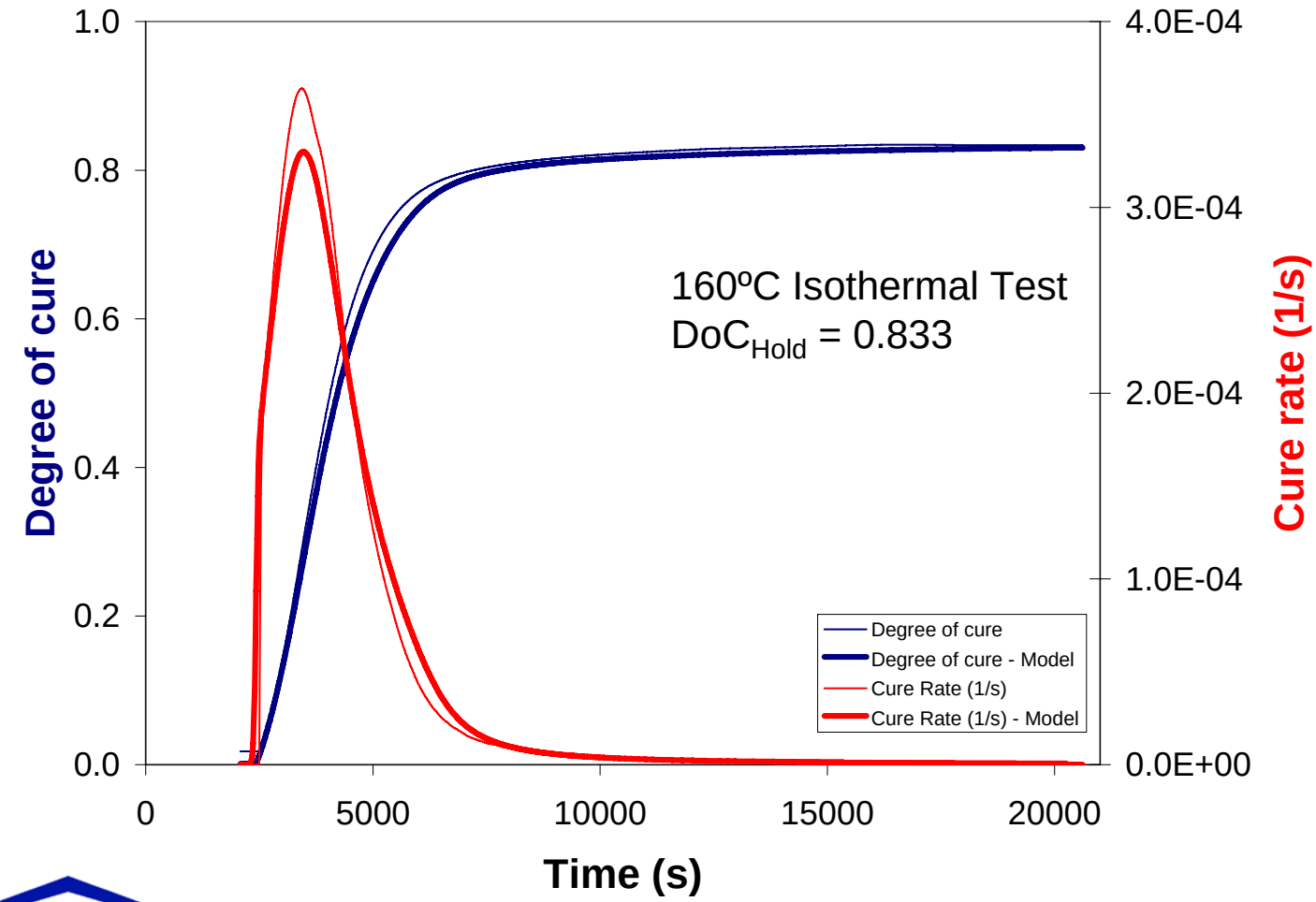
Isothermal Tests - 140ºC



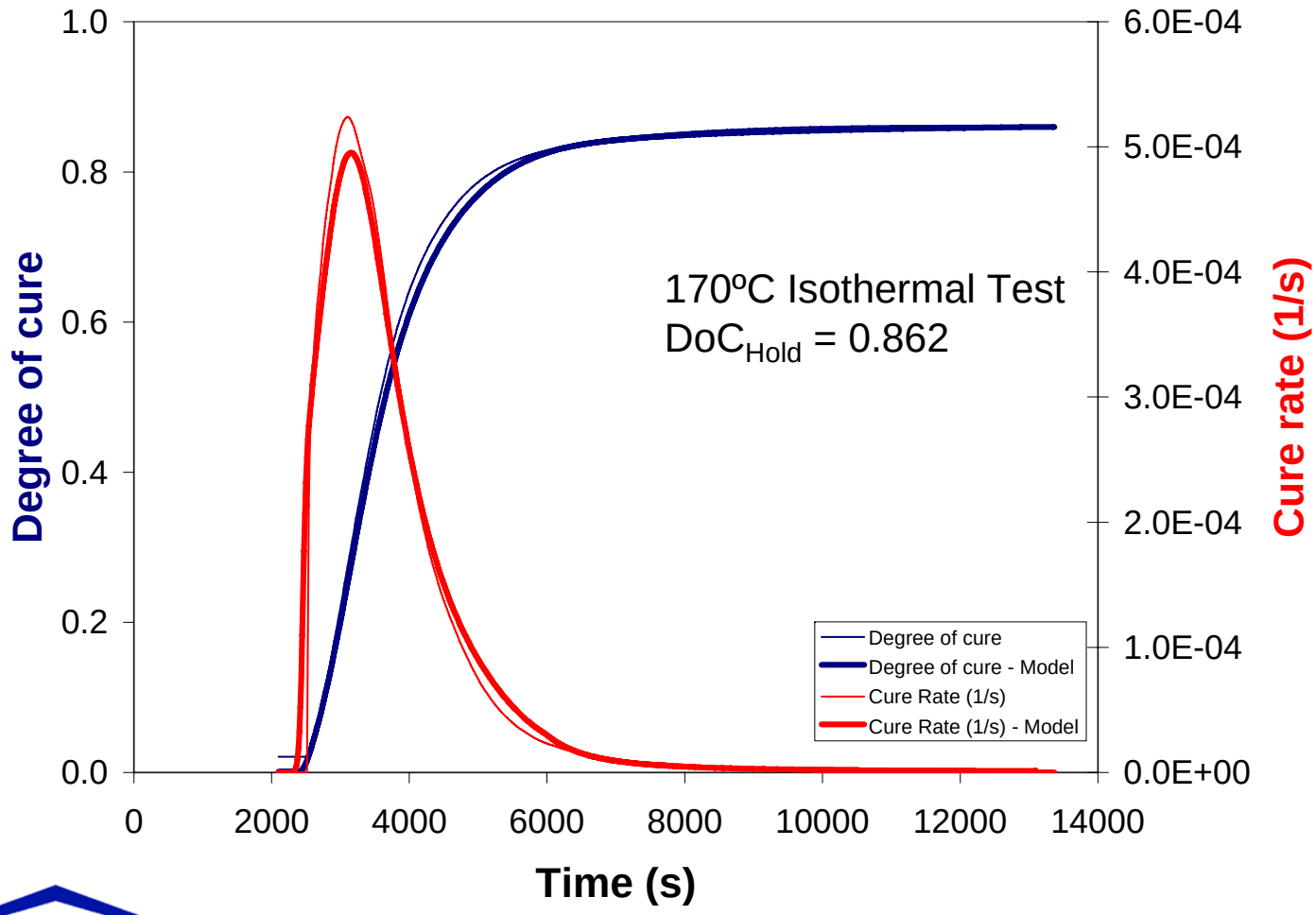
Isothermal Tests - 150ºC



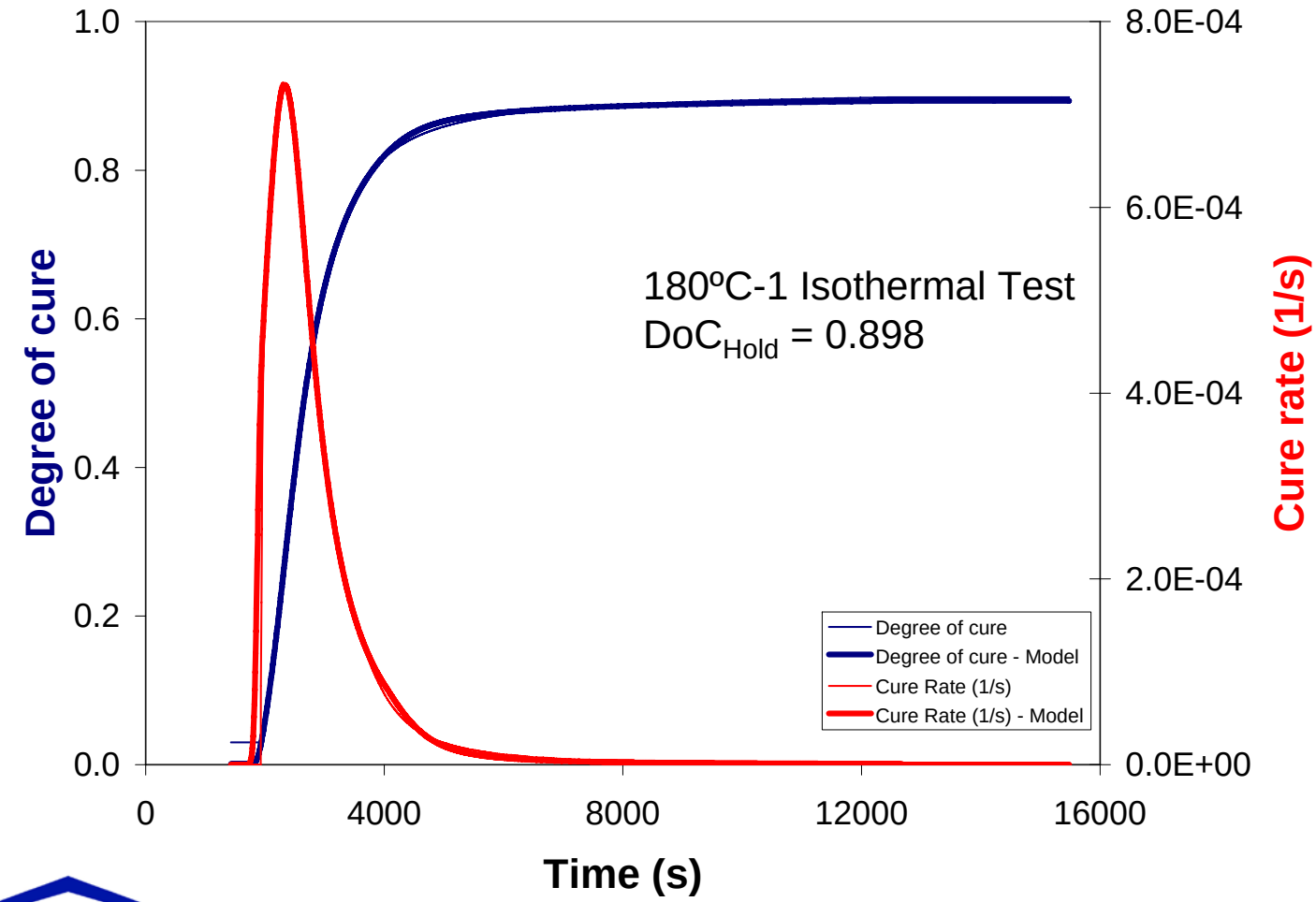
Isothermal Tests - 160ºC



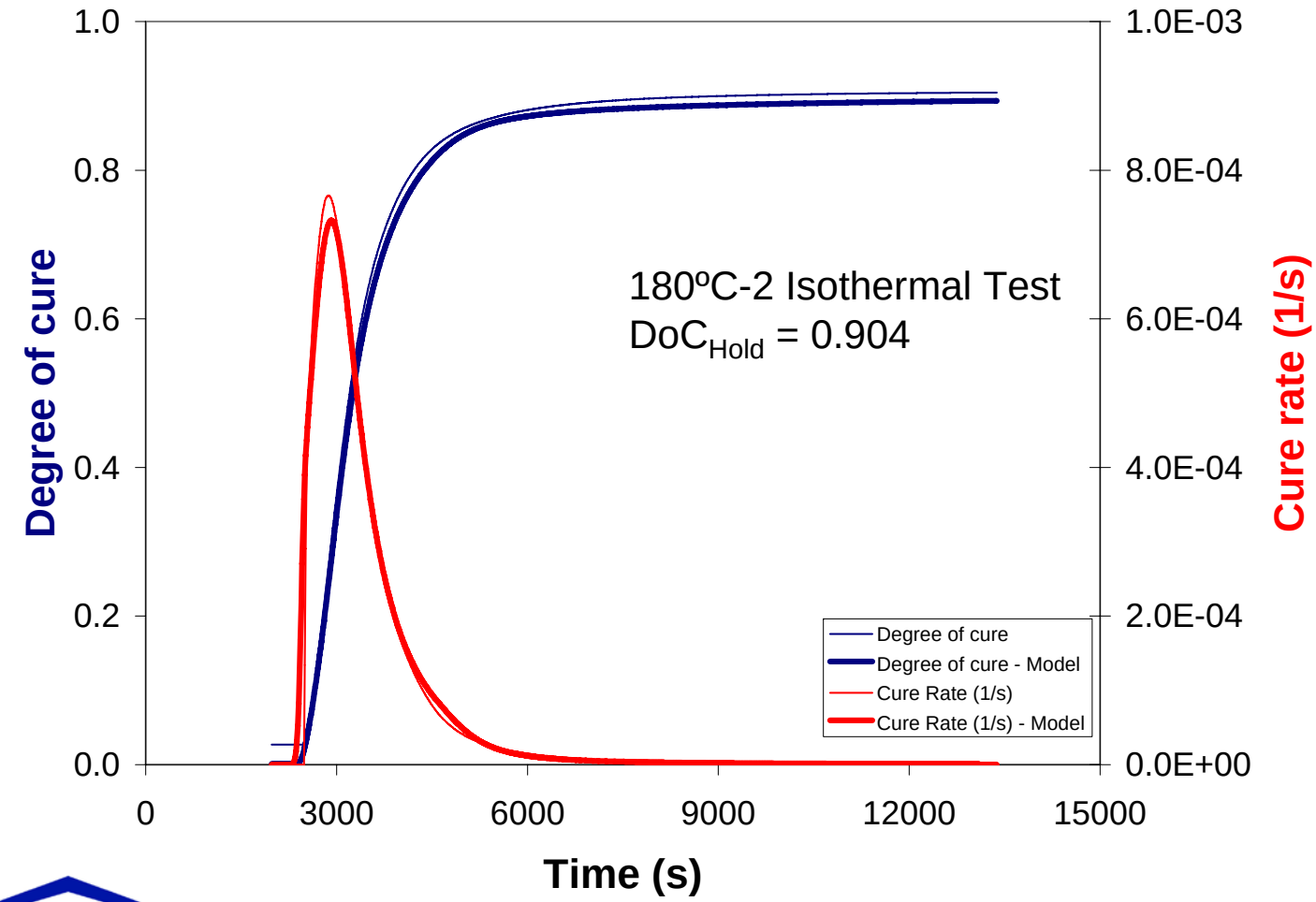
Isothermal Tests - 170ºC



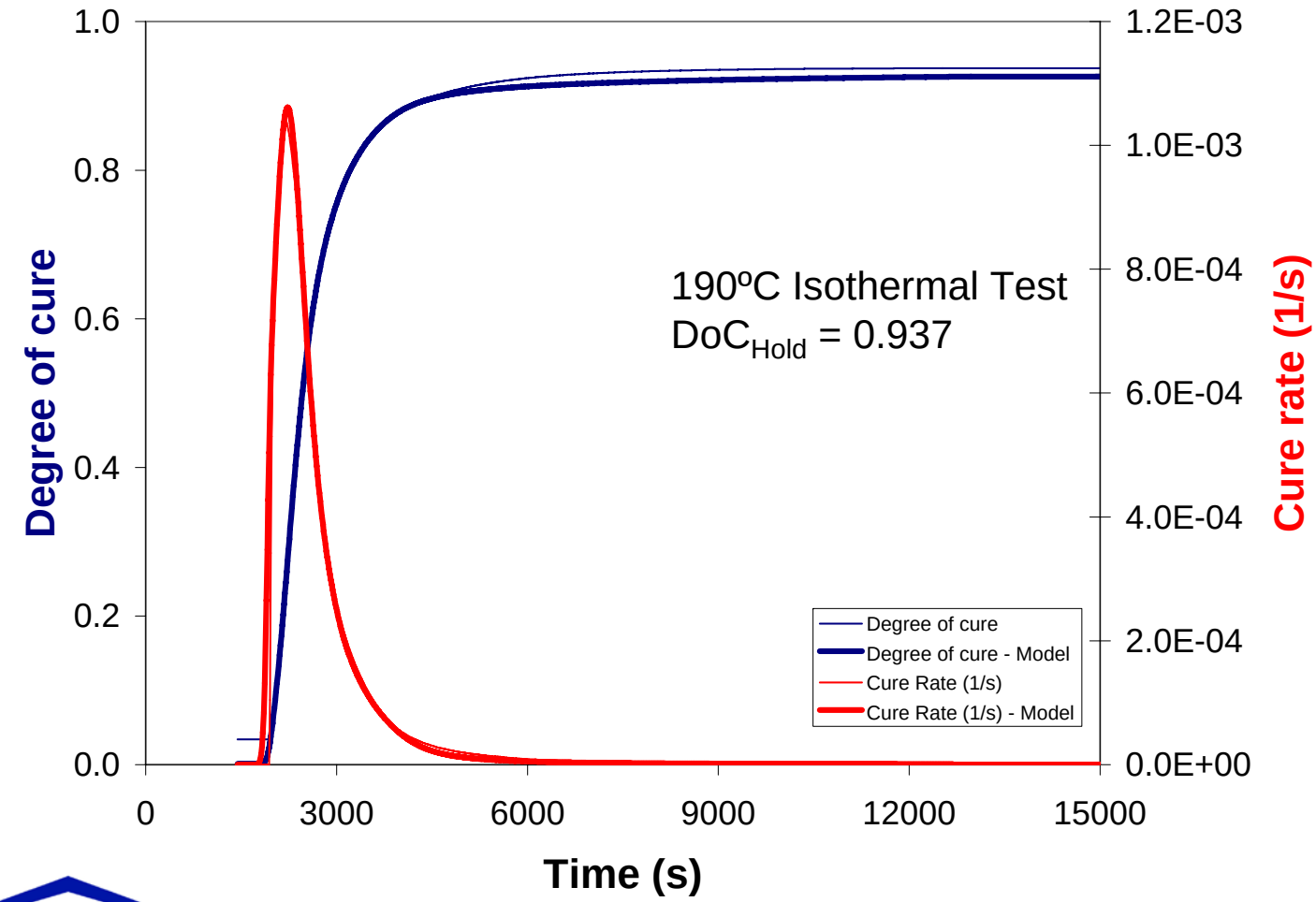
Isothermal Tests - 180ºC-1



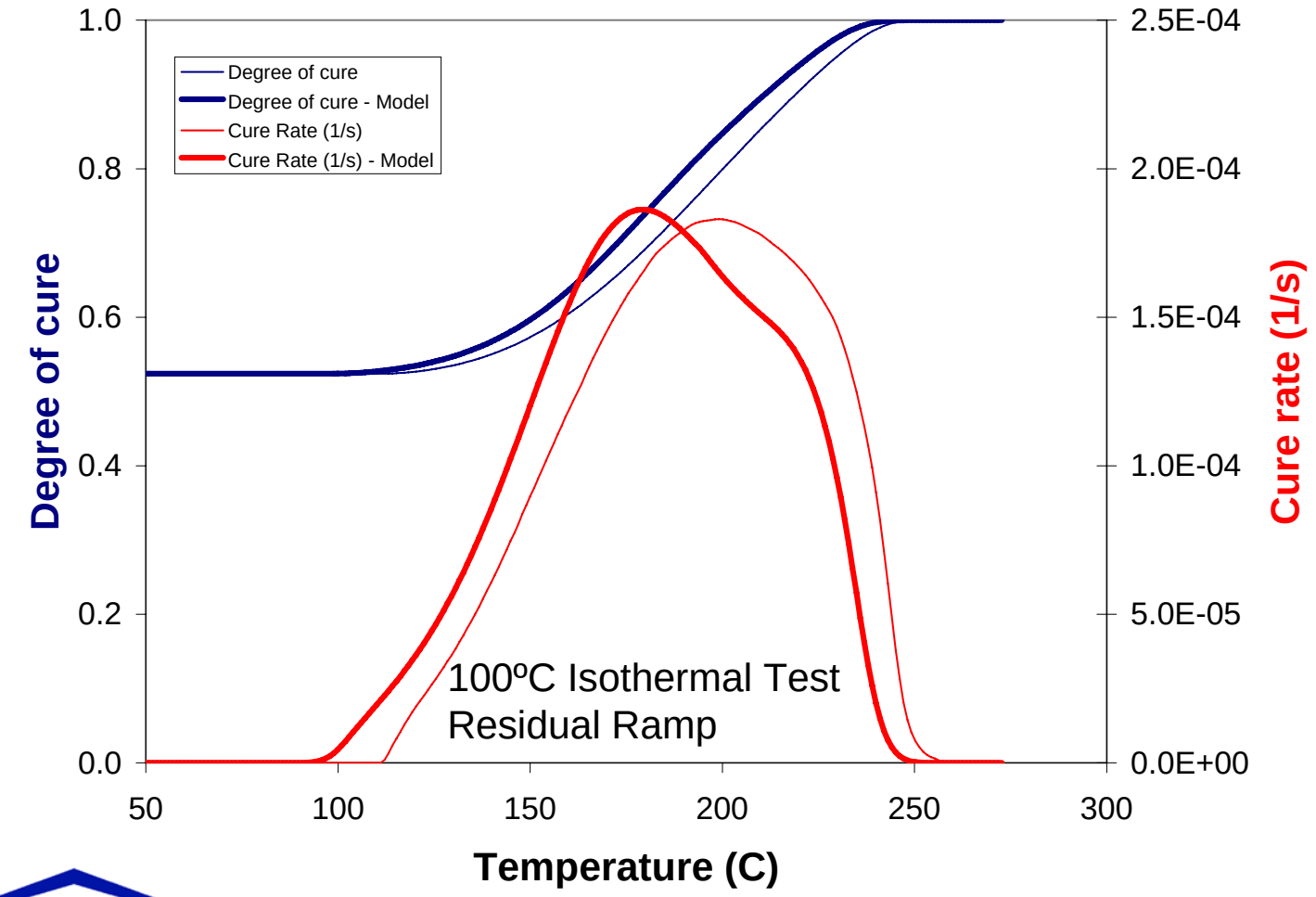
Isothermal Tests - 180ºC-2



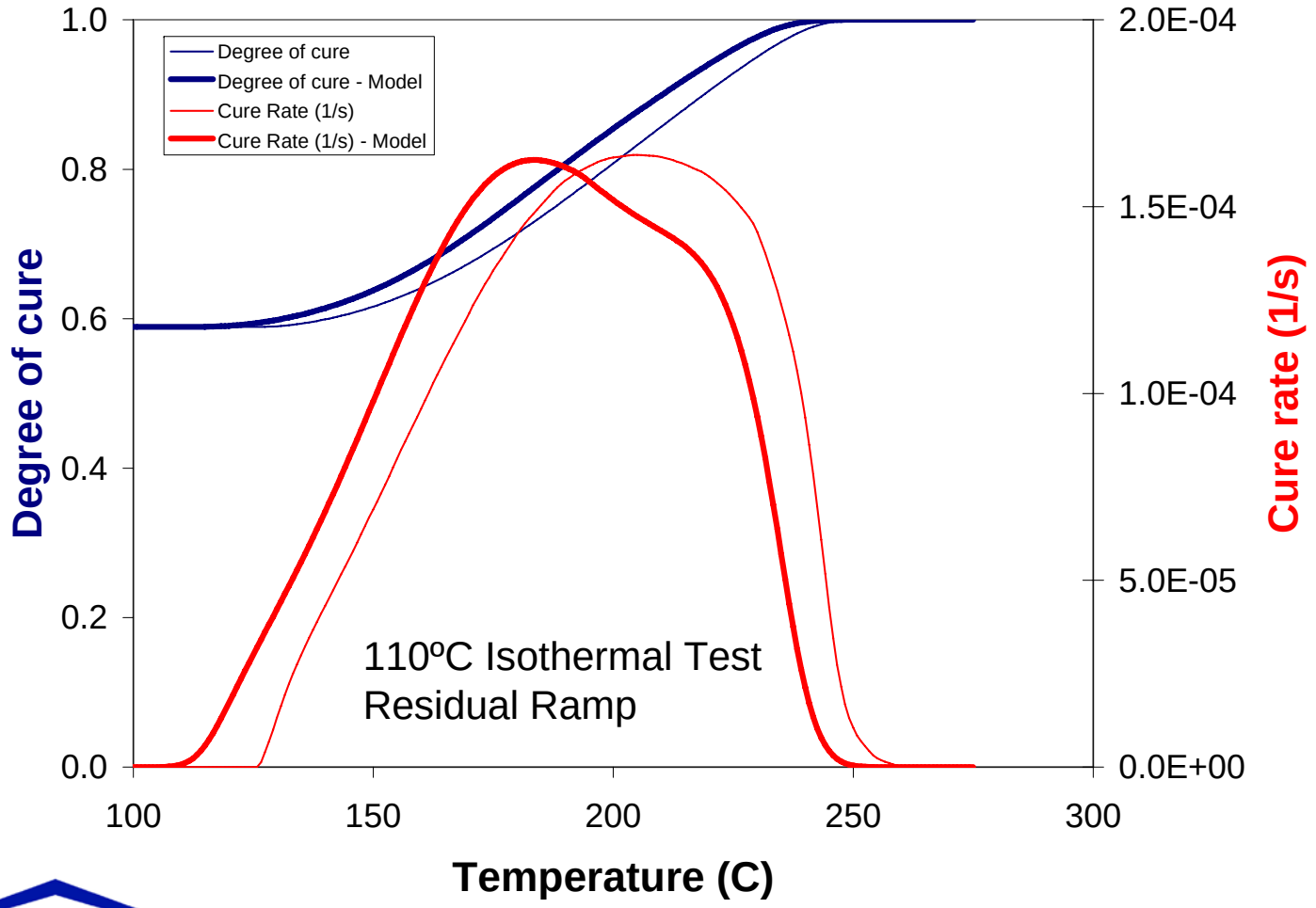
Isothermal Tests - 190ºC



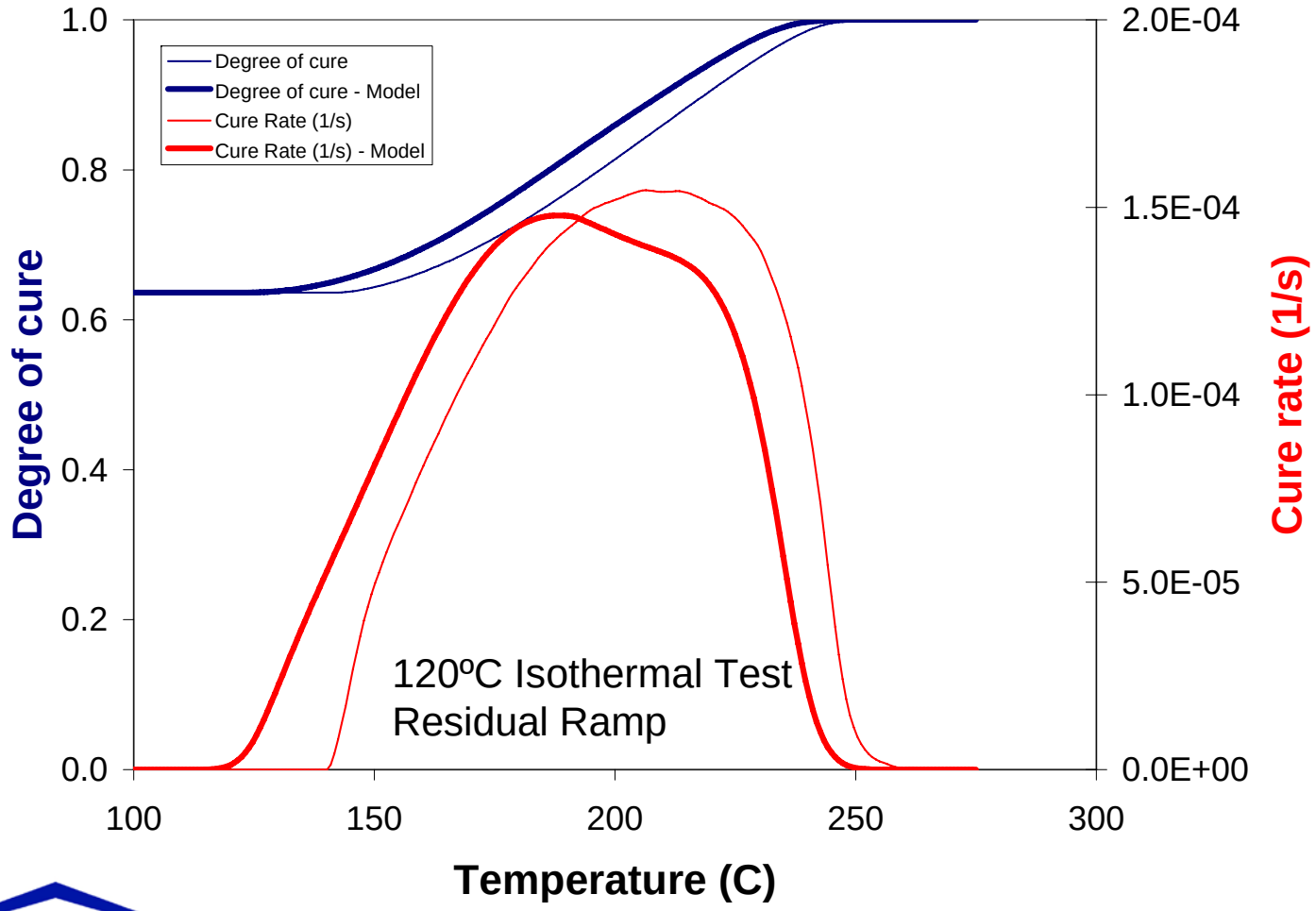
Isothermal Tests - 100ºC



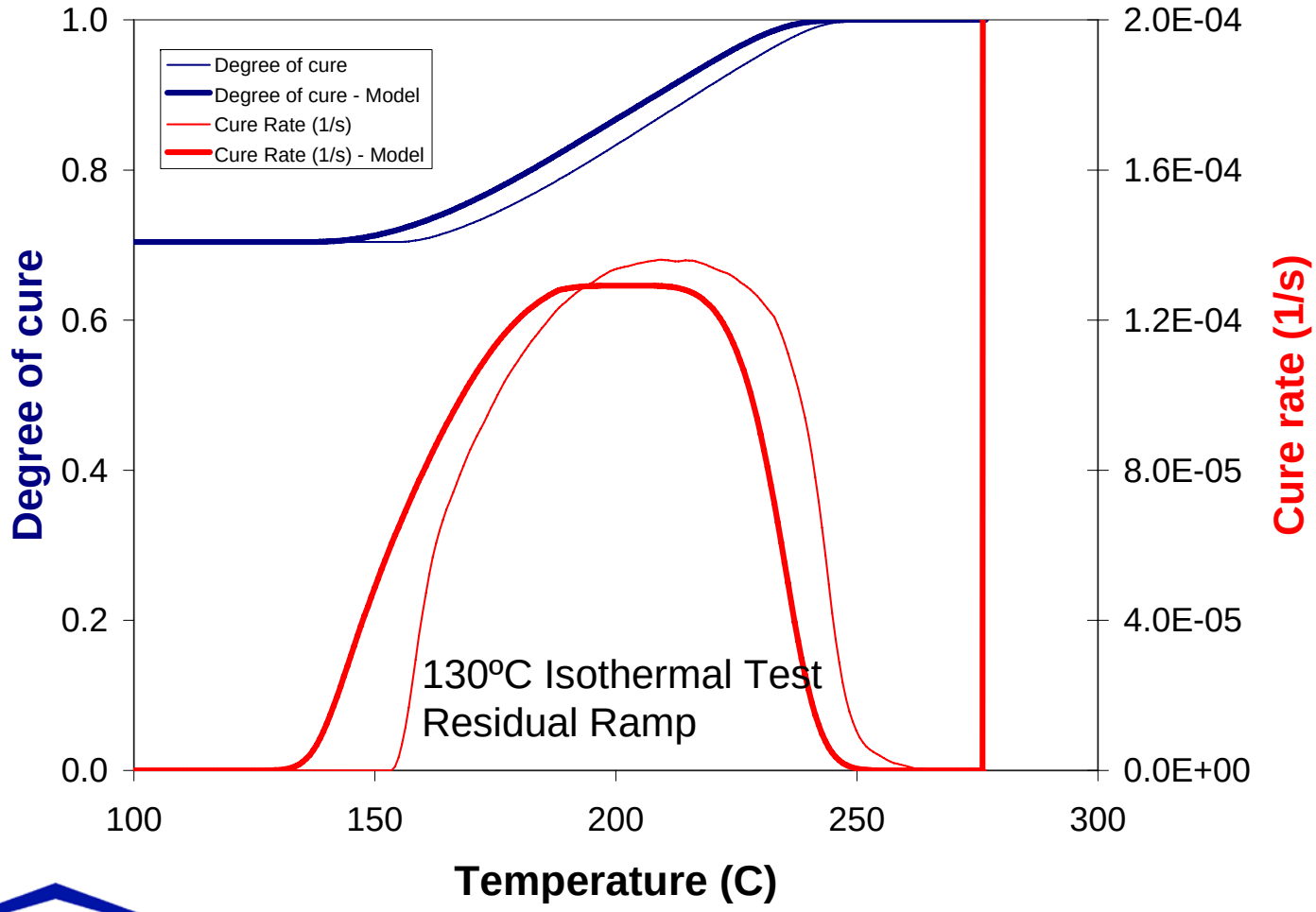
Isothermal Tests - 110ºC



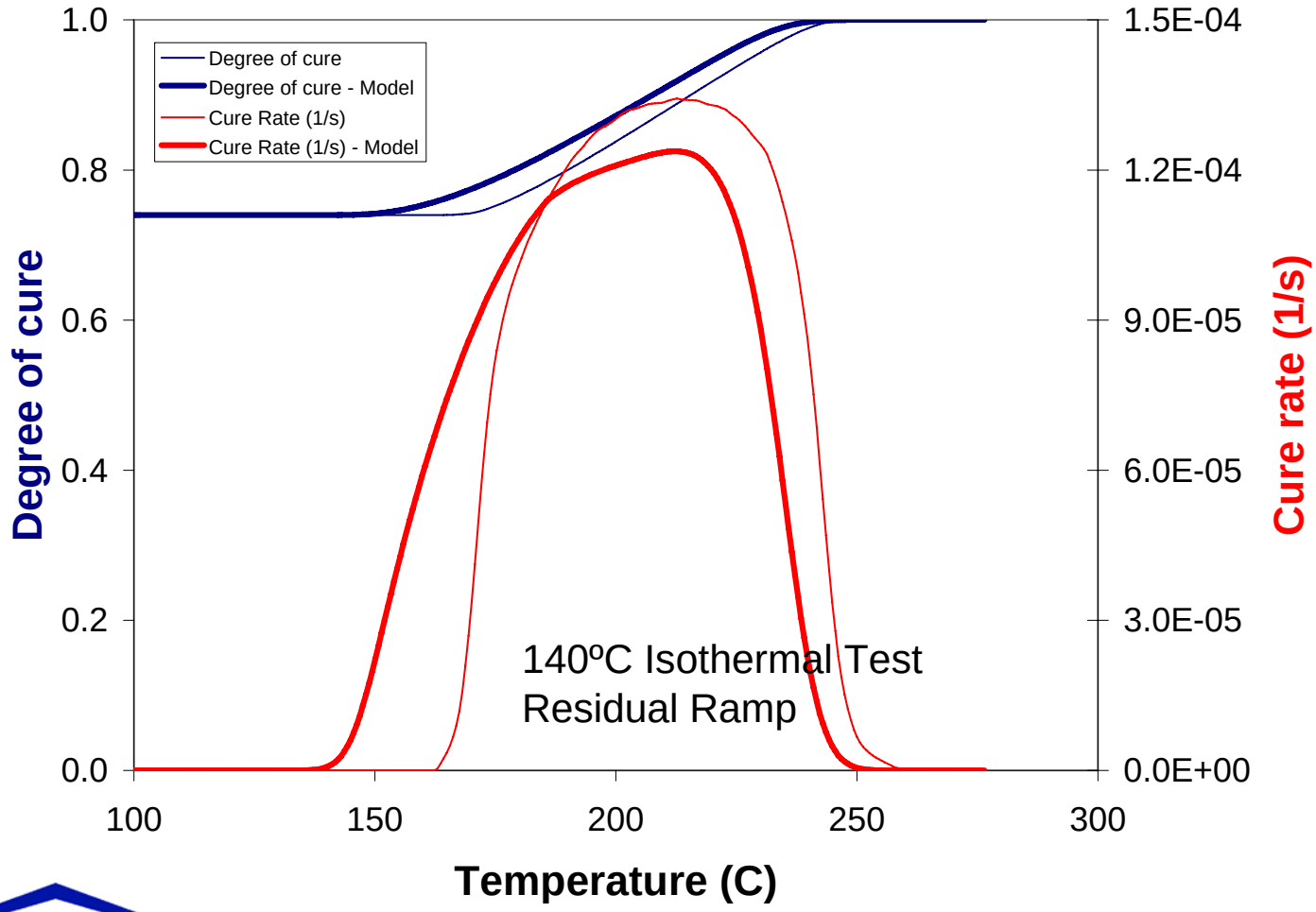
Isothermal Tests - 120ºC



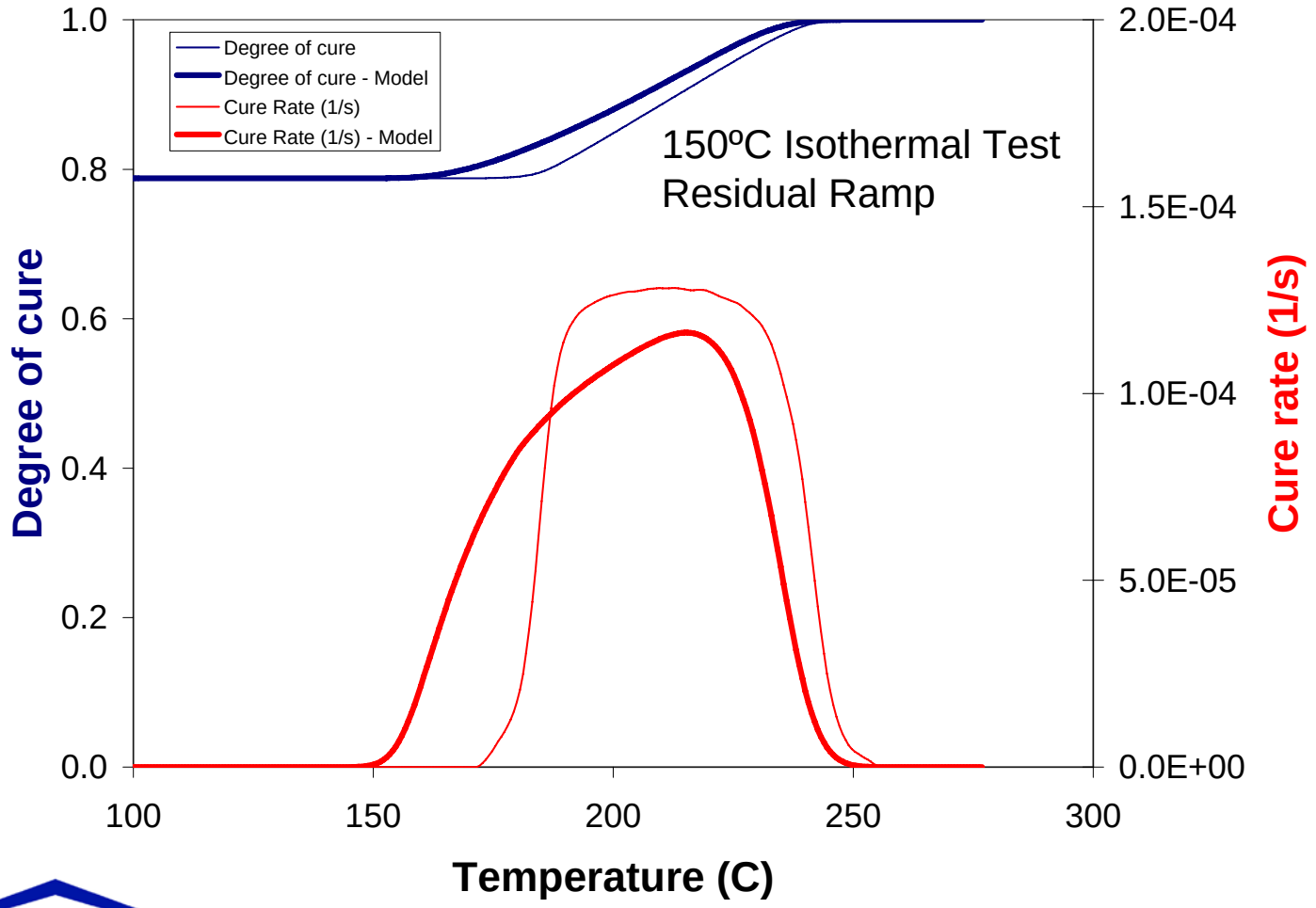
Isothermal Tests - 130ºC



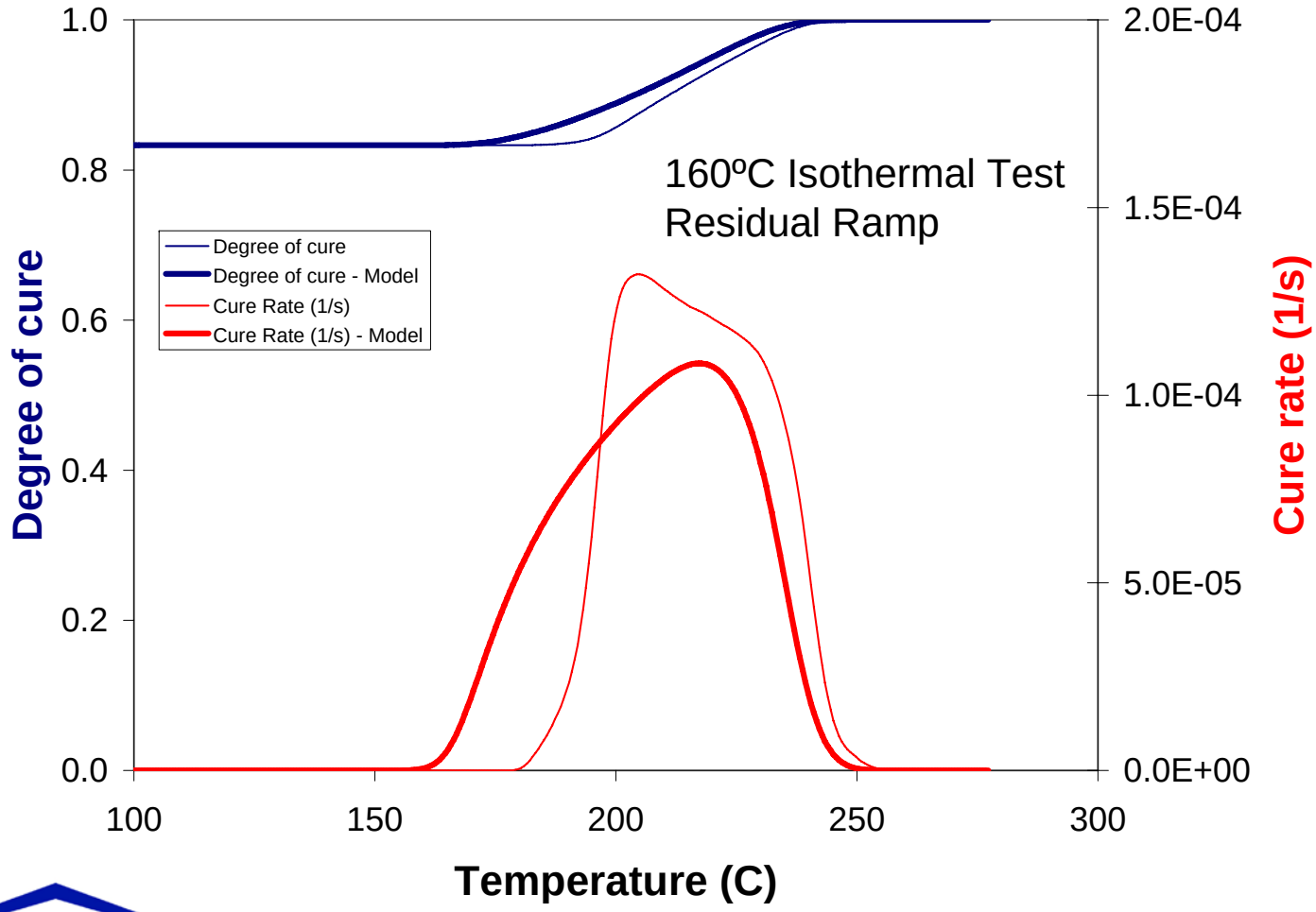
Isothermal Tests - 140ºC



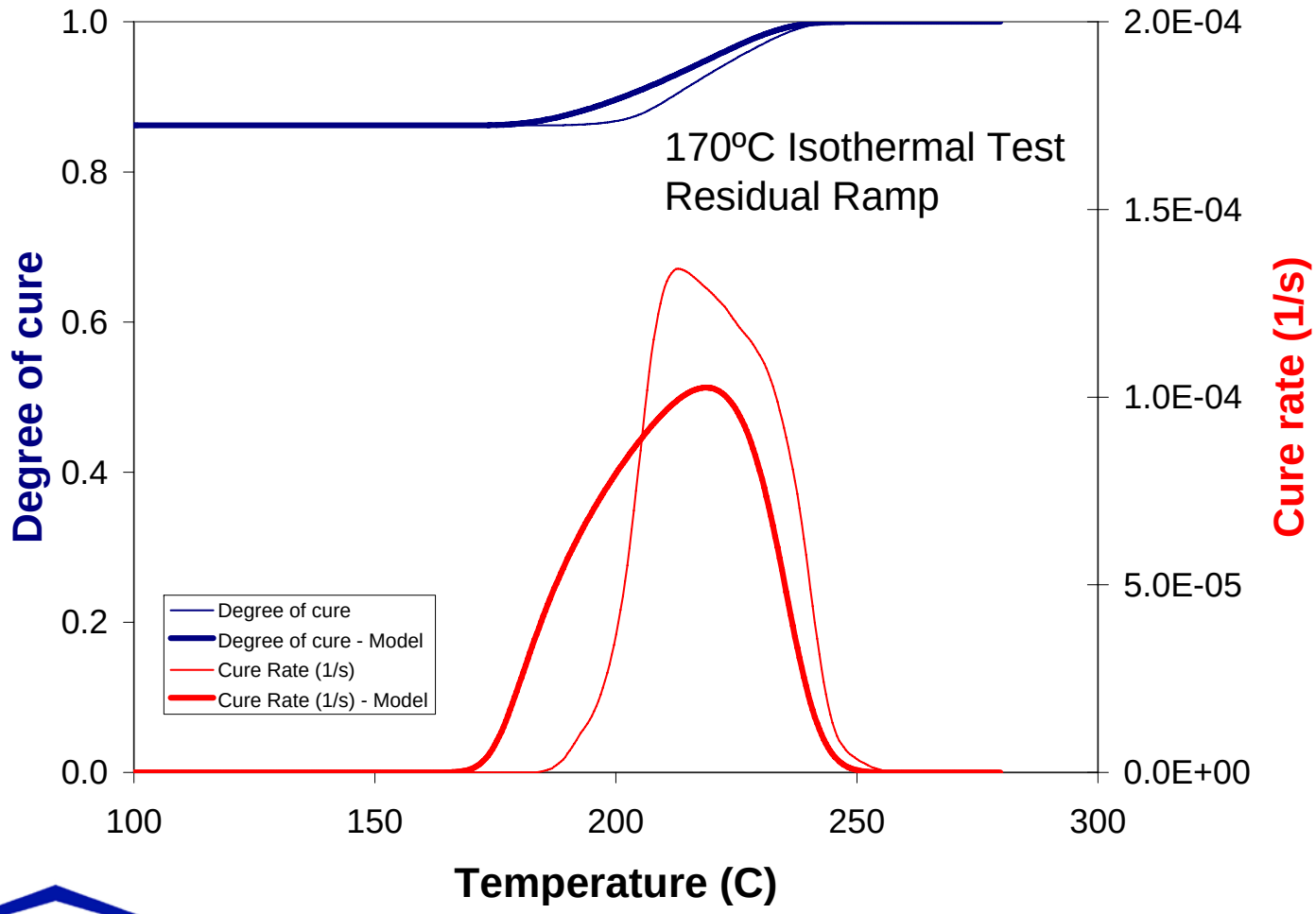
Isothermal Tests - 150ºC



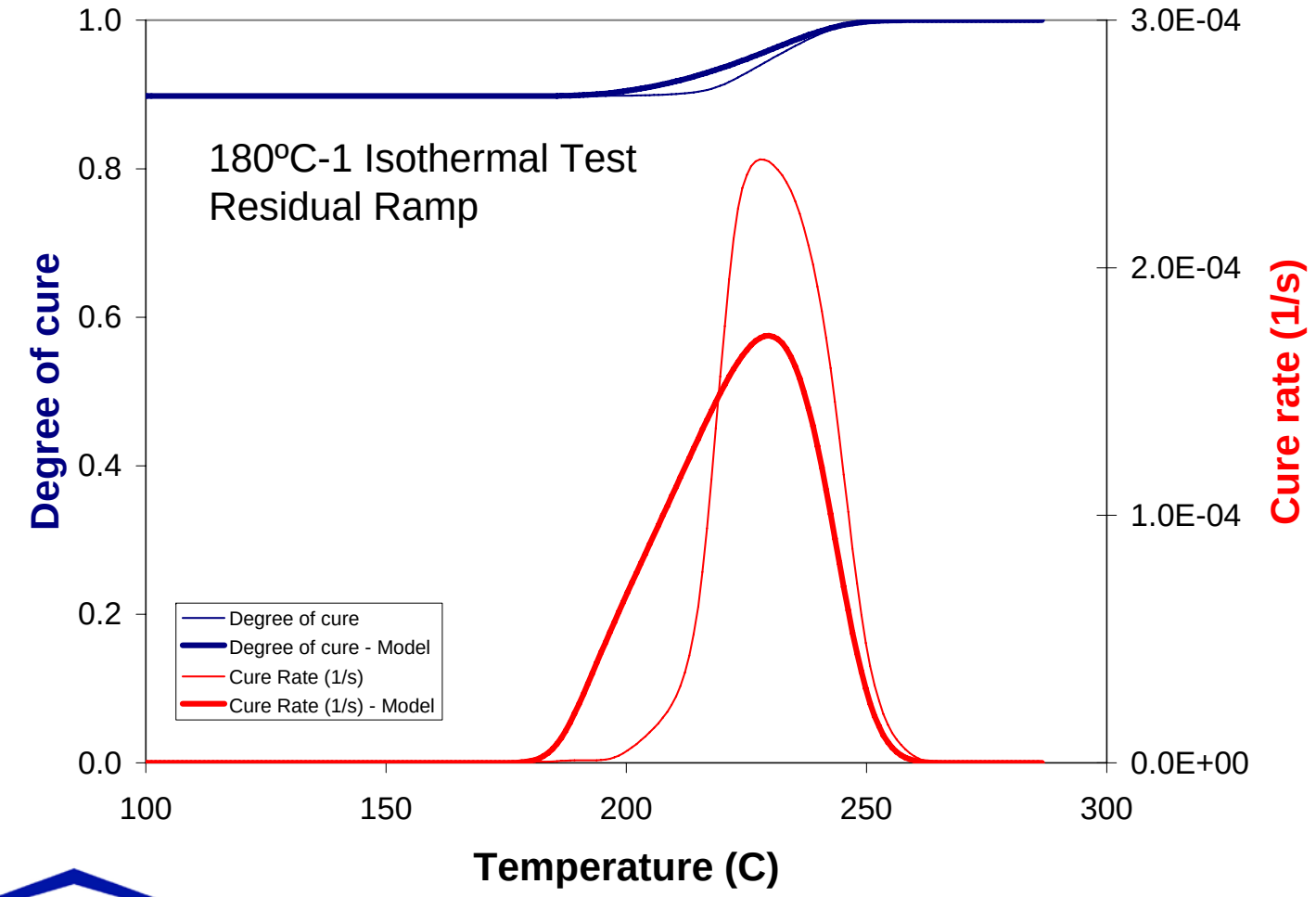
Isothermal Tests - 160ºC



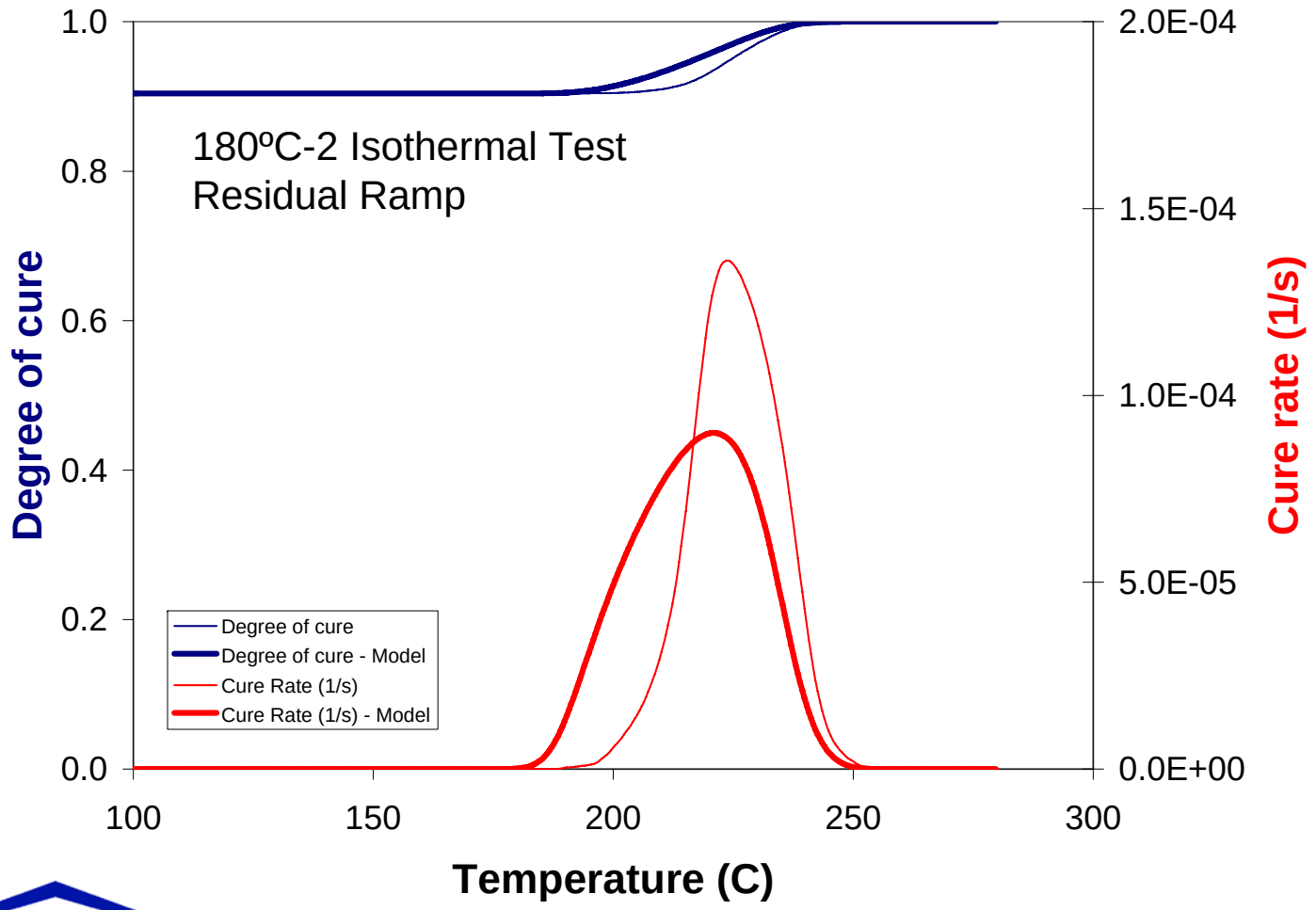
Isothermal Tests - 170ºC



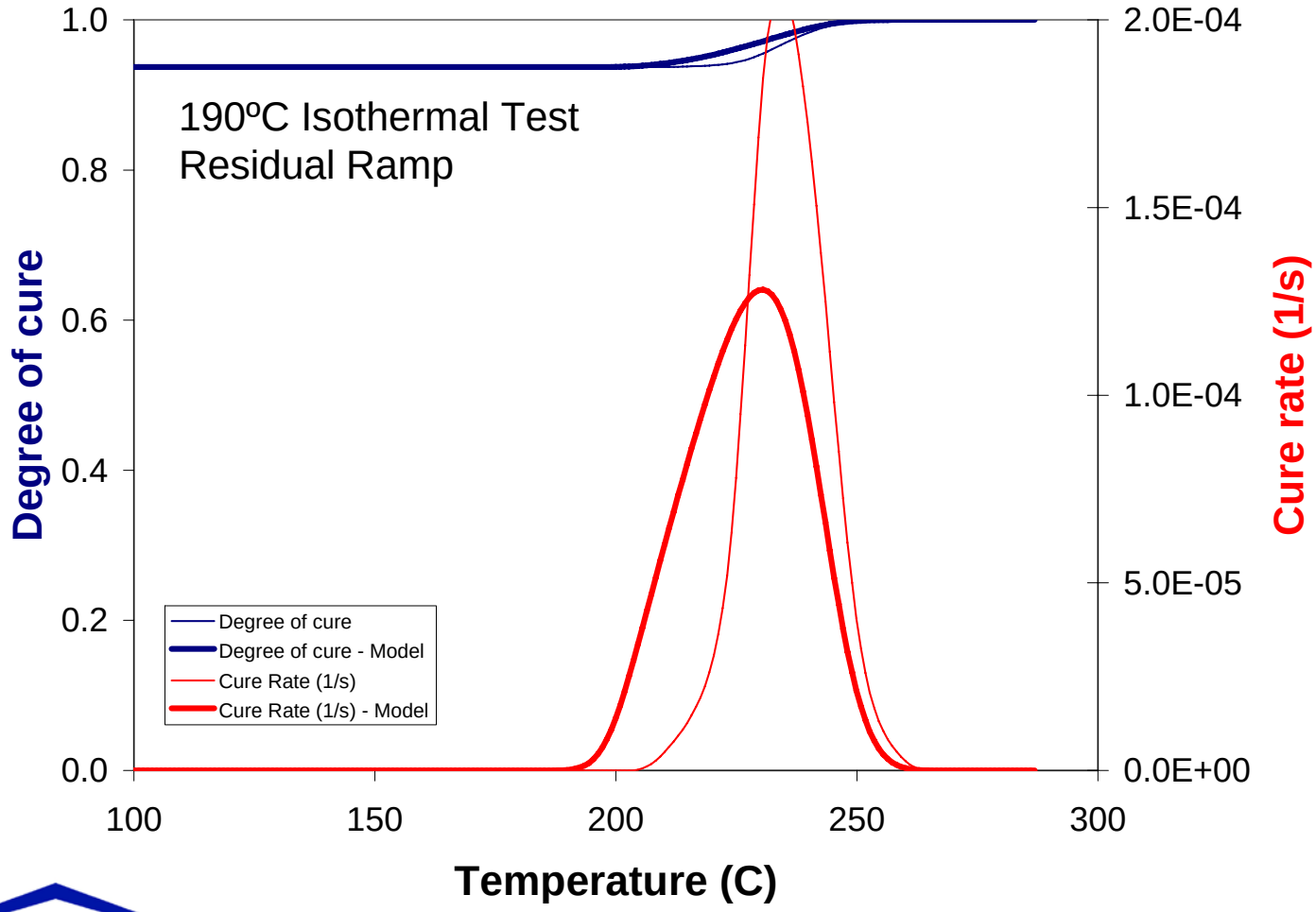
Isothermal Tests - 180ºC-1



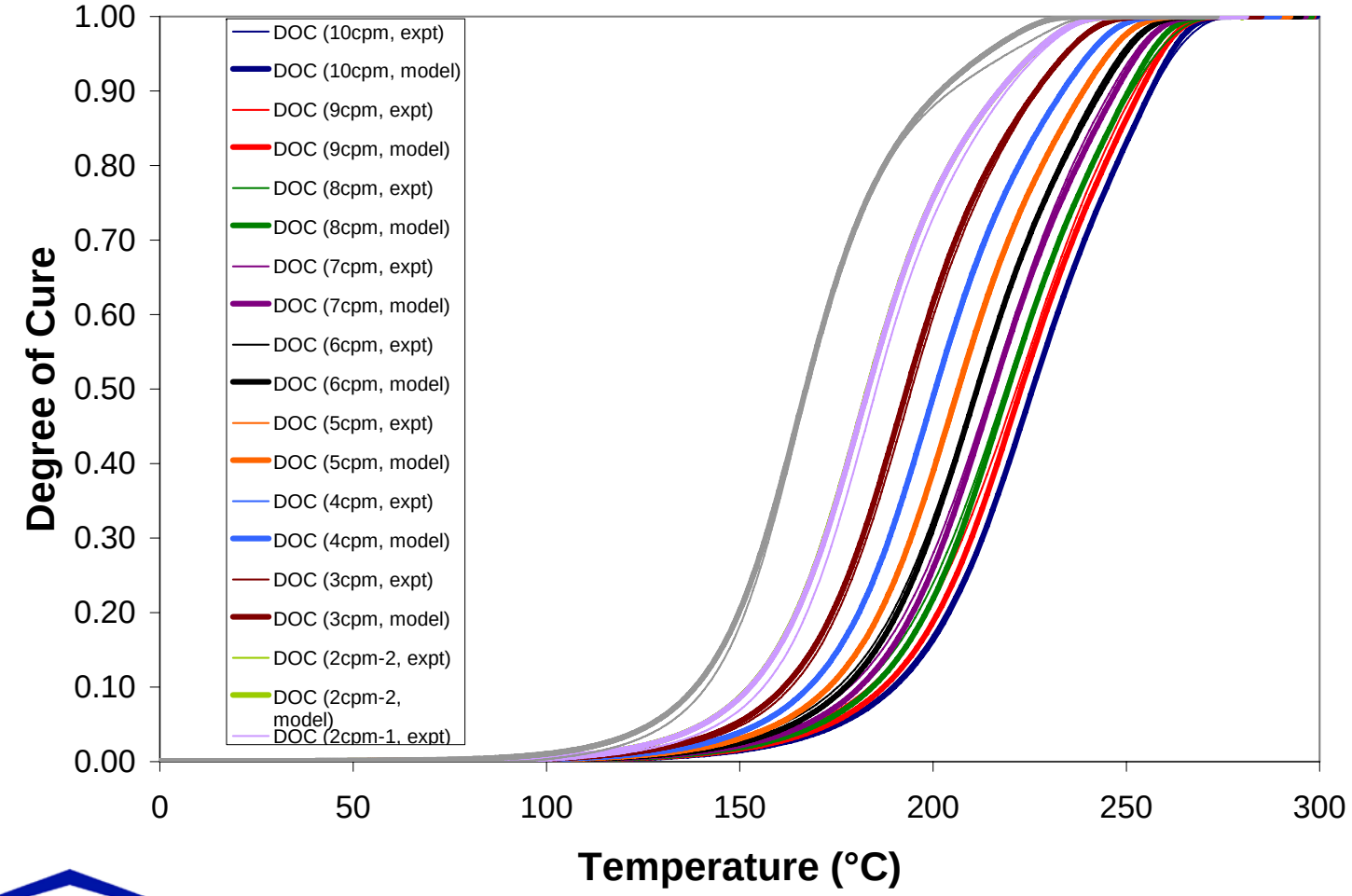
Isothermal Tests - 180ºC-2



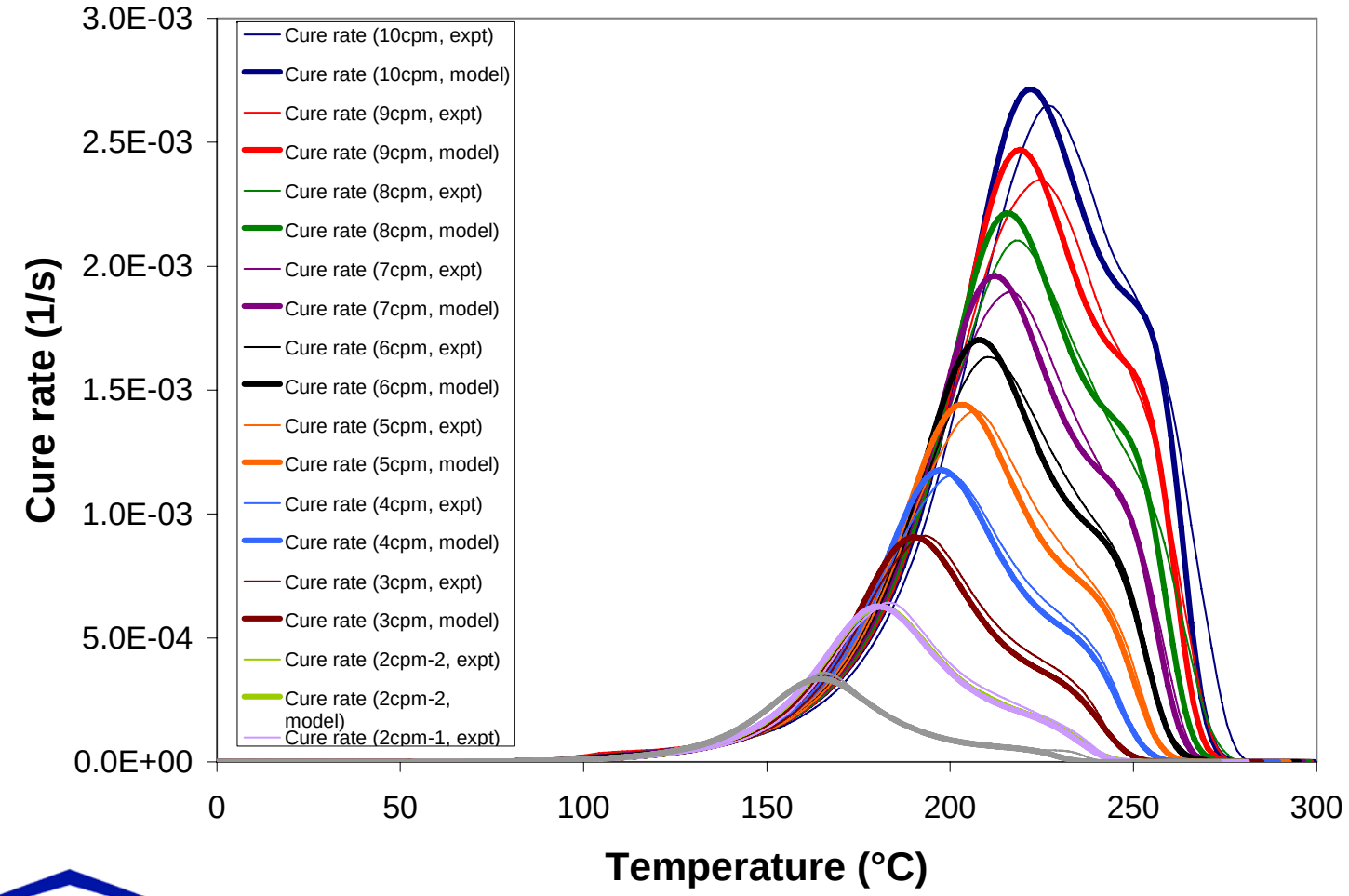
Isothermal Tests - 190ºC



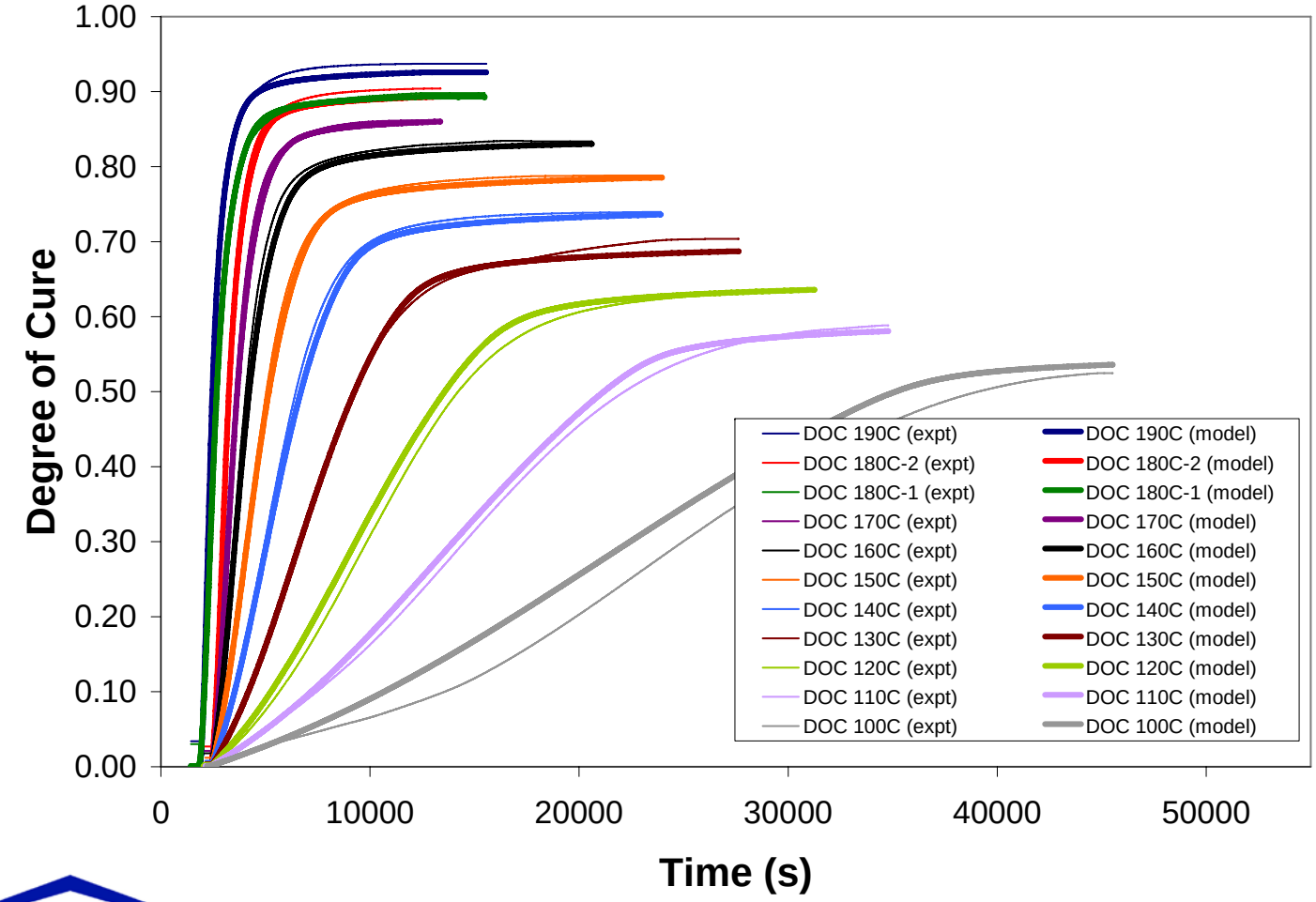
Dynamic Tests – All



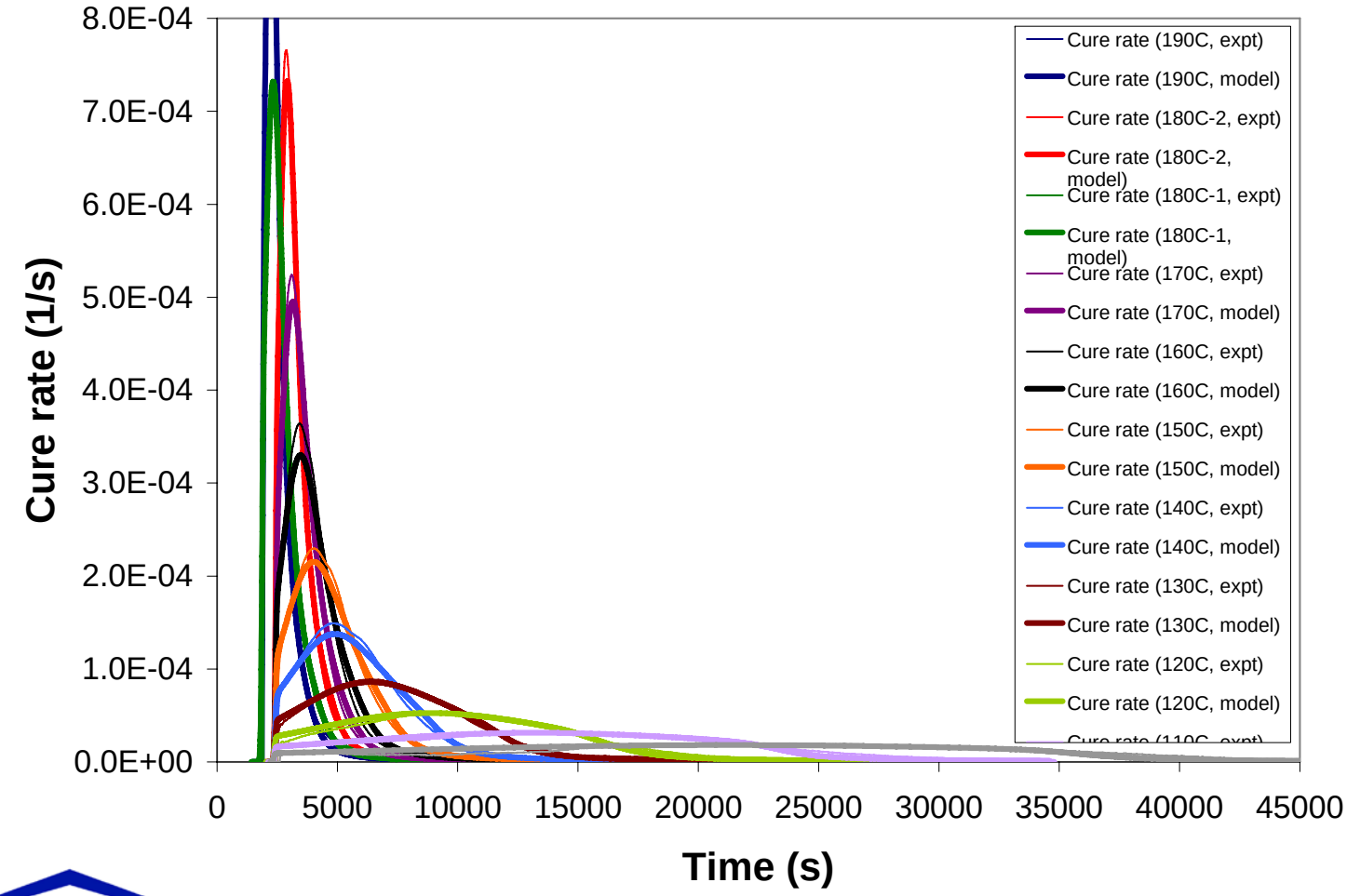
Dynamic Tests – All



Isothermal Tests – All

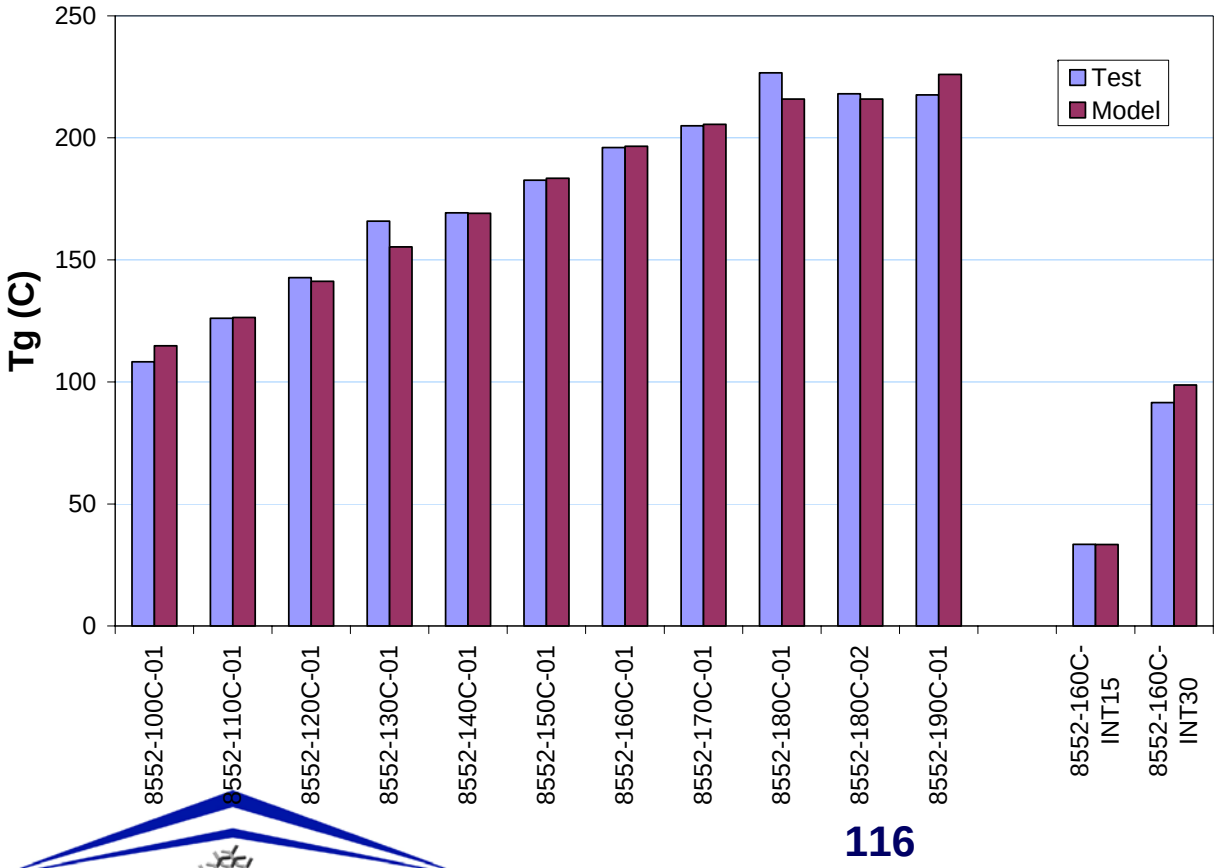


Isothermal Tests – All



Post-Hold Tg

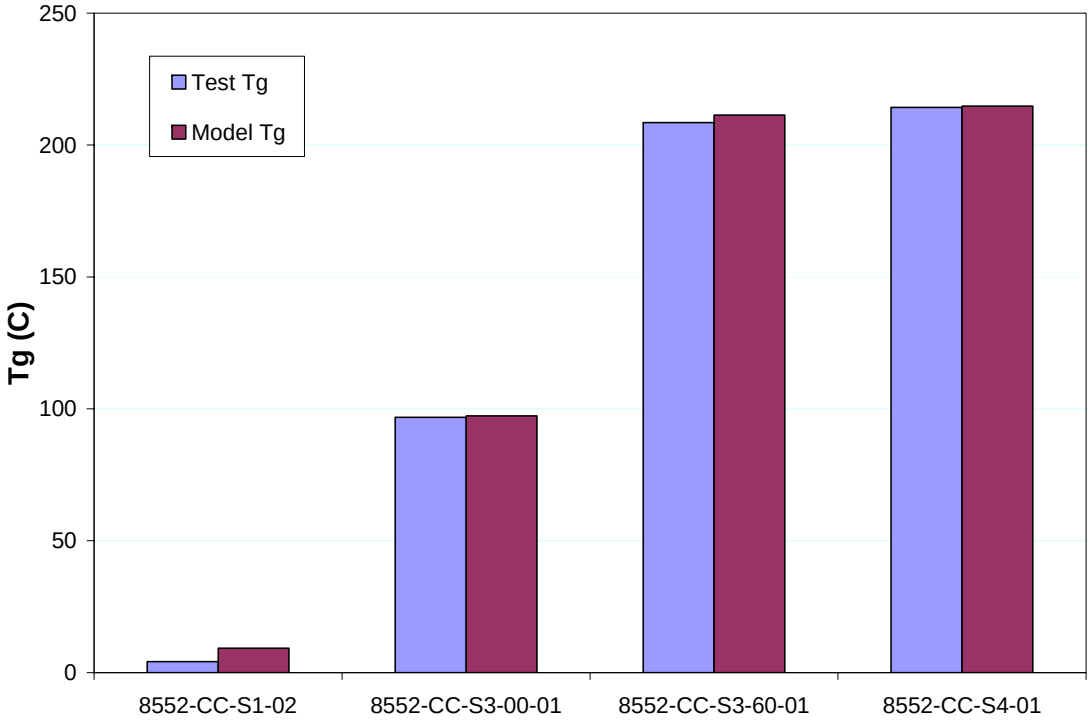
Tg values at the end of the hold segment of the isothermal tests were compared to the predictions of the cure kinetics model. Good agreement was observed.

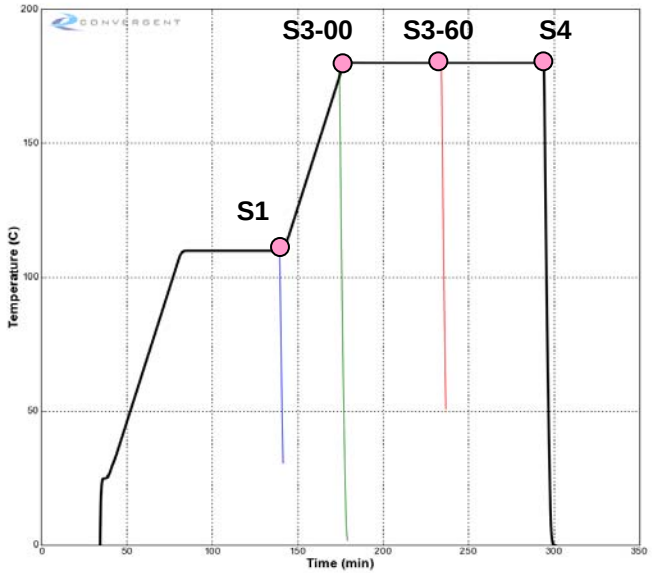


Test	Post-Hold Tg (°C)	
	Test	Model
8552-MISO-100C-01	108.2	114.8
8552-MISO-110C-01	126.1	126.4
8552-MISO-120C-01	142.7	141.2
8552-MISO-130C-01	165.9	155.3
8552-MISO-140C-01	169.3	169.1
8552-MISO-150C-01	182.7	183.4
8552-MISO-160C-01	196.0	196.6
8552-MISO-170C-01	204.9	205.6
8552-MISO-180C-01	226.7	215.9
8552-MISO-180C-02	218.1	215.9
8552-MISO-190C-01	217.6	226.0
8552-160C-INT15	33.5	33.4
8552-160C-INT30	91.5	98.8

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.



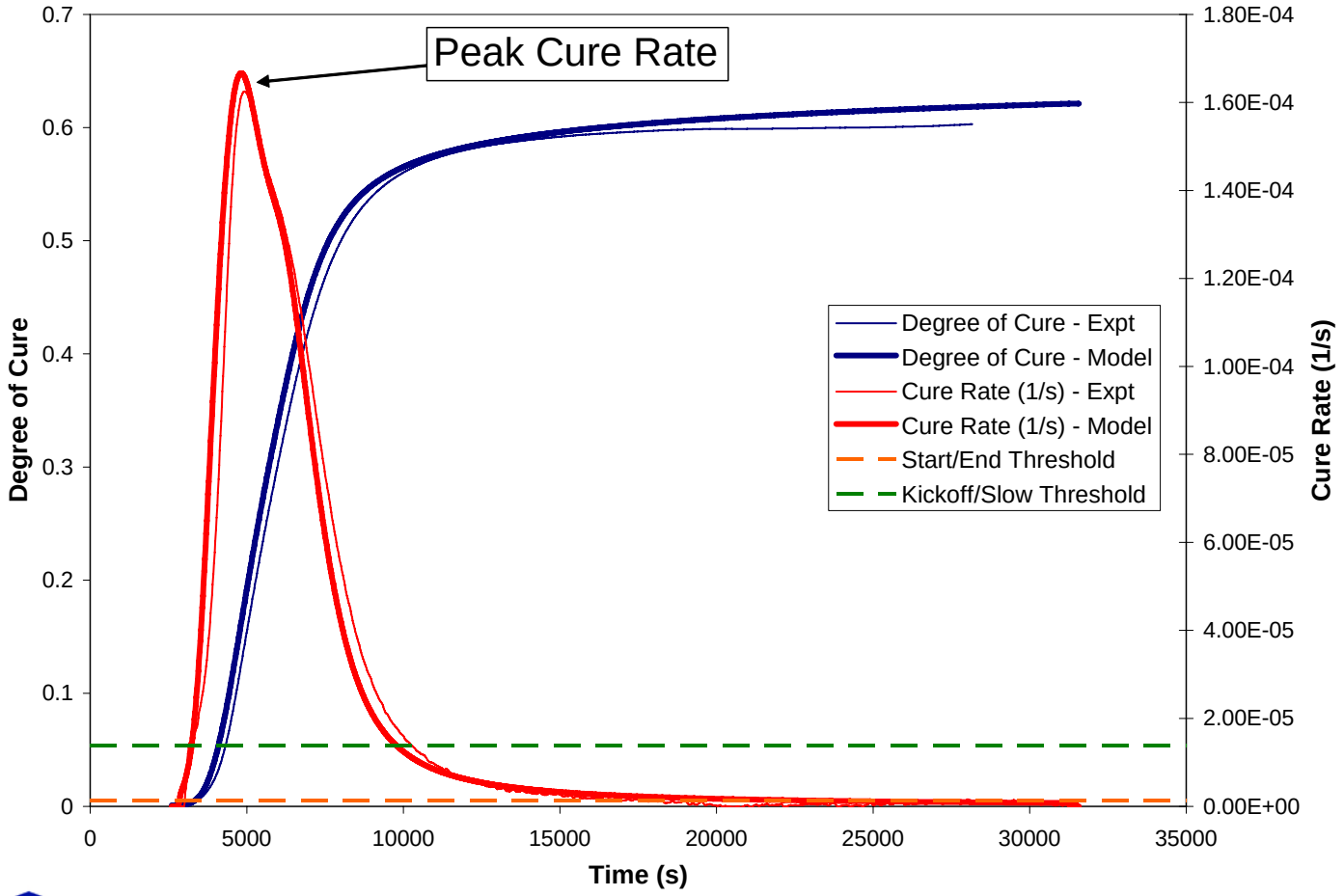


Test	Tg (ºC)	
	Test	Model
8552-CC-S1-02	4.2	9.3
8552-CC-S3-00-01	96.8	97.4
8552-CC-S3-60-01	208.5	211.4
8552-CC-S4-01	214.3	214.8

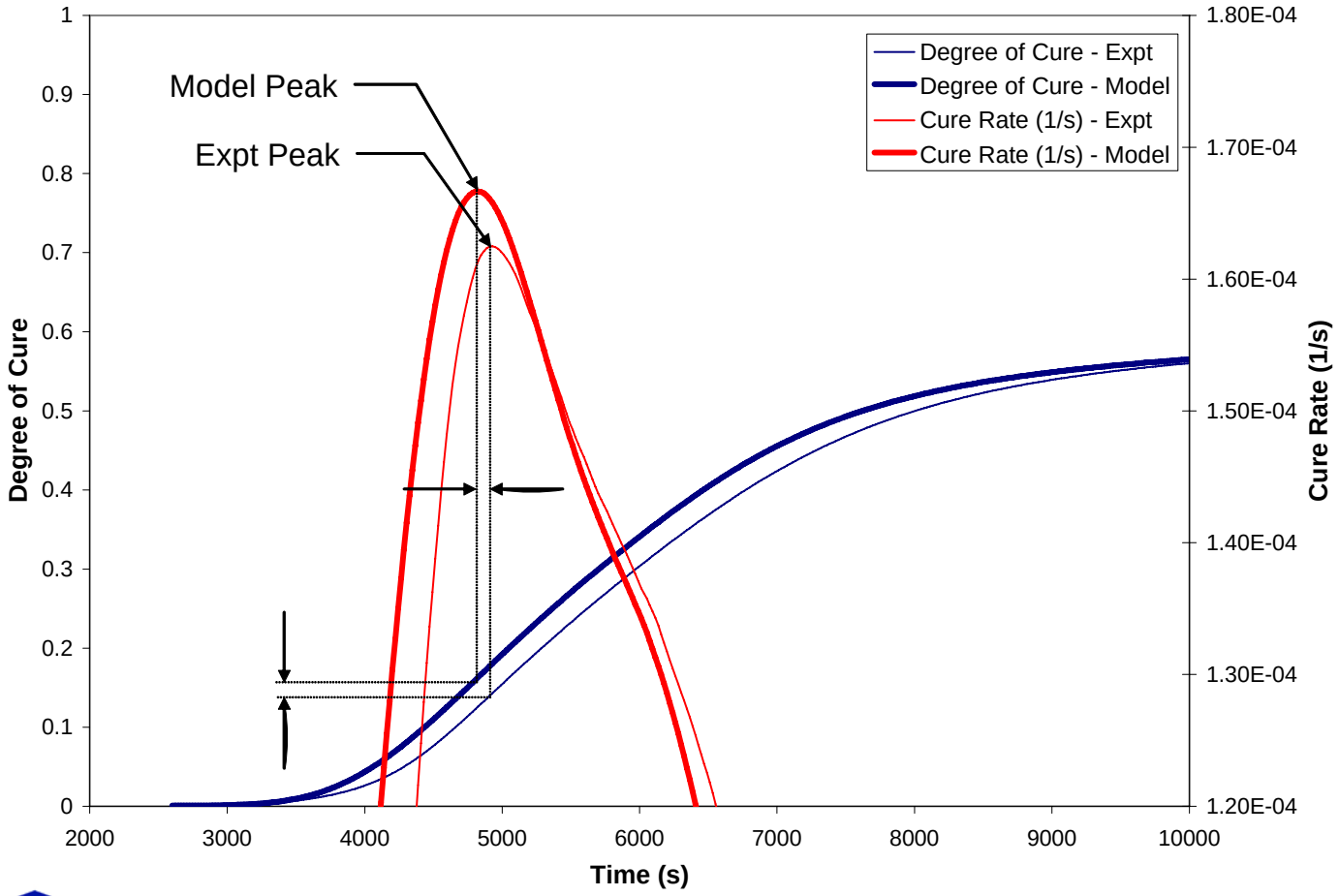
Goodness of Fit - Overview

- Goodness of fit is measured by comparing the test to the model prediction at several key points in the cure history.
- Both timing and DOC error are considered.

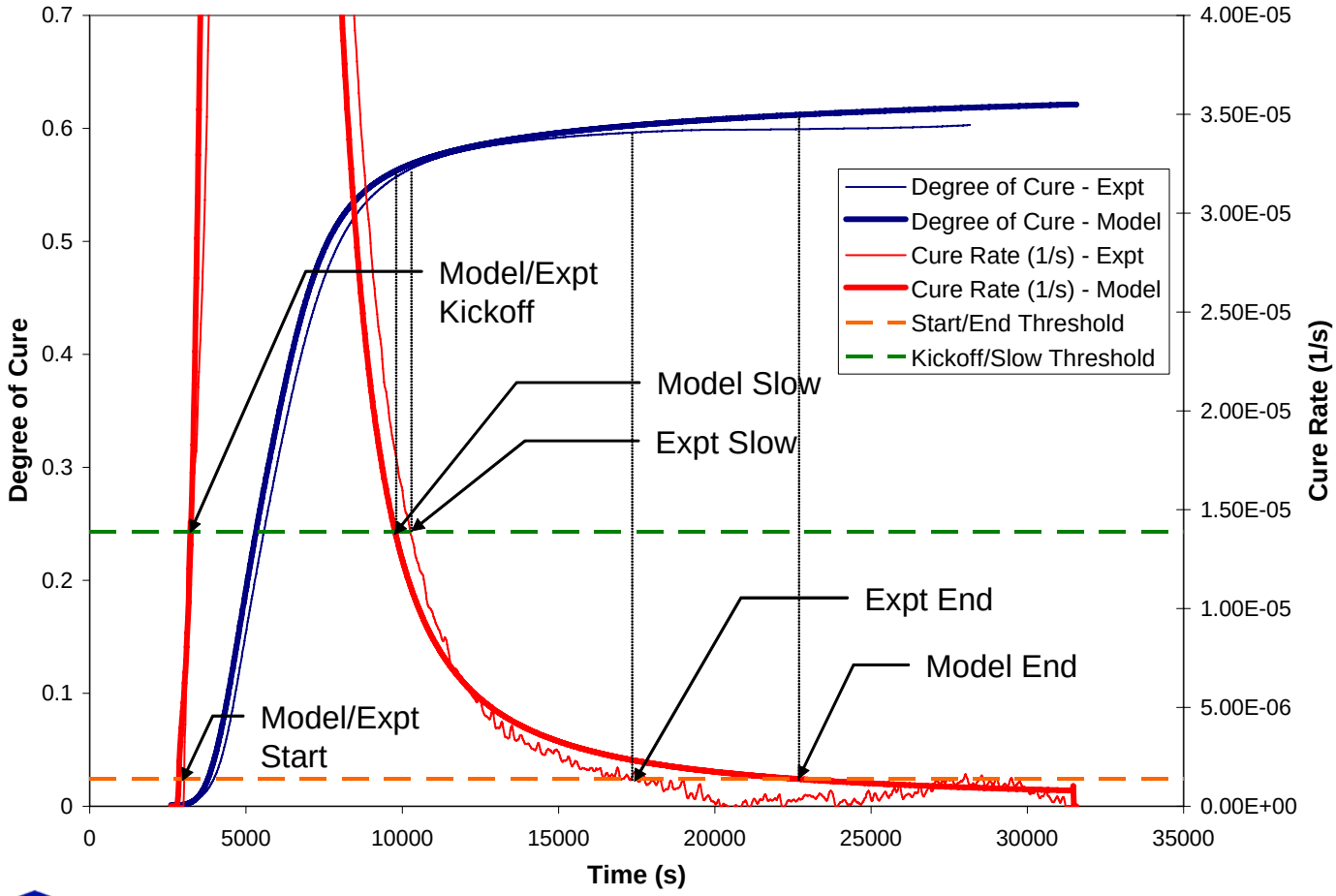
Goodness of Fit – Key Points



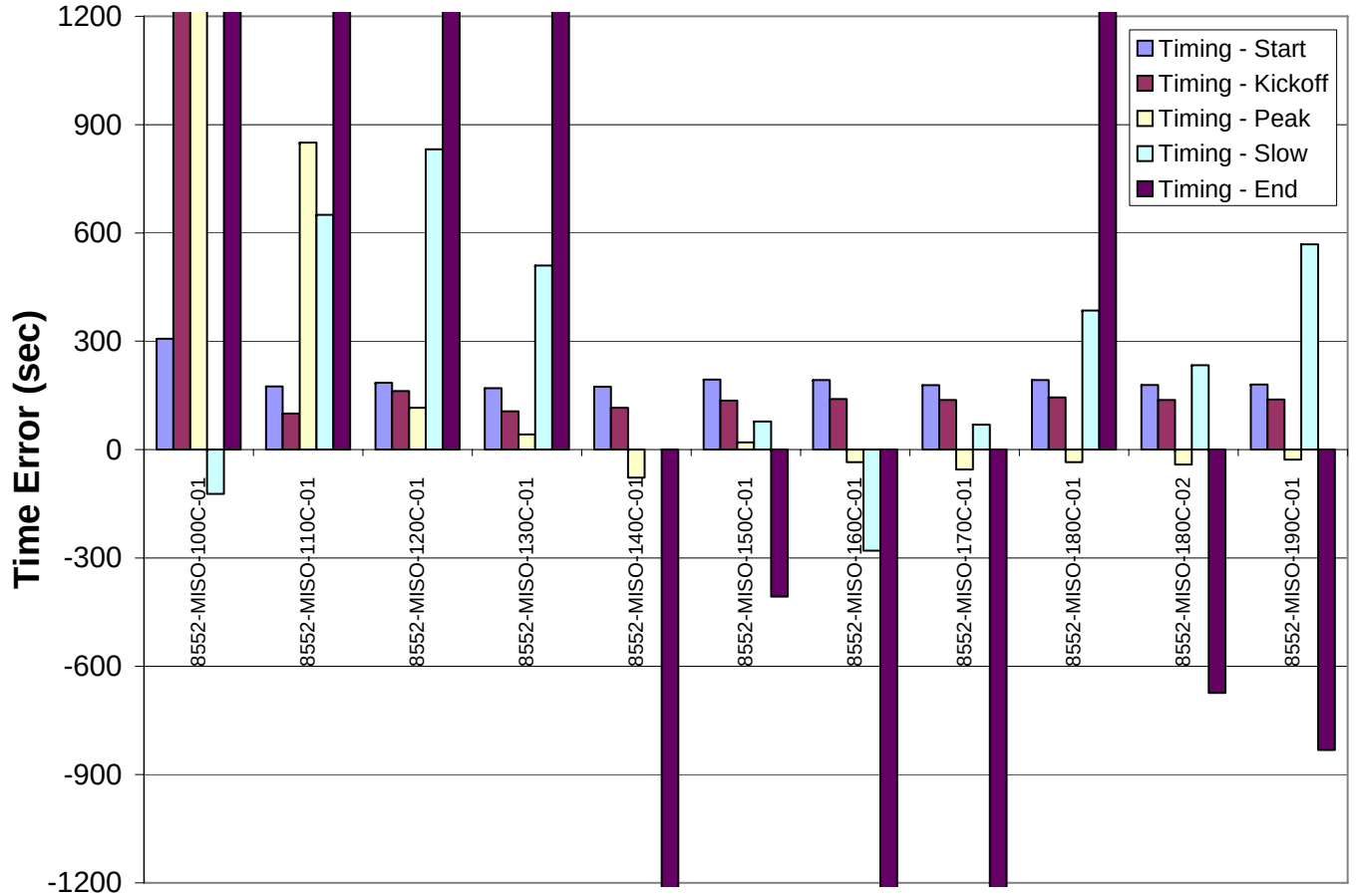
Goodness of Fit – Peak



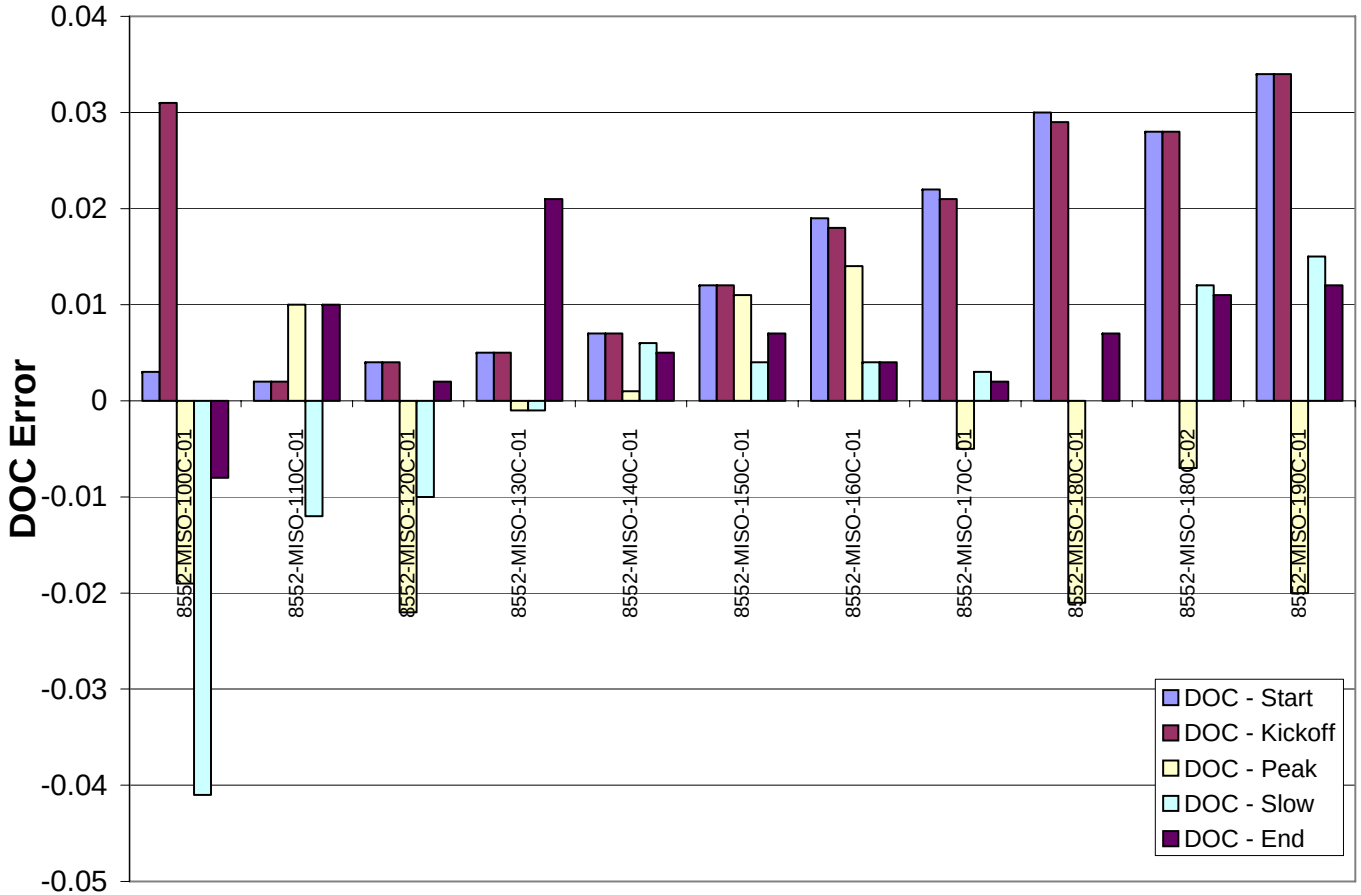
Goodness of Fit – Other



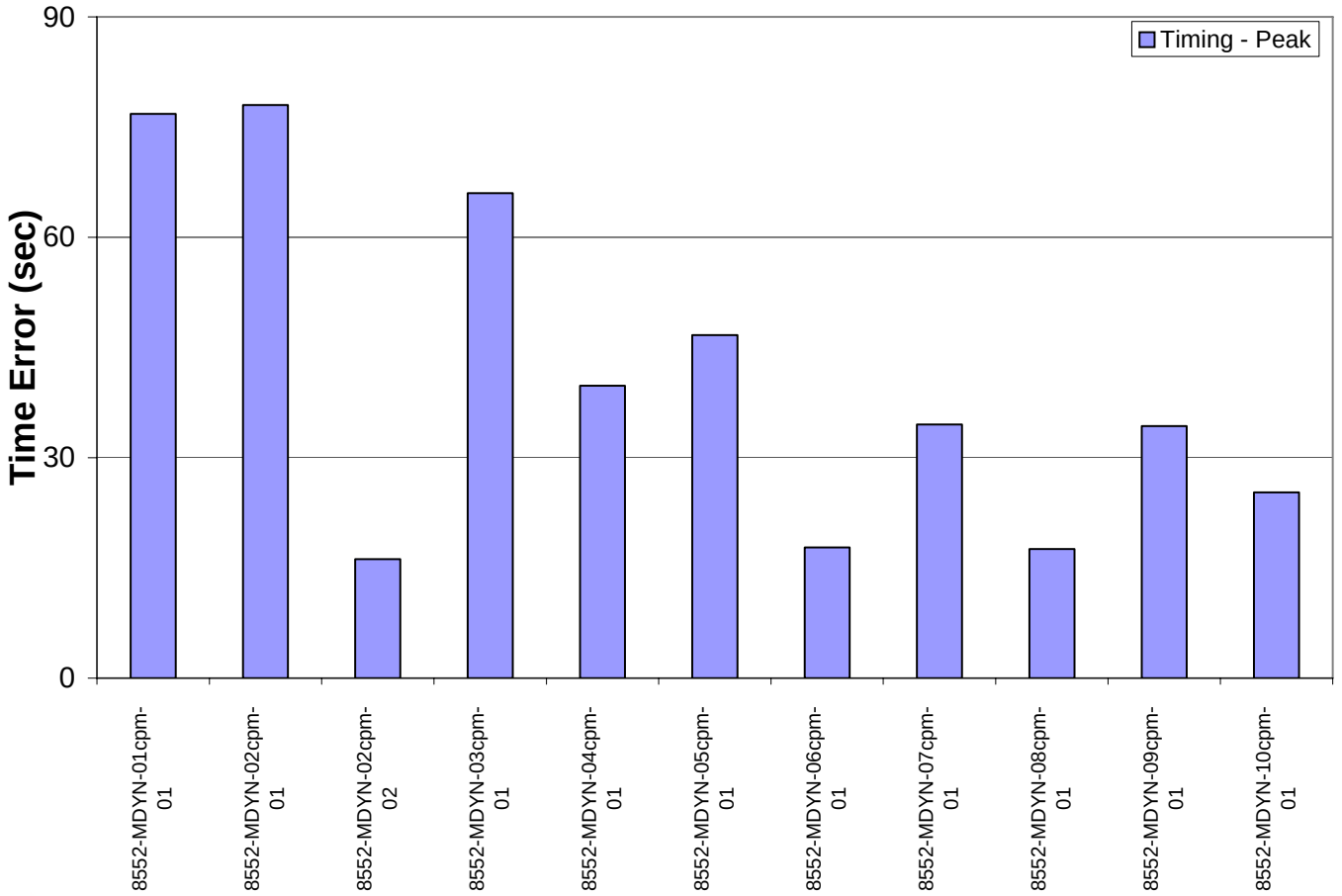
Goodness of Fit – Iso Timing



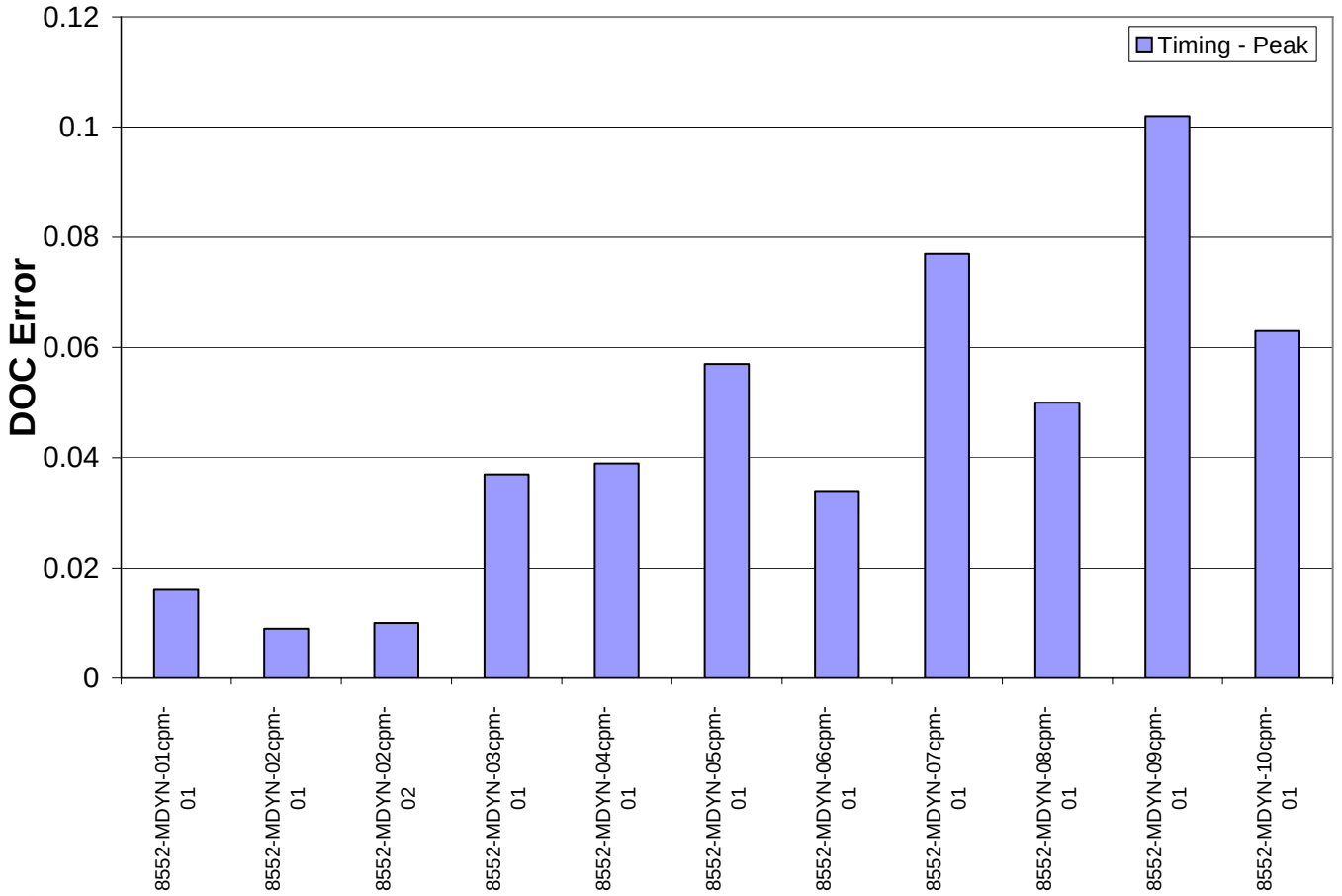
Goodness of Fit – Iso DOC



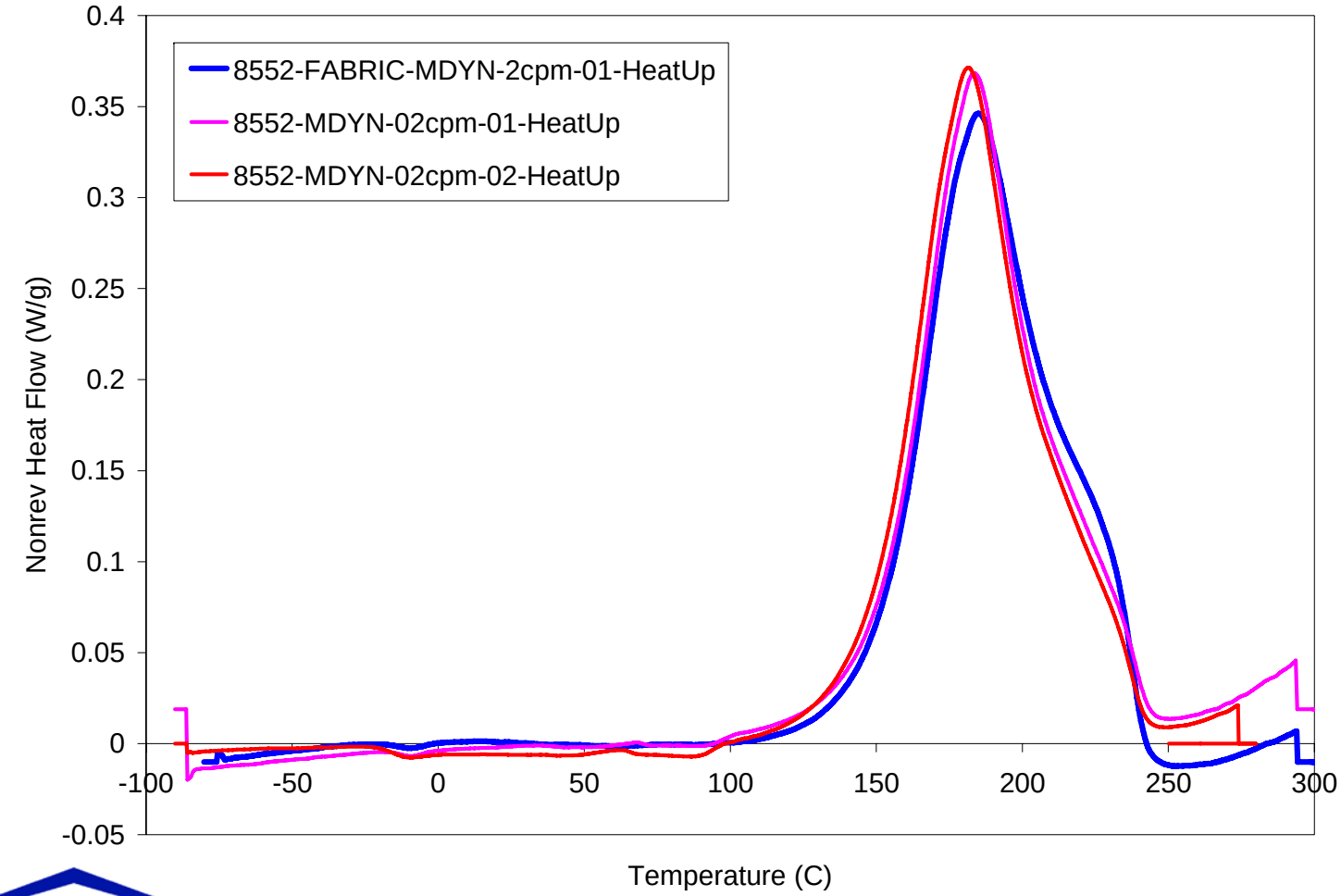
Goodness of Fit – Dyn Timing



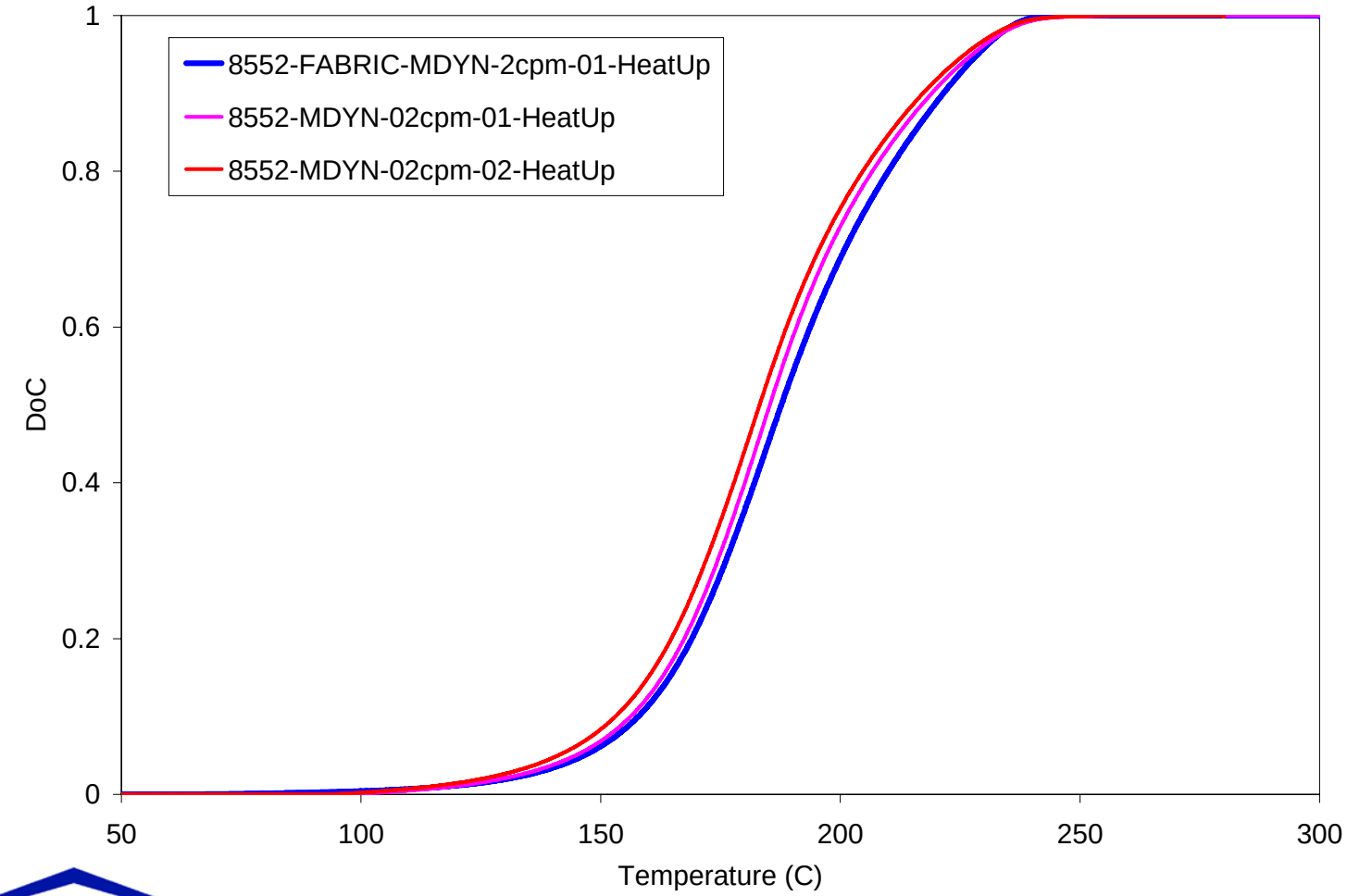
Goodness of Fit – Dyn DOC



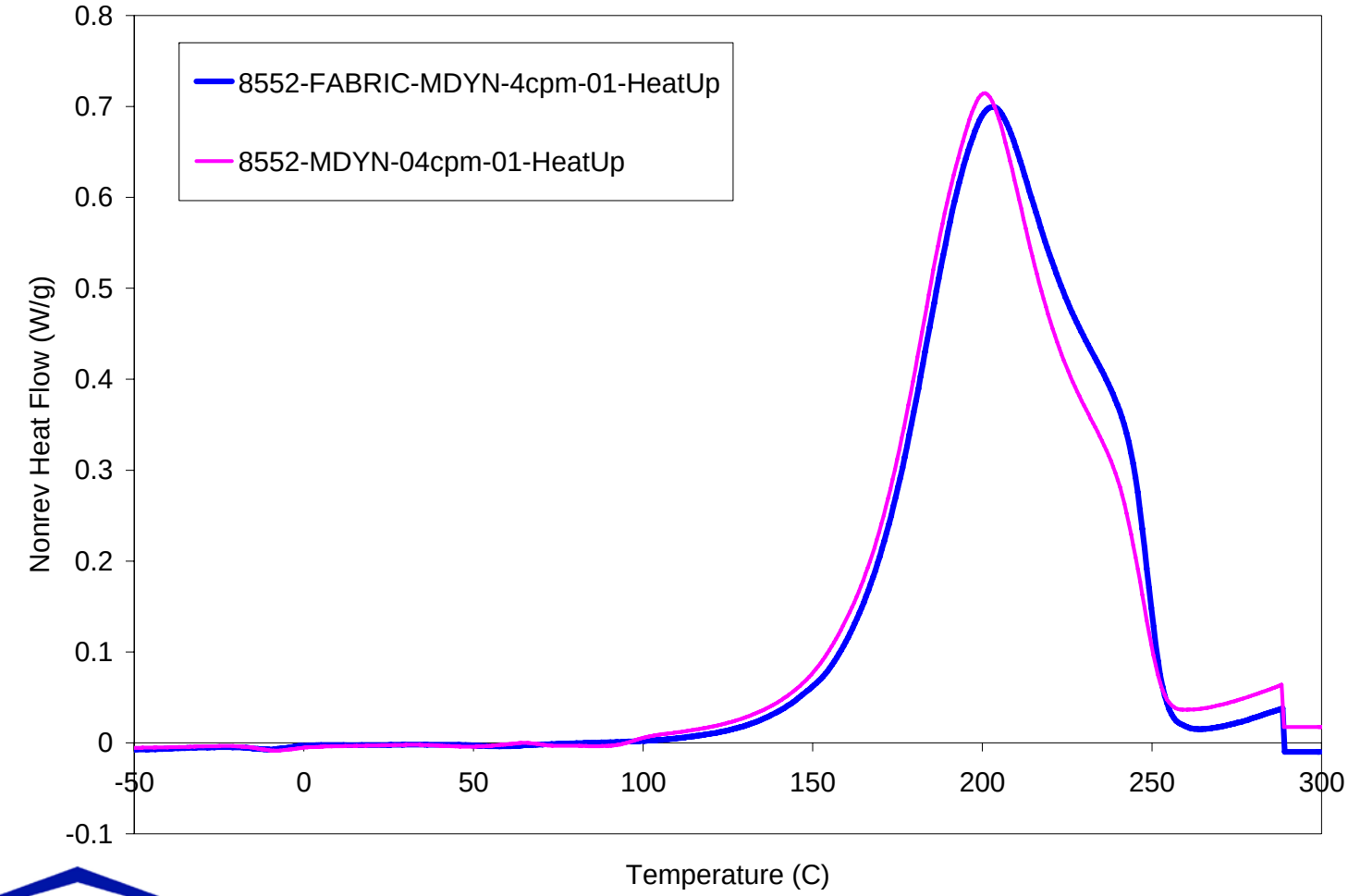
Other Forms - Fabric



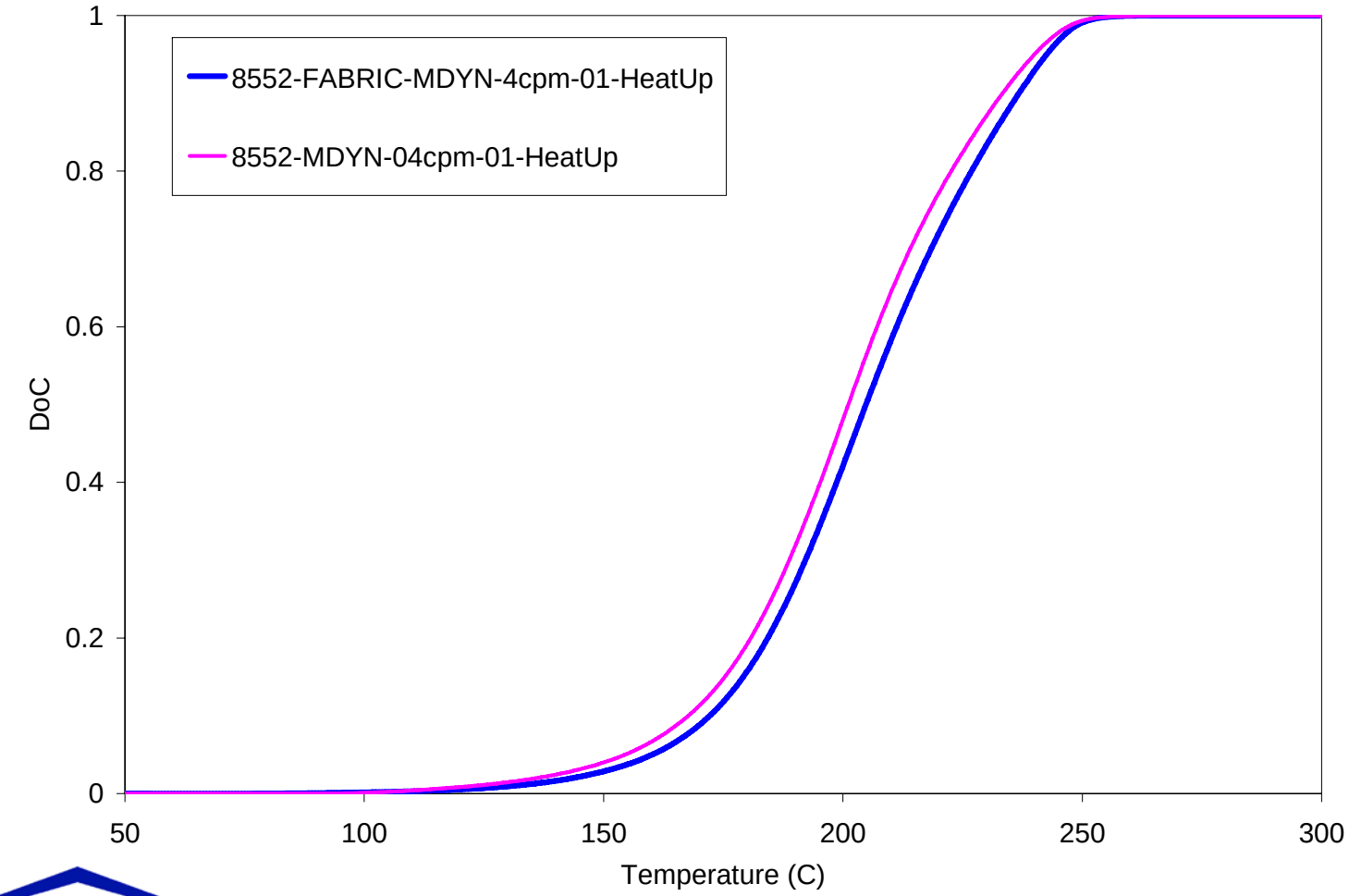
Other Forms - Fabric



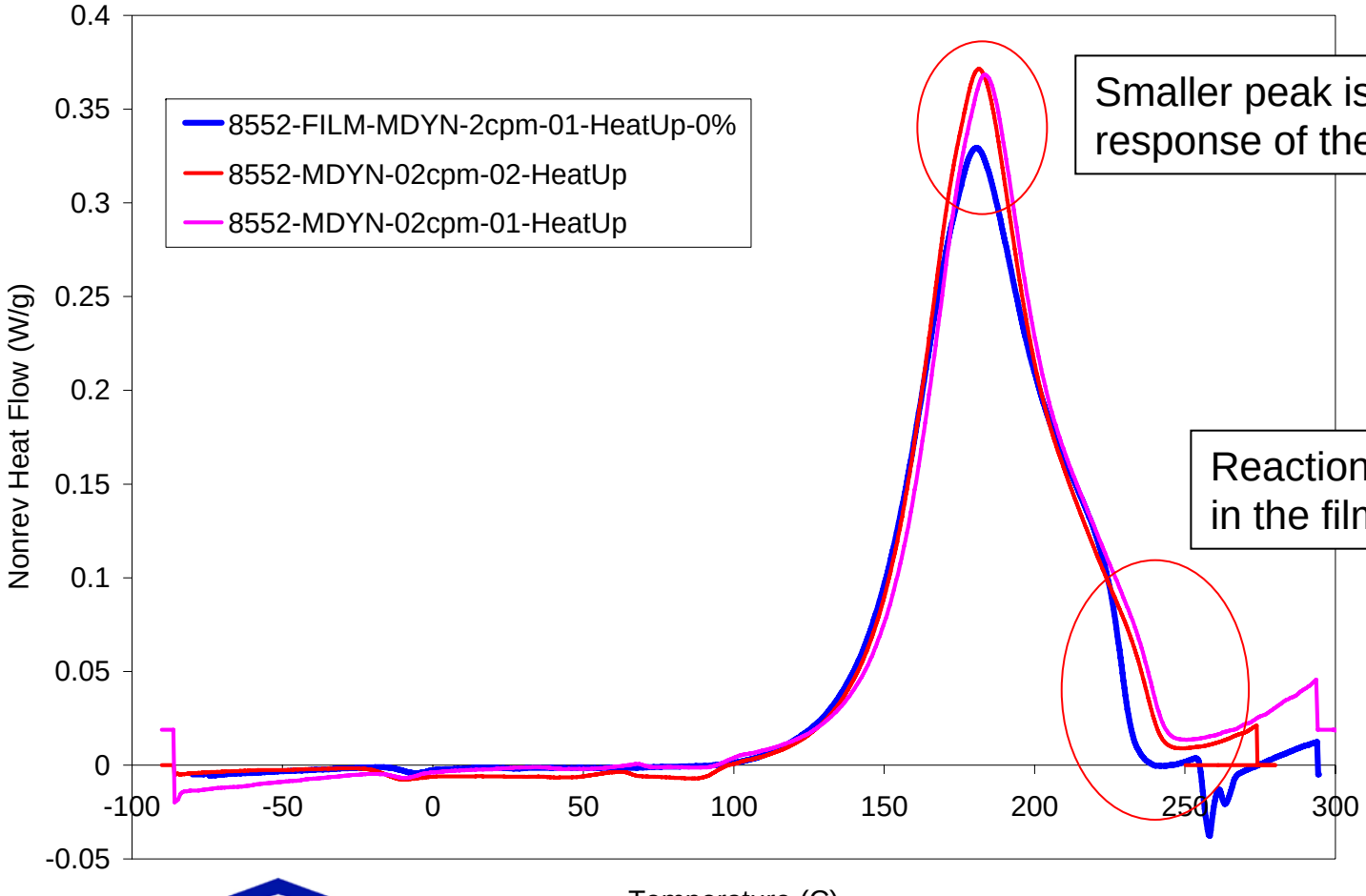
Other Forms - Fabric



Other Forms - Fabric



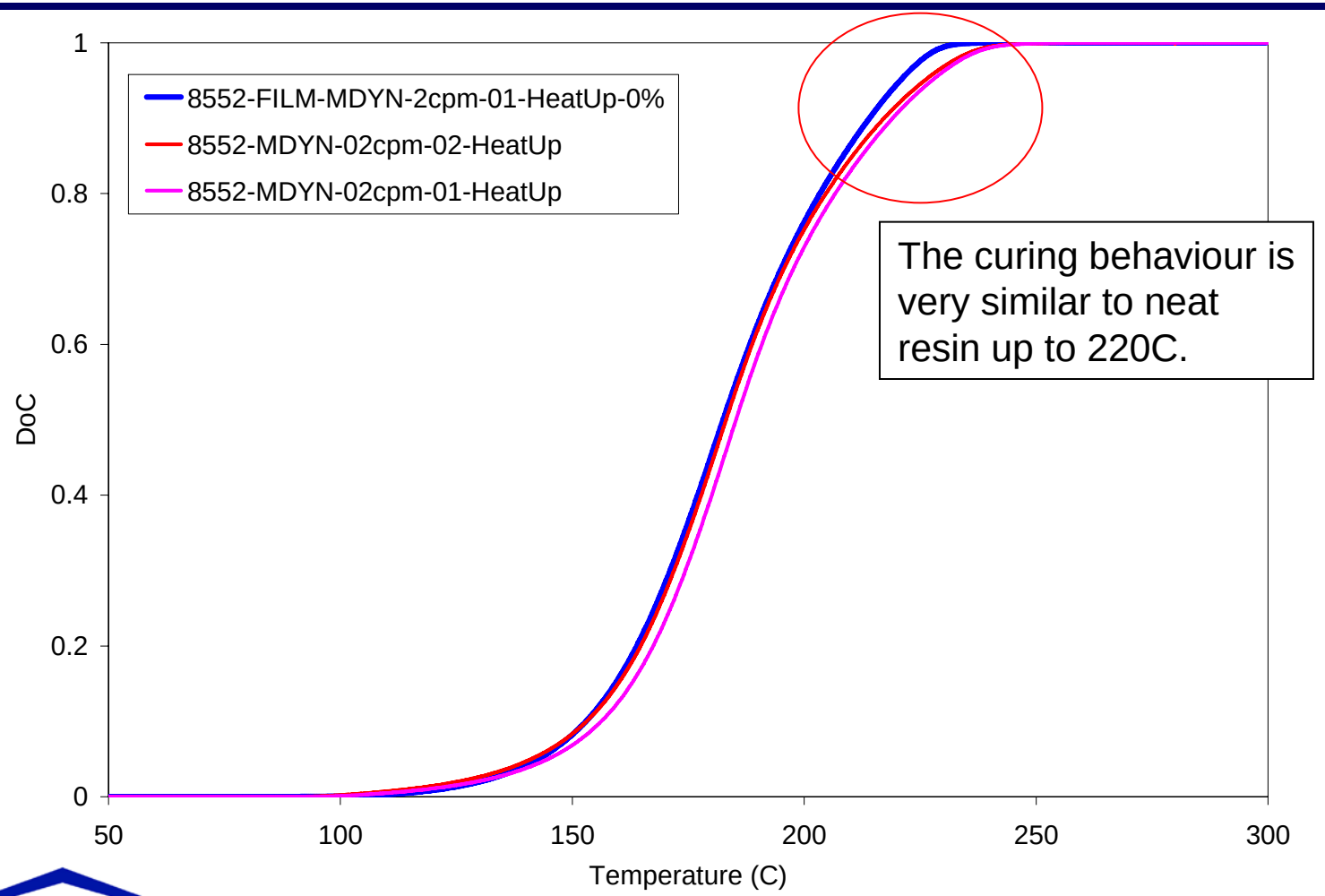
Other Forms - Film



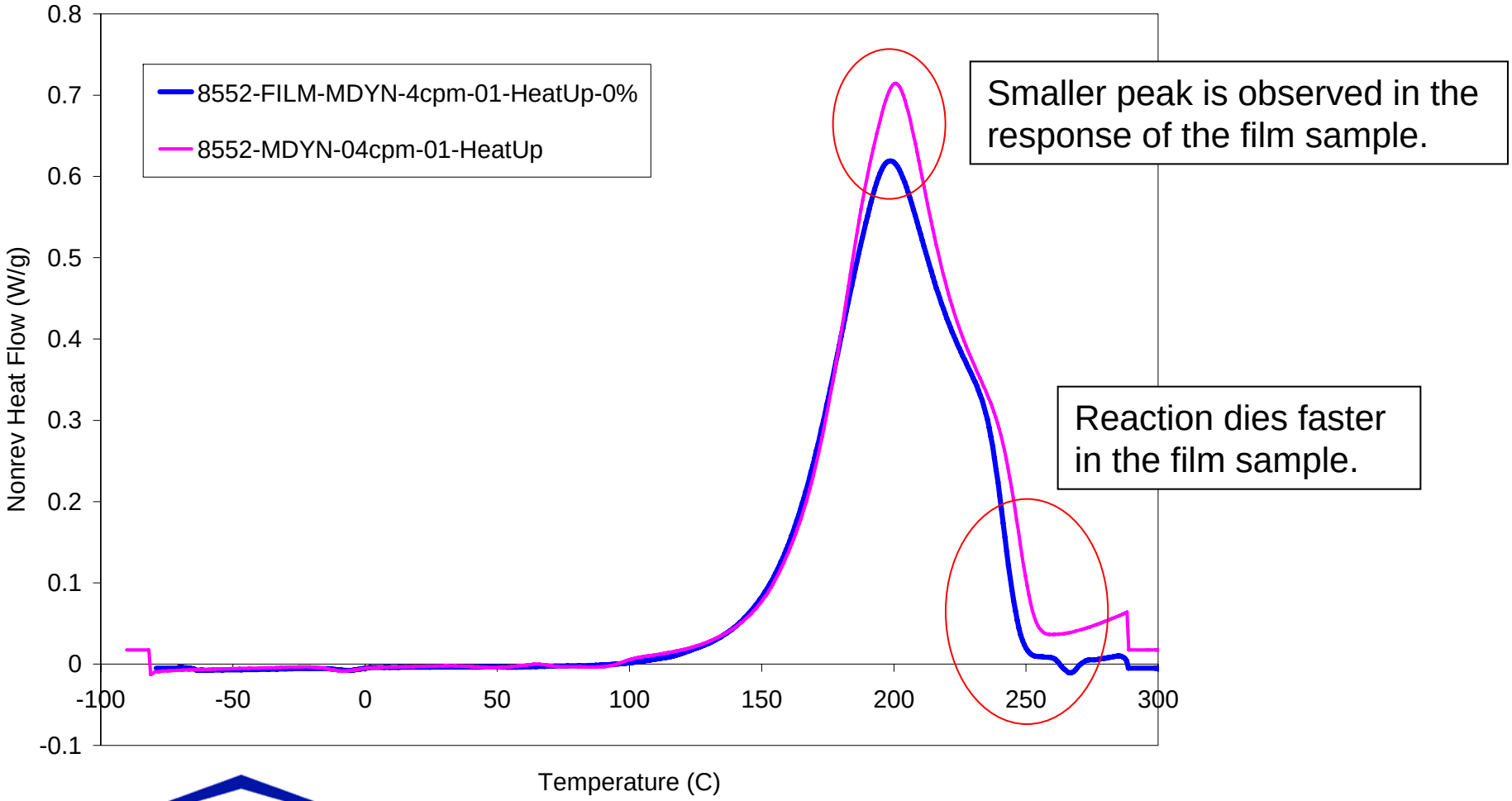
Smaller peak is observed in the response of the film sample.

Reaction dies faster in the film sample.

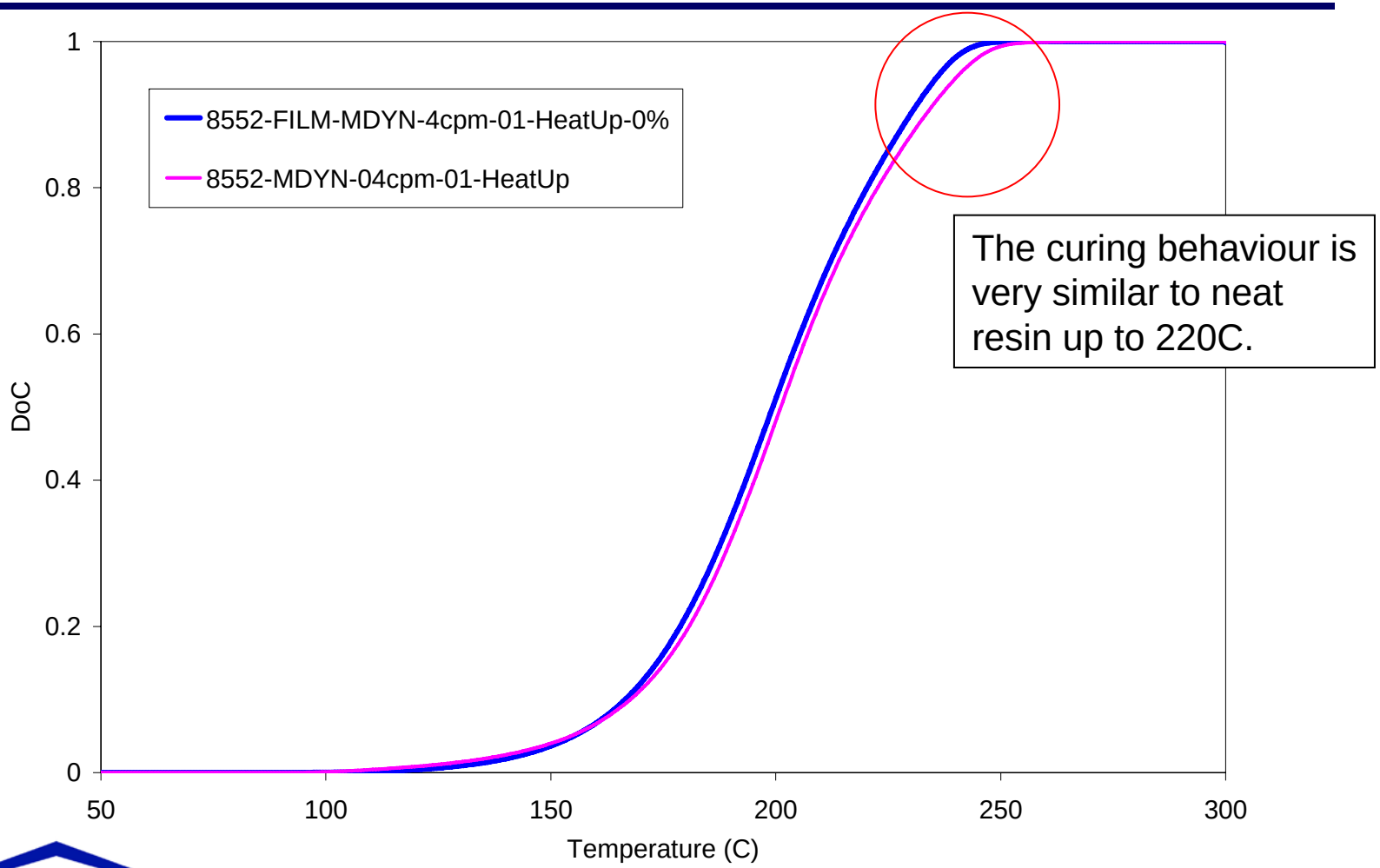
Other Forms - Film



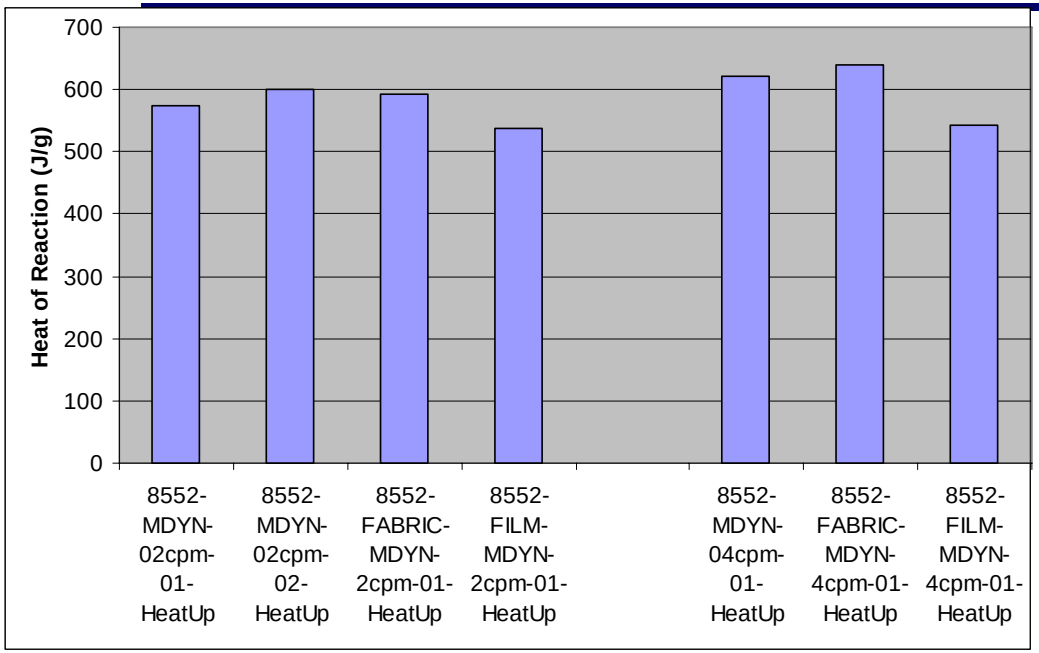
Other Forms - Film



Other Forms - Film



Other Forms – HR Values



Lower total heat of reaction is measured for the film compared to the neat resin. Considering the similarities between the responses of neat resin and film up to 220C, the model (developed based on neat resin) is considered valid for resin film. The error in the total HR of the film results in conservative estimation of exhothermic events.

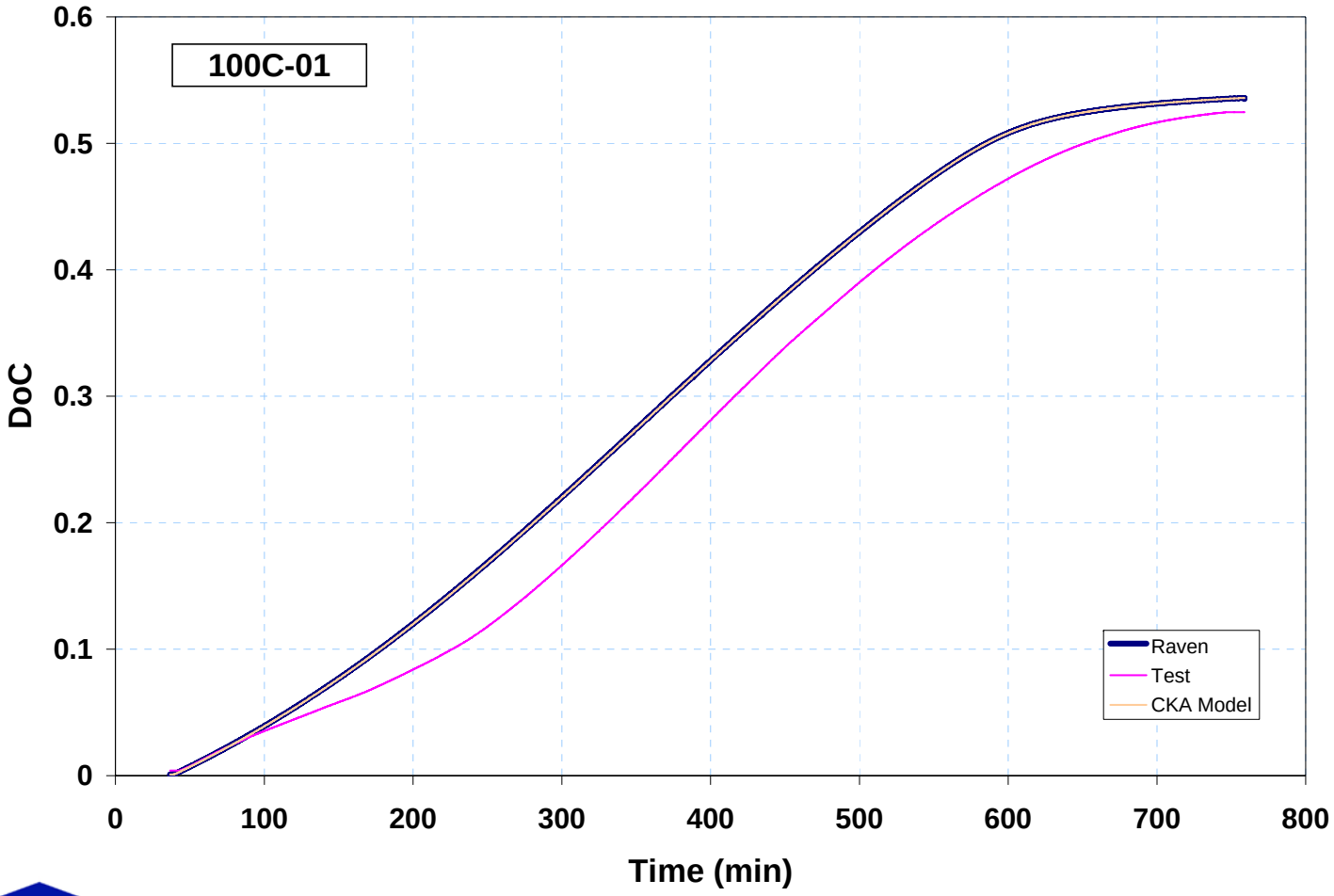
The fabric response is similar to the neat resin. The heat flow measured for the fabric samples was divided by the resin content (0.38), based on the material datasheet. The total HF resulted was in agreement with neat resin.

Test	Tg		Raw	Adjustment Ratio	Adjusted
	initial	Final	HR		HR
	(C)	(C)	(J/G)		(J/G)
8552-MDYN-02cpm-01-HeatUp	-8.15	202.95	575.43	1	575.43
8552-MDYN-02cpm-02-HeatUp	-10.09	217.28	601.65	1	601.65
8552-FABRIC-MDYN-2cpm-01-HeatUp	-7.88	200.78	225.22	0.38	592.68
8552-FILM-MDYN-2cpm-01-HeatUp	-5.07	209.67	537.95	1	537.95
8552-MDYN-04cpm-01-HeatUp	-6.66	203.1	621.82	1	621.82
8552-FABRIC-MDYN-4cpm-01-HeatUp	-8.11	195.81	243.50	0.38	640.80
8552-FILM-MDYN-4cpm-01-HeatUp	-4.43	206.03	543.48	1	543.48

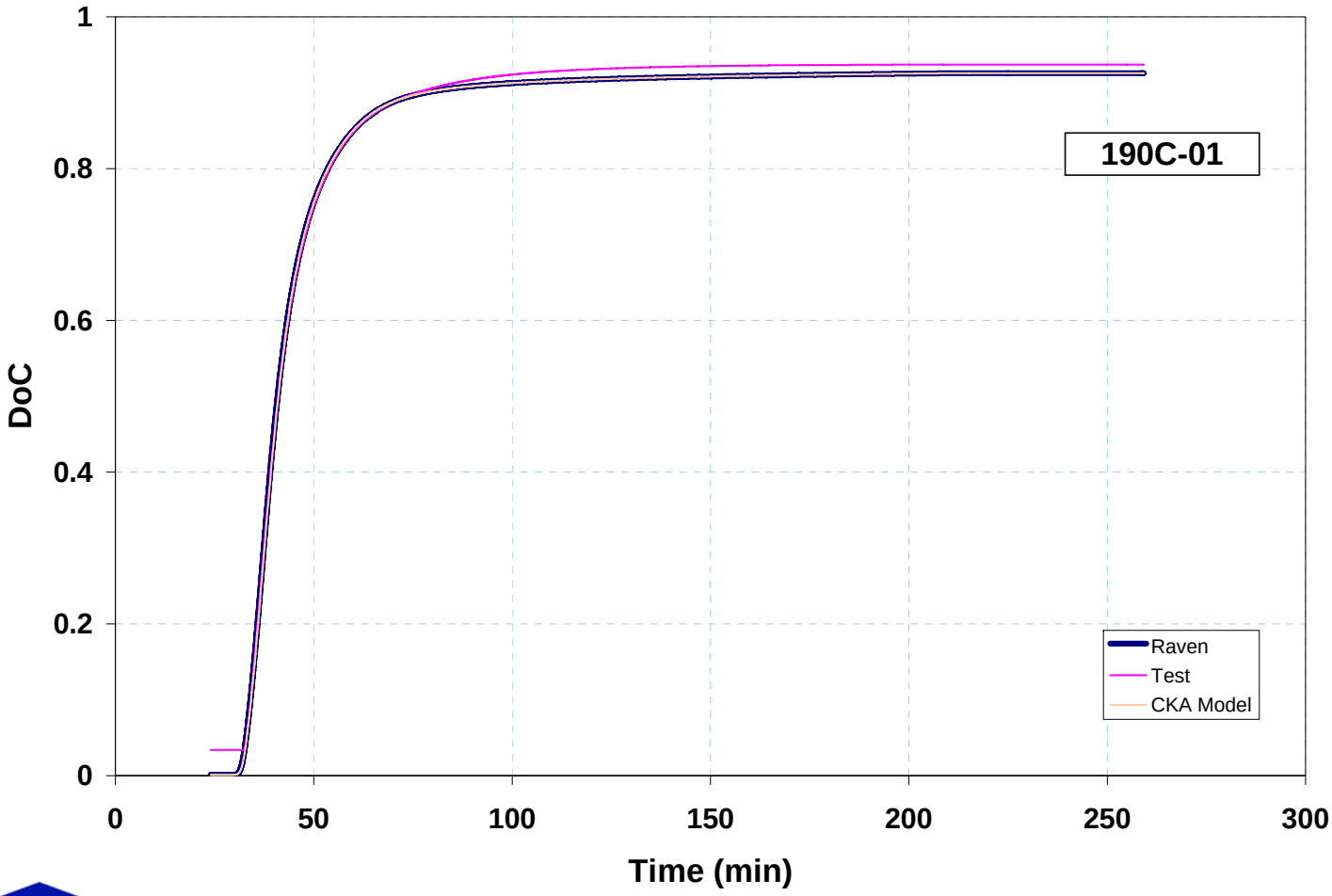
Assuming a mass resin content of 0.38, the fibre volume fraction is calculated to be 54.5%, which compares to the MDS reported laminate fraction of 58%.

More tests on fabric are done but not analysed yet.

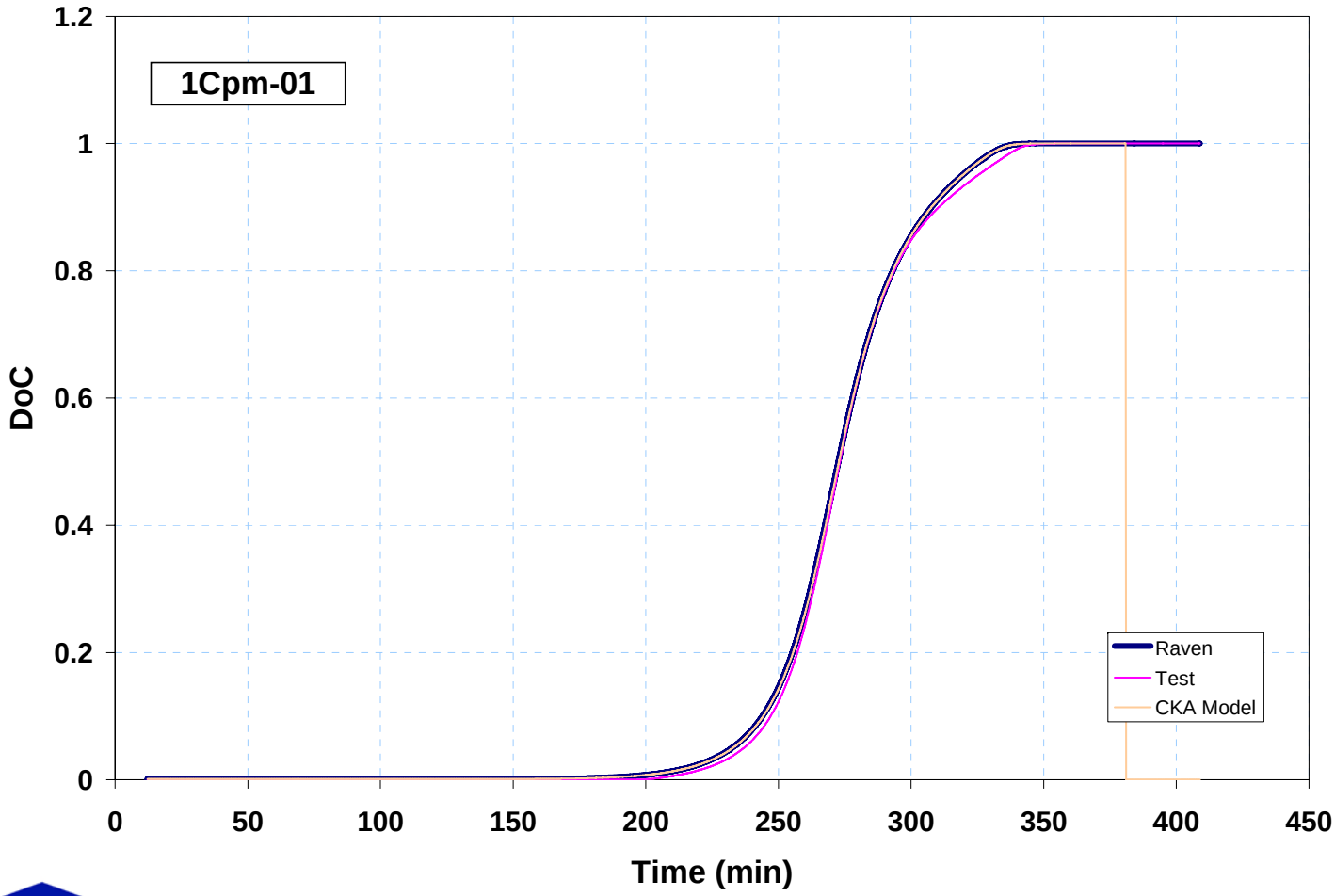
Material Model Verification – 100C



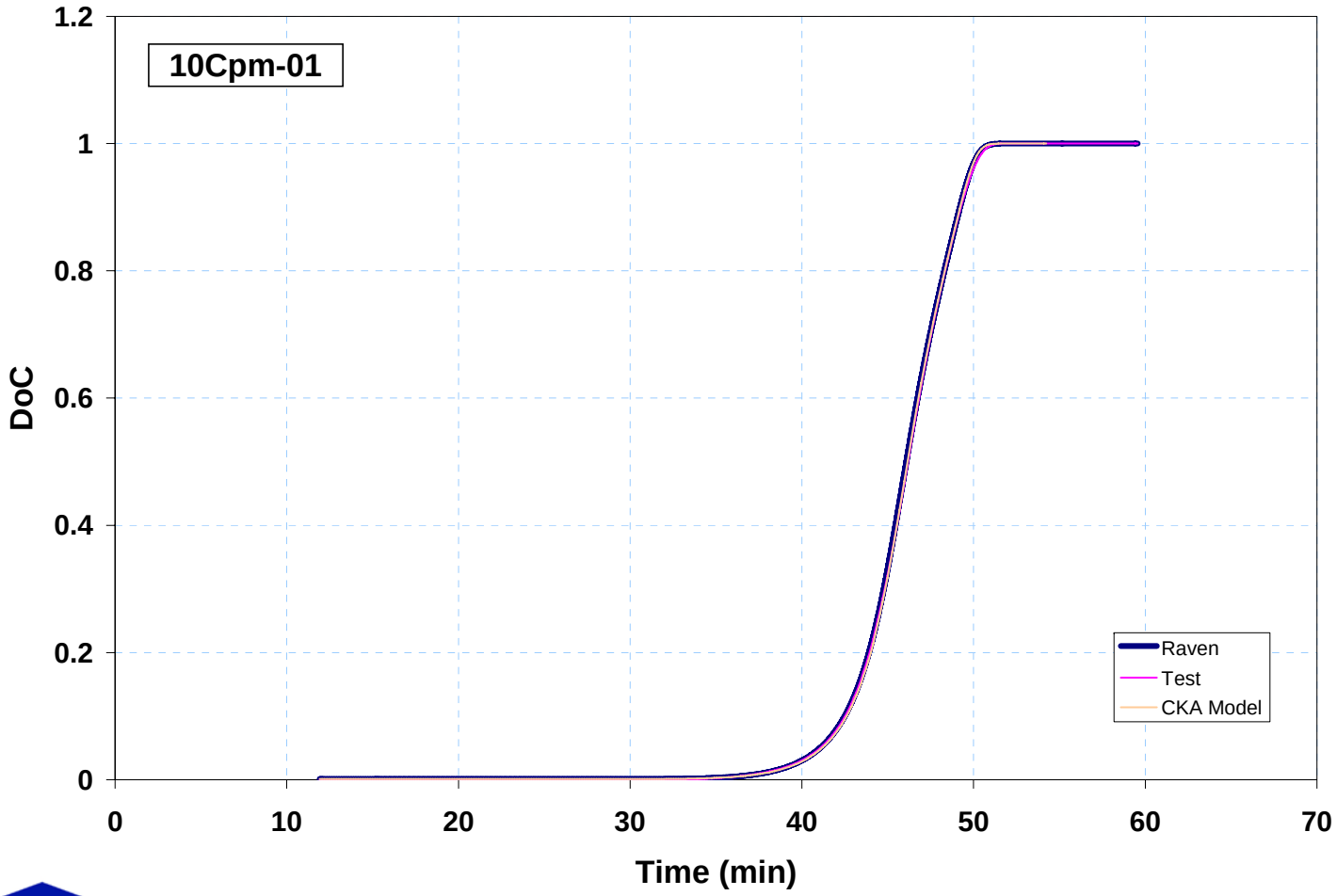
Material Model Verification – 190C



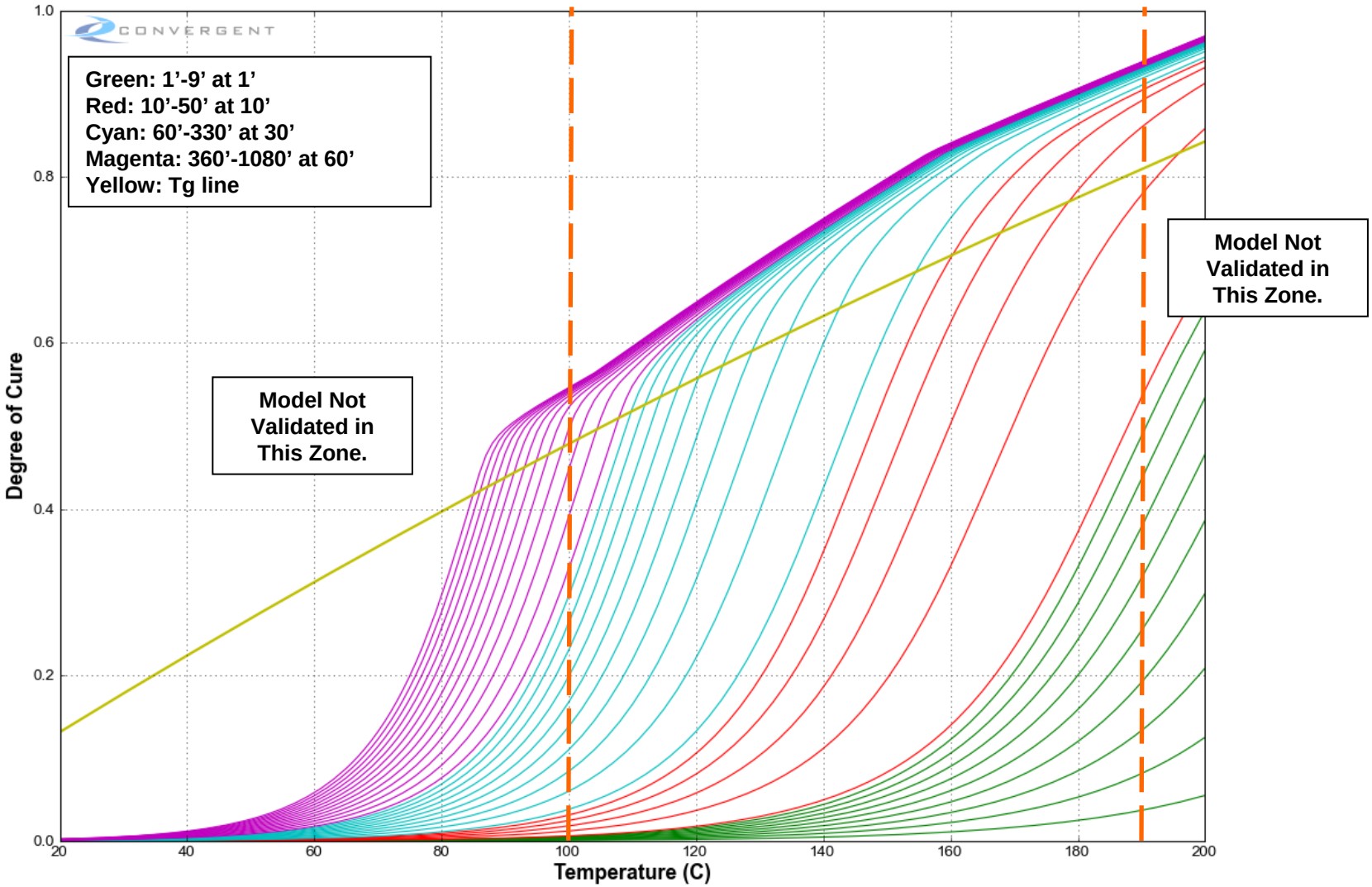
Material Model Verification – 1Cpm



Material Model Verification – 1Cpm

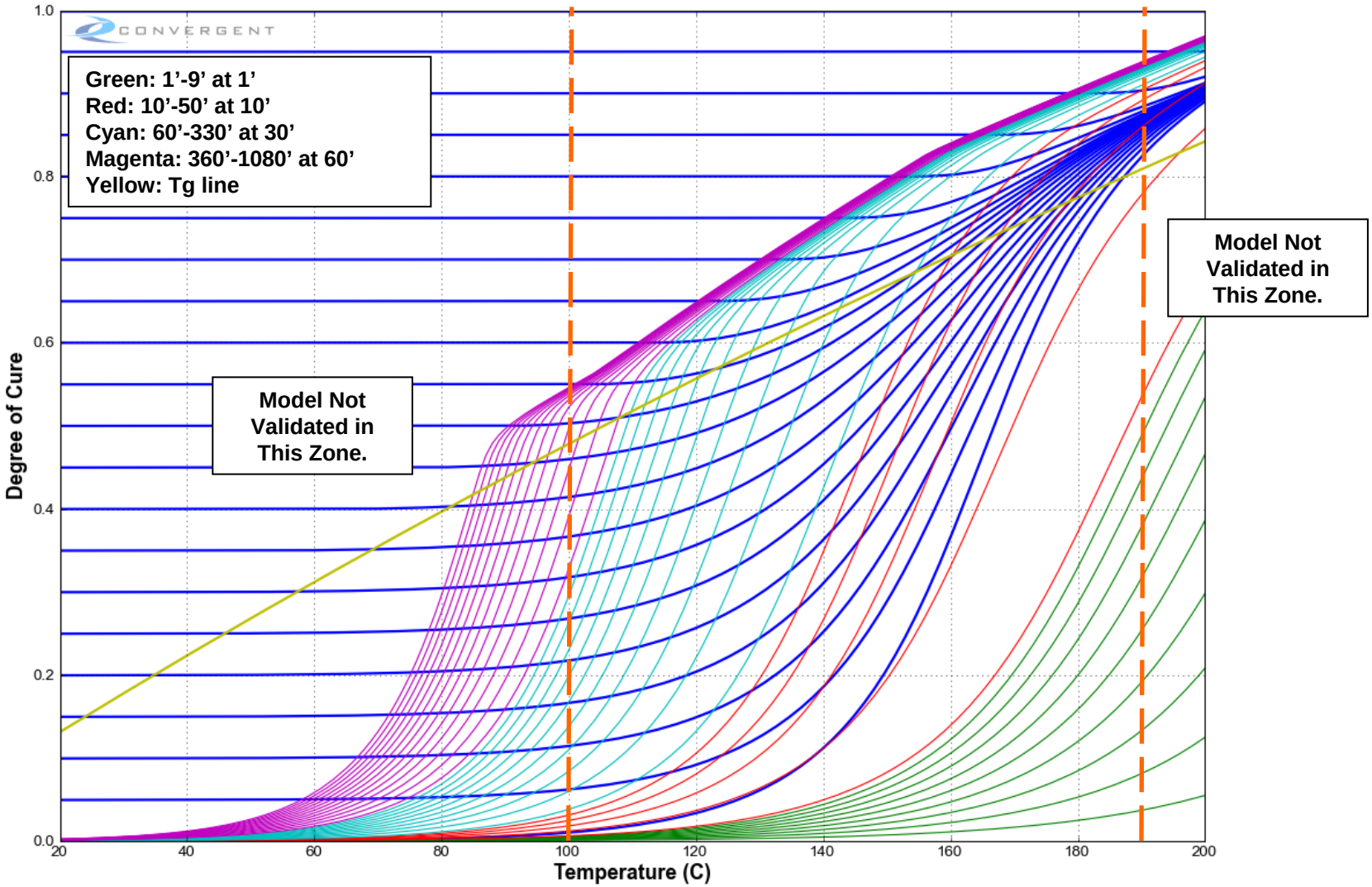


HEXPLY 8552 – Version 1.0



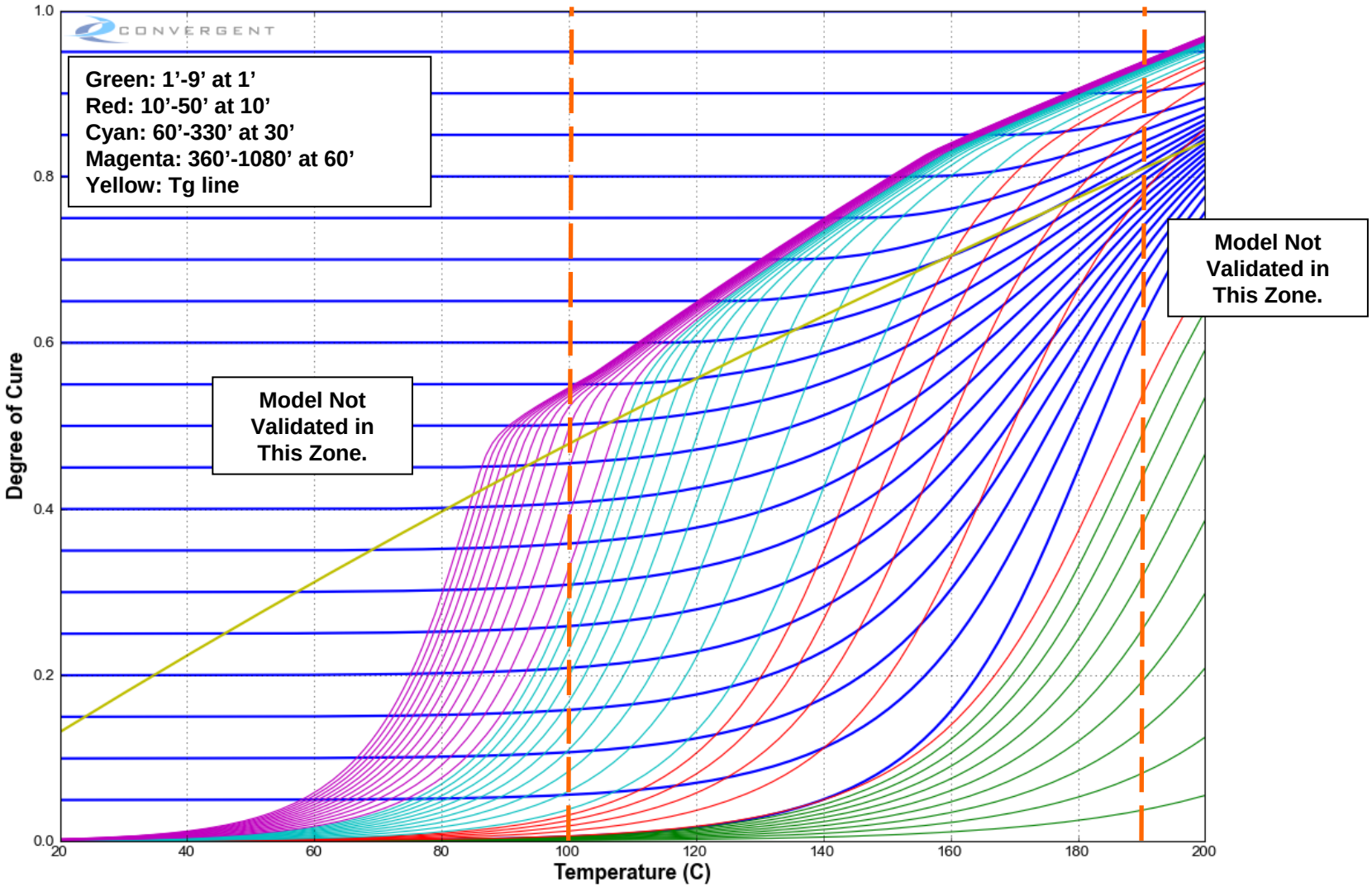
HEXPLY 8552 – Version 1.0

1°C/min



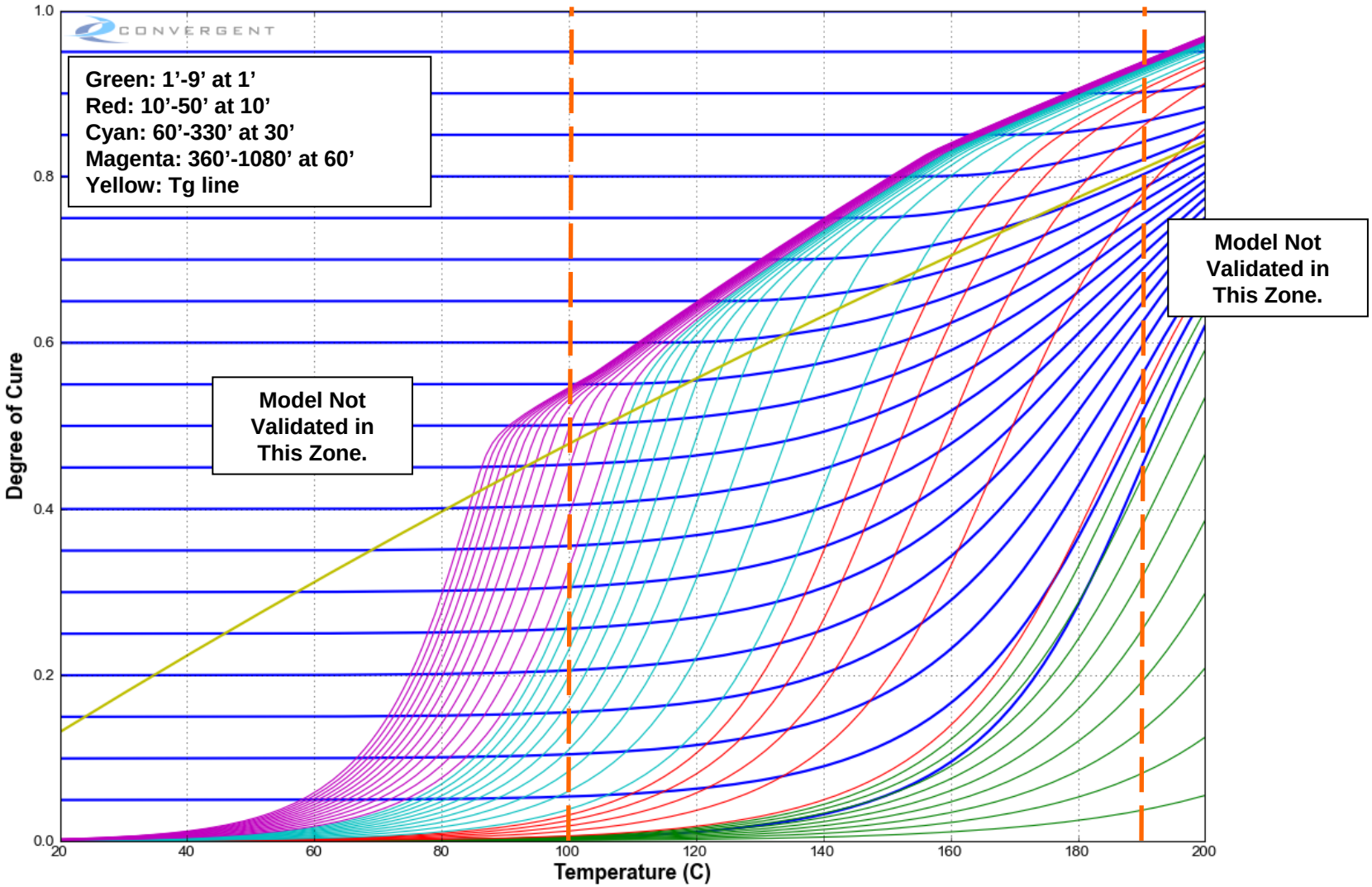
HEXPLY 8552 – Version 1.0

2°C/min



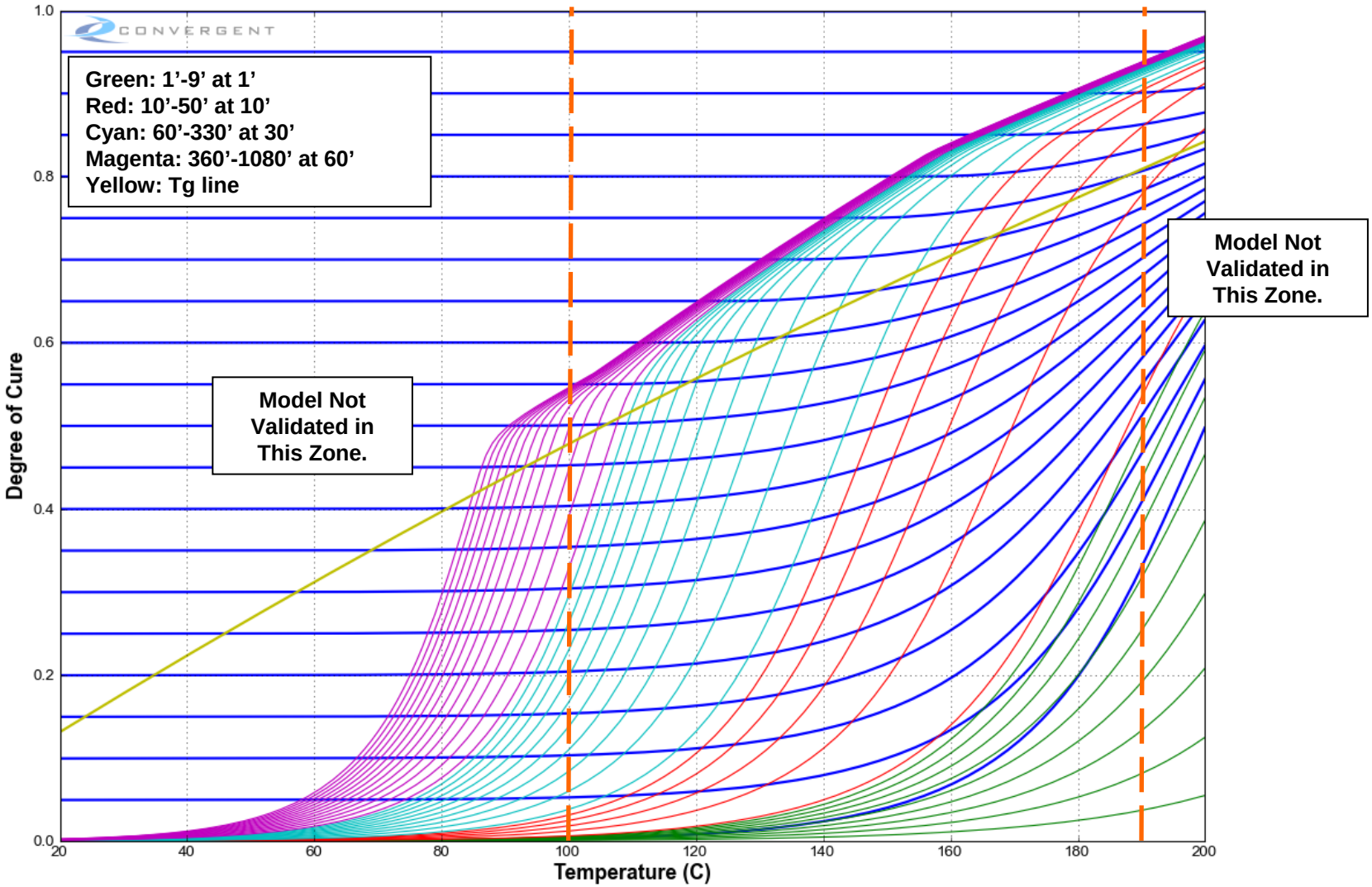
HEXPLY 8552 – Version 1.0

3°C/min



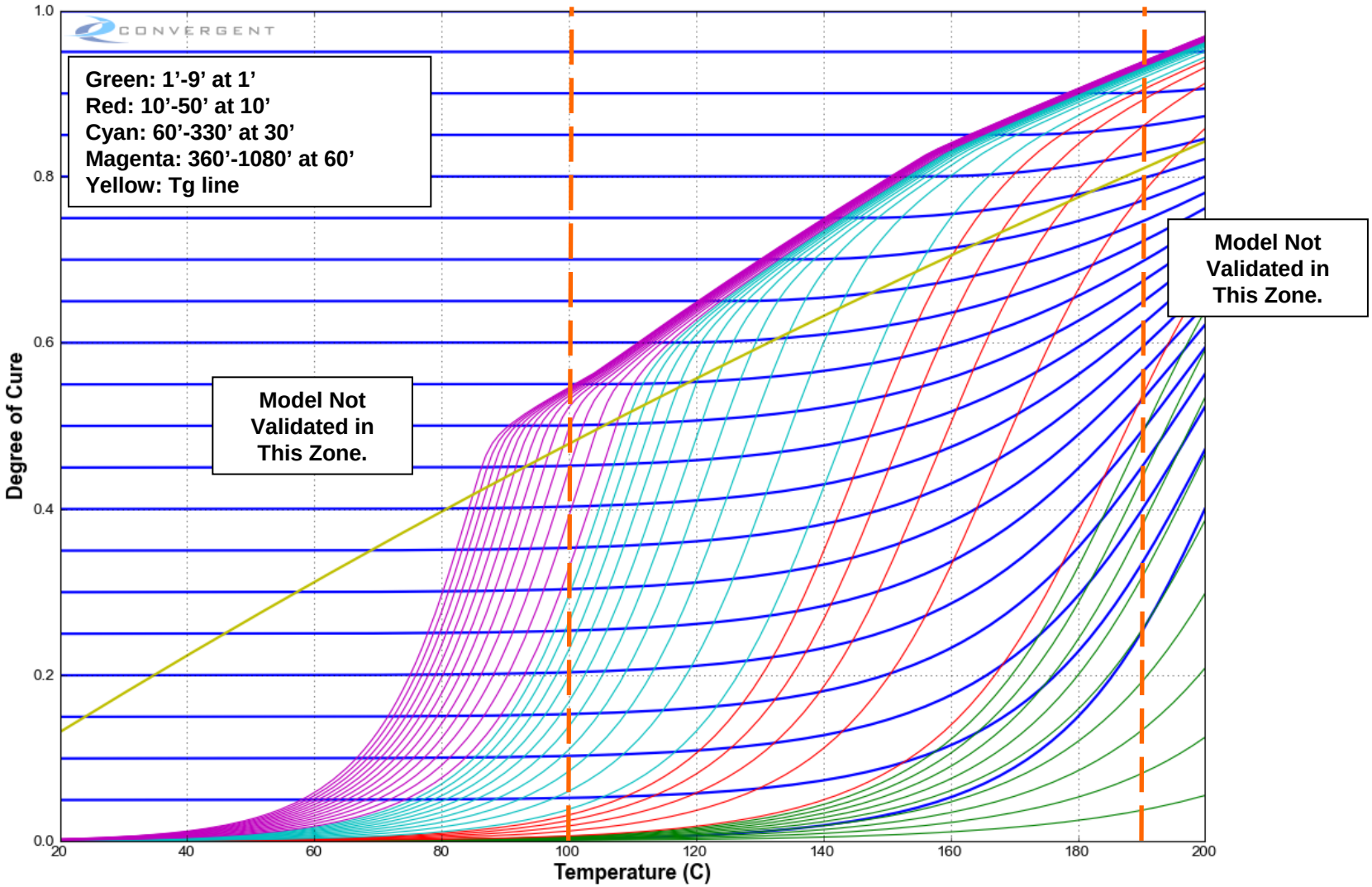
HEXPLY 8552 – Version 1.0

4°C/min



HEXPLY 8552 – Version 1.0

5°C/min



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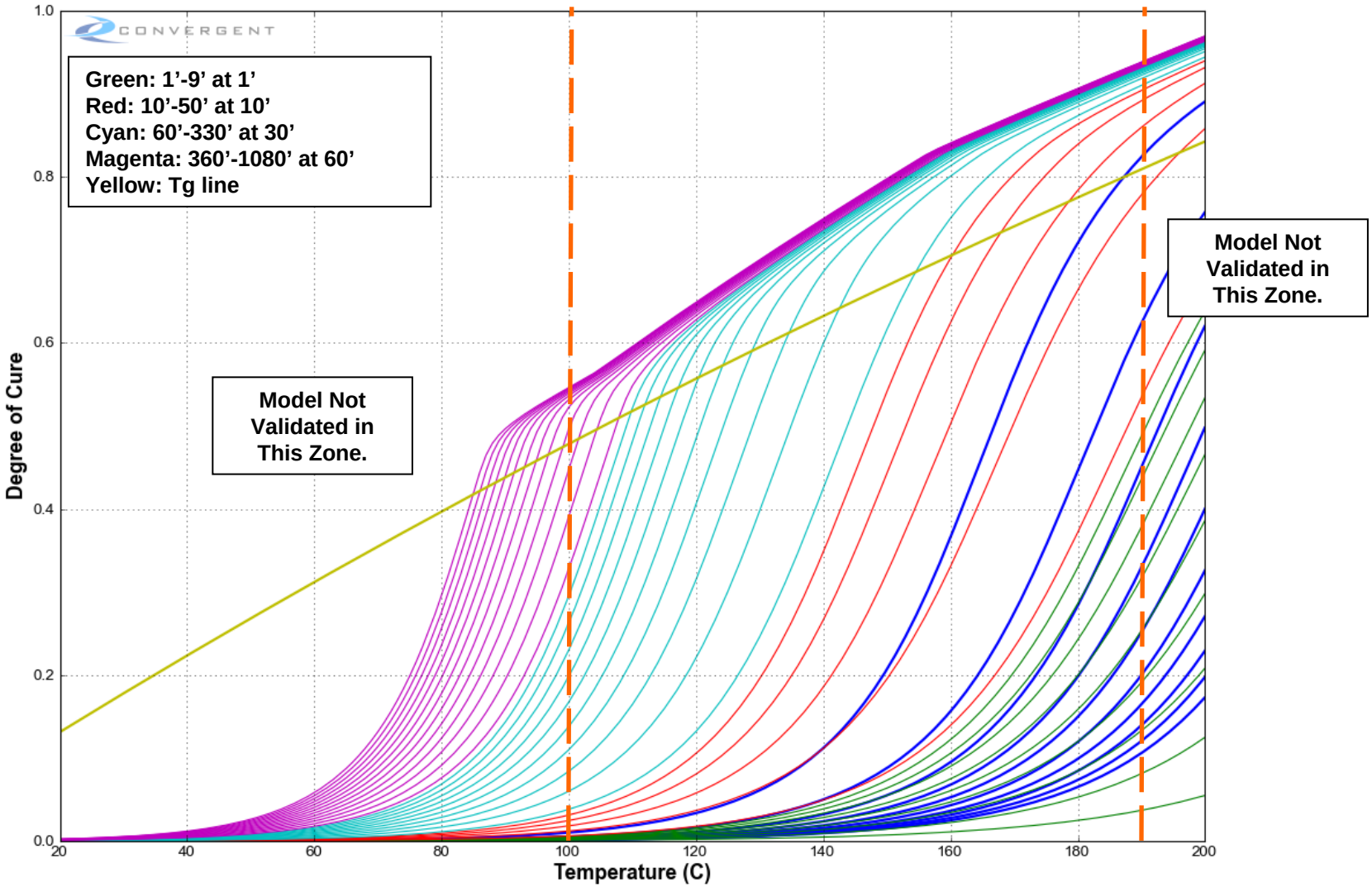
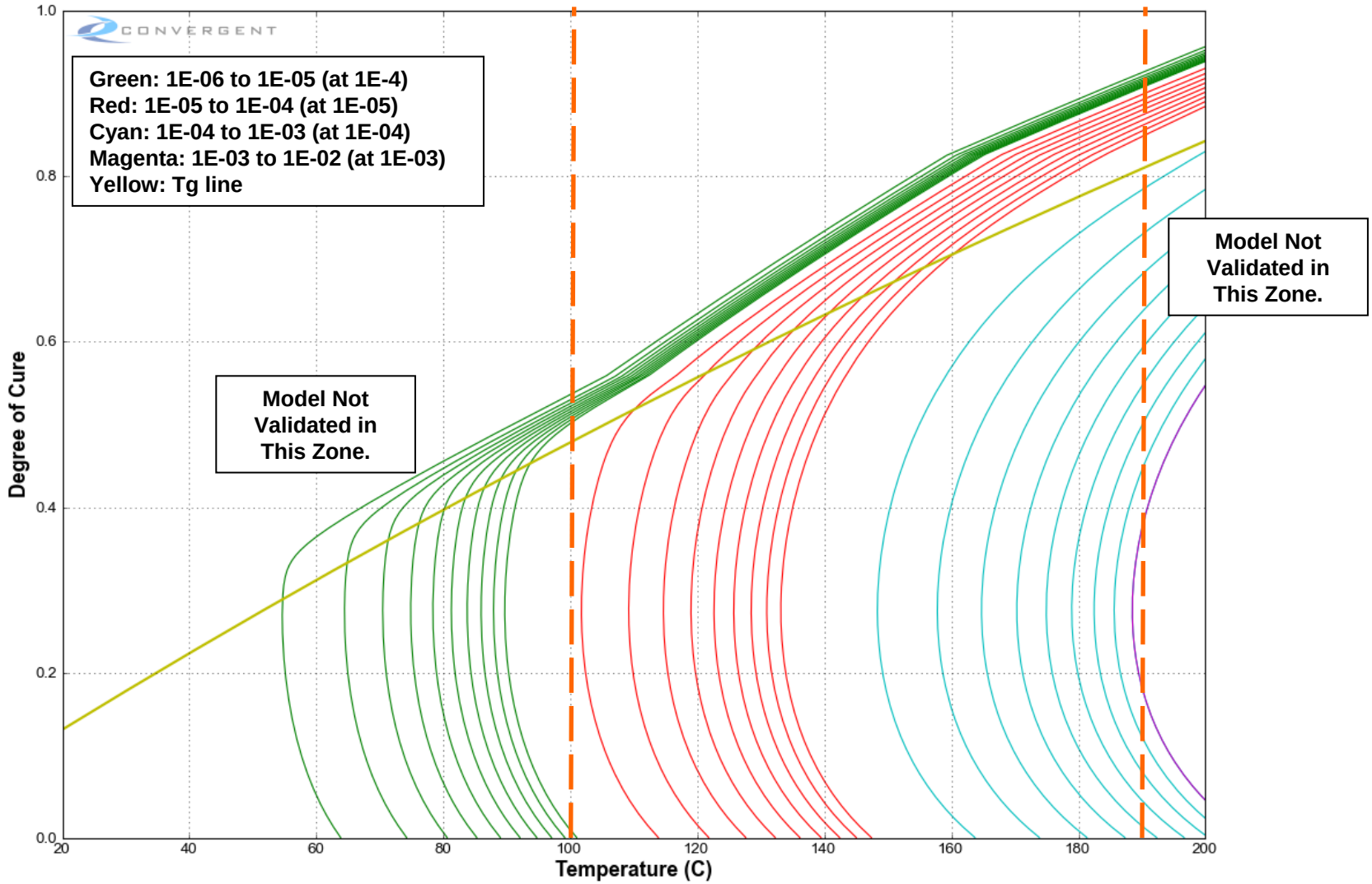
1-10°C/min Uncured



HEXPLY 8552 – Version 1.0

Cure Rate



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') as its real part and an elastic complex part (η''), defined as:

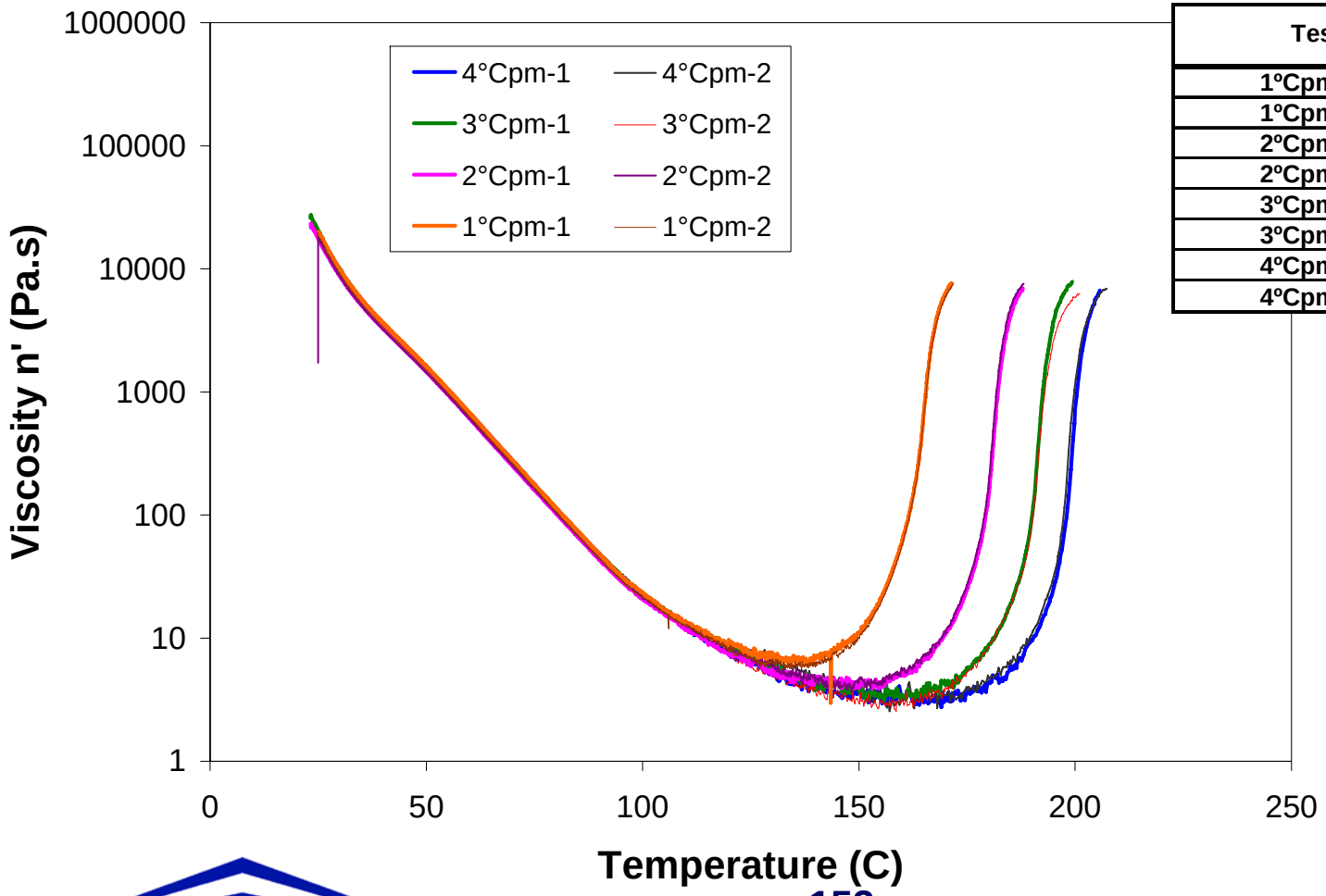
$$\eta^* = \eta' + i\eta'' \qquad \text{where} \qquad \eta' = \frac{G''}{\omega} \quad \text{and} \quad \eta'' = \frac{G'}{\omega}$$

For low shear rates, the dynamic viscosity corresponds mainly to the real part of the complex viscosity and can be considered the steady state viscosity of the fluid[1]. In other words:

$$\boxed{\mu = \eta'}$$

The viscosity model developed here is based on the measured η', i.e. the dynamic viscosity response of the material.

Raw Data



Test	Temperature (ºC)	
	Start	End
1ºCpm-01	25.1	171.4
1ºCpm-02	24.9	171.8
2ºCpm-01	23.3	187.9
2ºCpm-02	25	188.1
3ºCpm-01	23.2	199.3
3ºCpm-02	23.4	201.1
4ºCpm-01	25	205.7
4ºCpm-02	24.5	207.3

Mathematical Model

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

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

where

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

x_g is the degree of cure at gelation

[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.

$$\mu = \mu_{01} e^{\frac{E_1}{RT}} + \mu_{02} e^{\frac{E_2}{RT}} \left(\frac{x_g}{x_g - x} \right)^{\left(A + Bx + Cx^2\right)}$$

$$\begin{cases} E_1 = 81{,}908 & \mu_{01} = 7.5 \times 10^{-11} \\ E_2 = 13{,}228 & \mu_{01} = 0.0481 \\ x_g = 0.545 & A = 2.466 \\ B = 0 & C = 0 \end{cases}$$

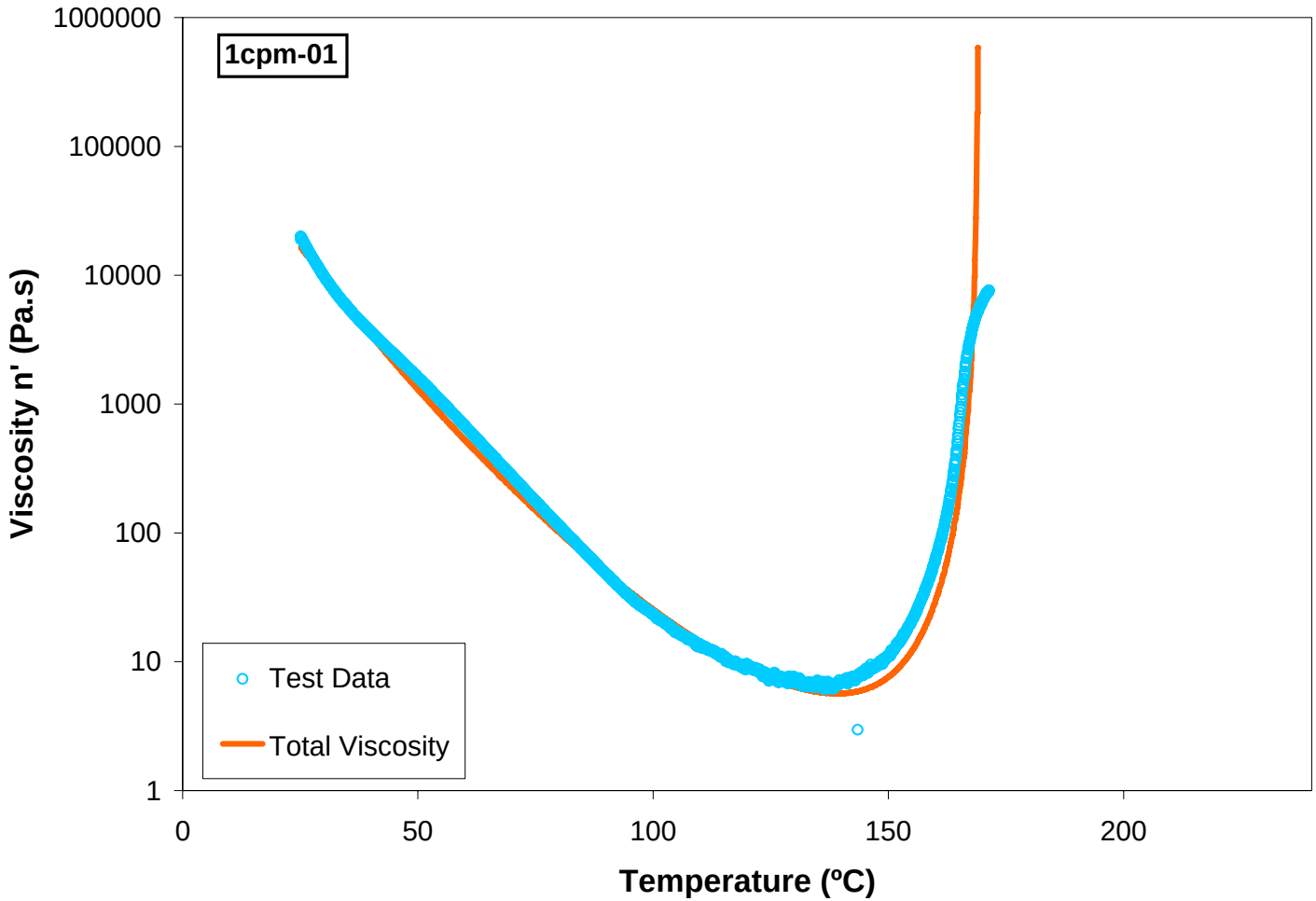
Model's range of validity

25ºC < Temperature < 250ºC

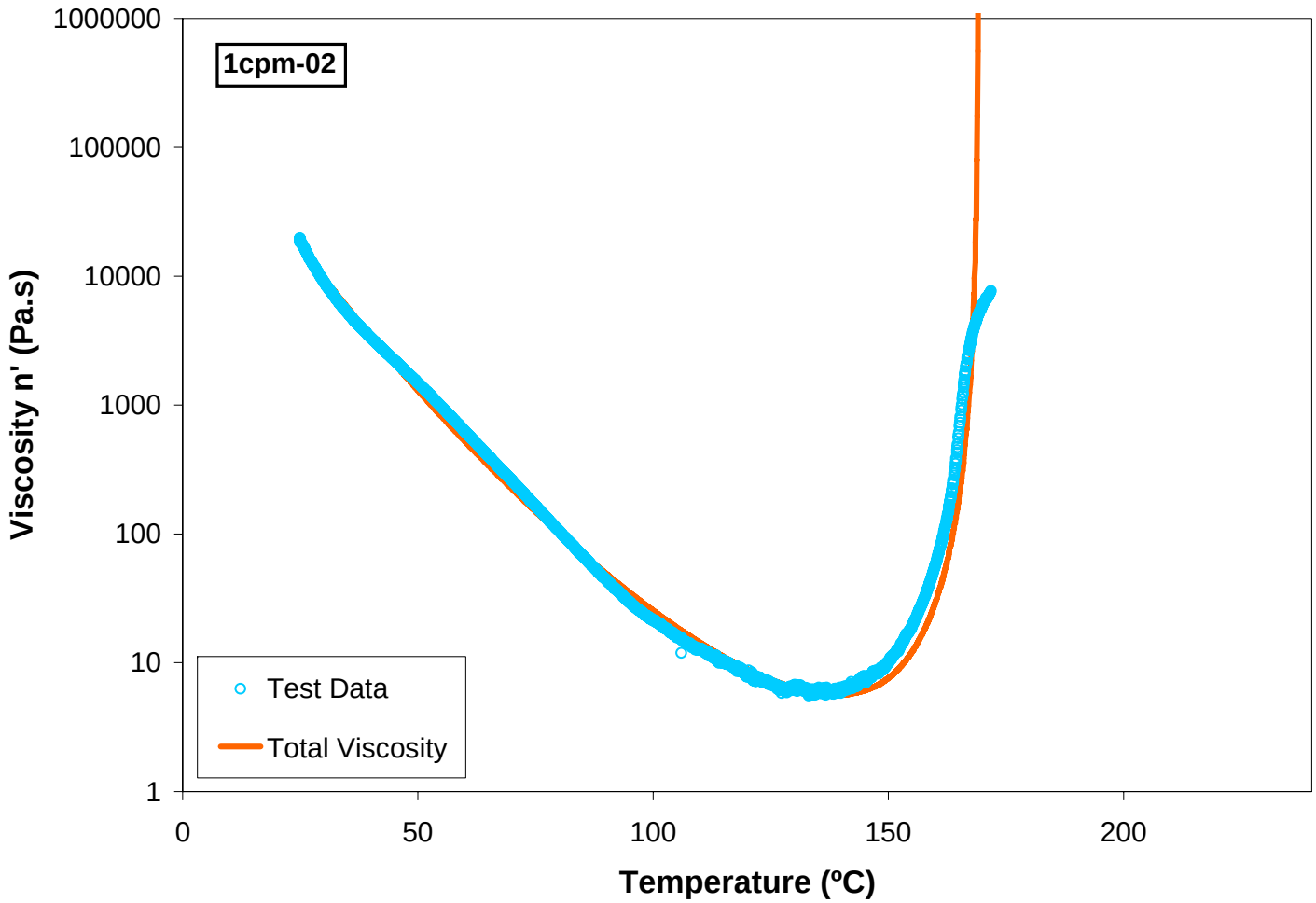
$1 < \eta' < 10^6$

Frequency: ω = 1 Hz

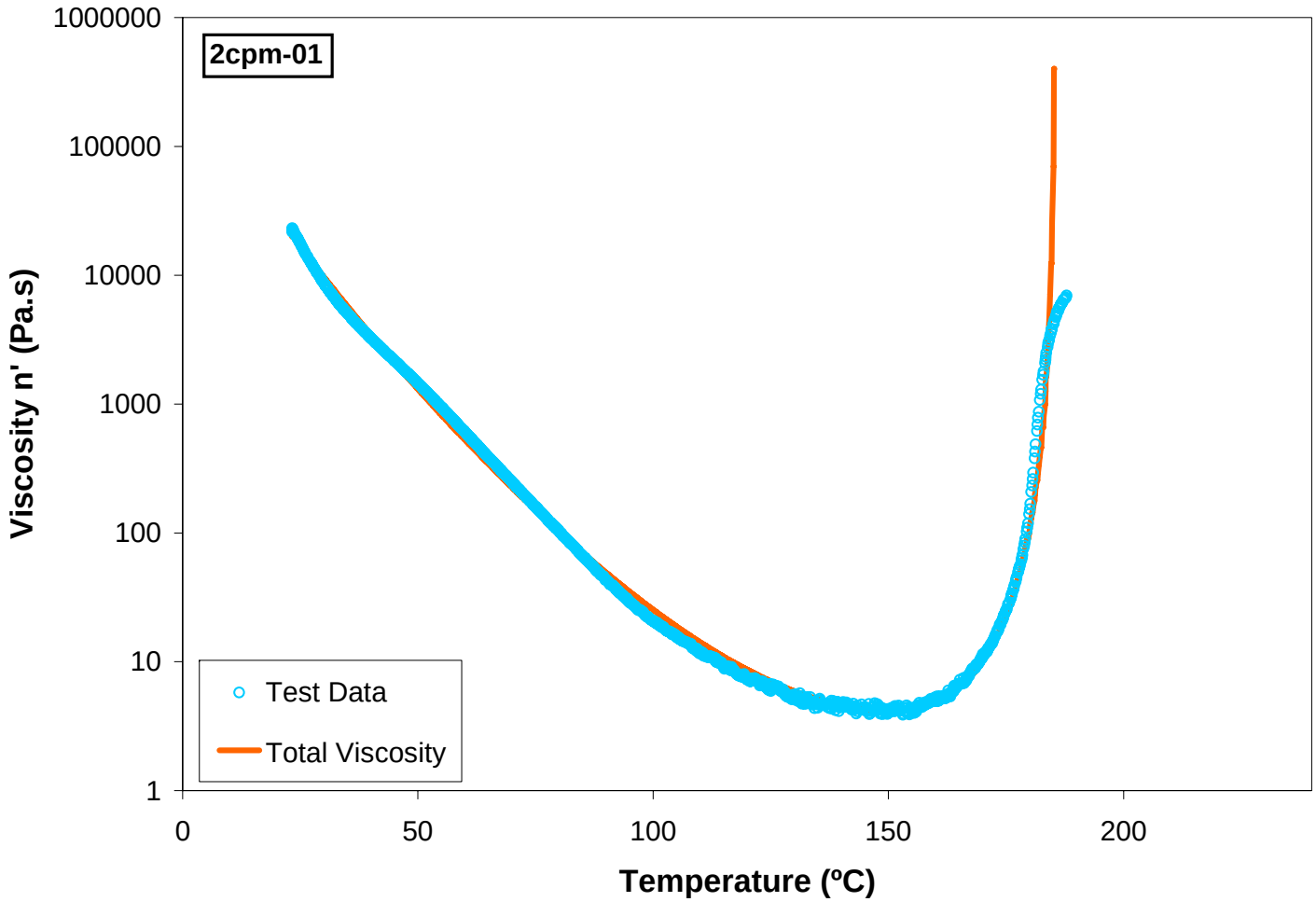
Dynamic Tests – 1cpm-1



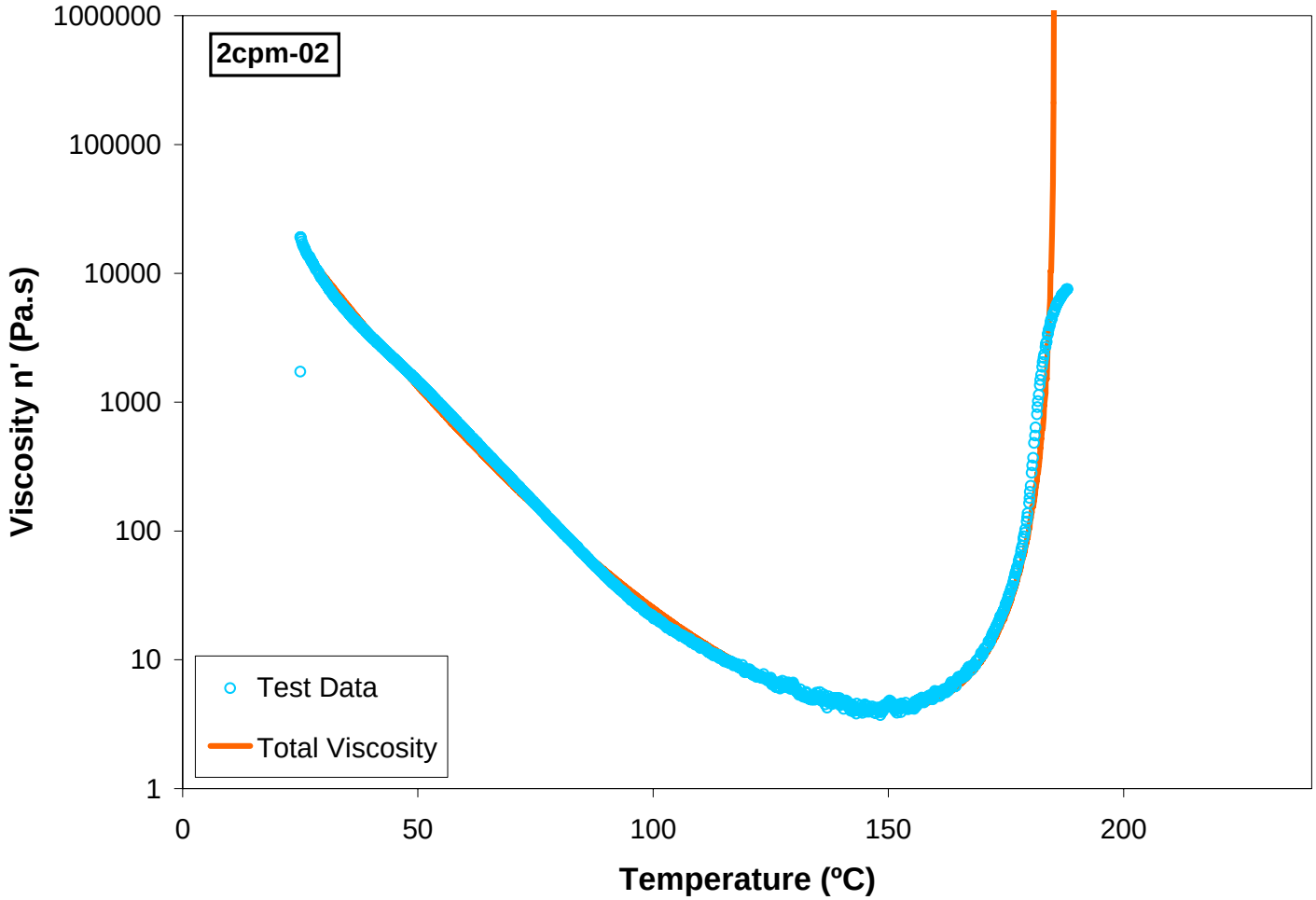
Dynamic Tests – 1cpm-2



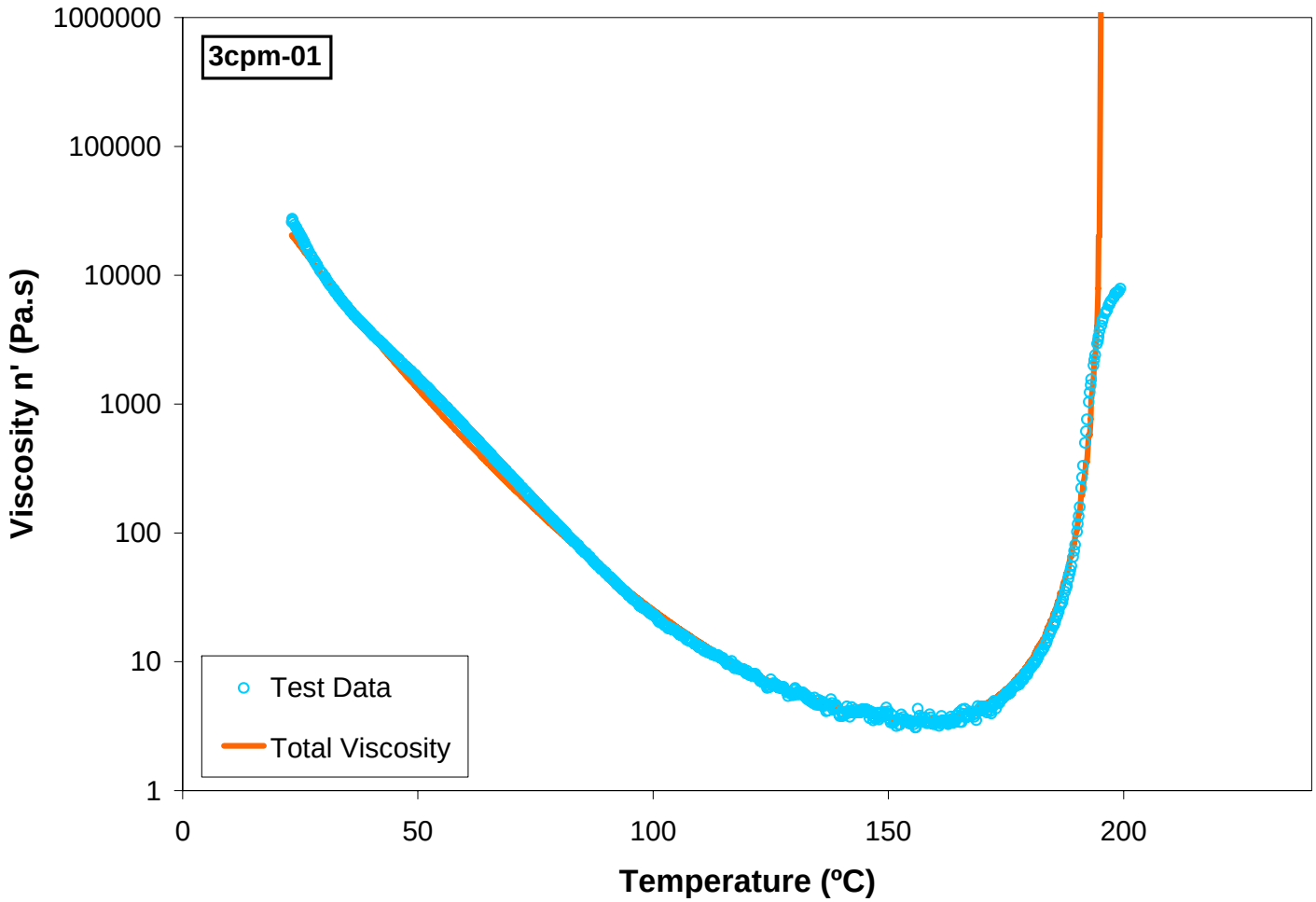
Dynamic Tests – 2cpm-1



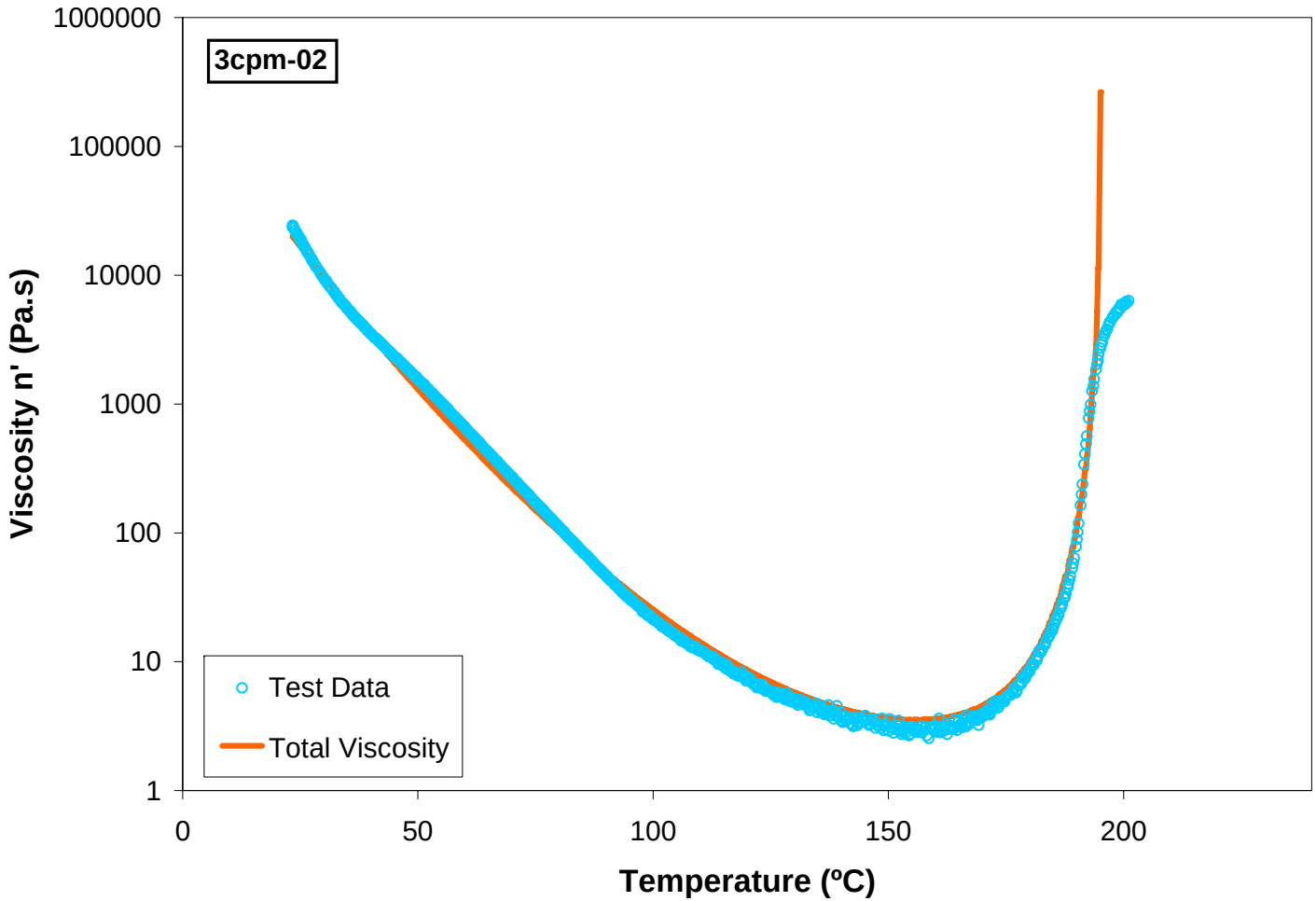
Dynamic Tests – 2cpm-2



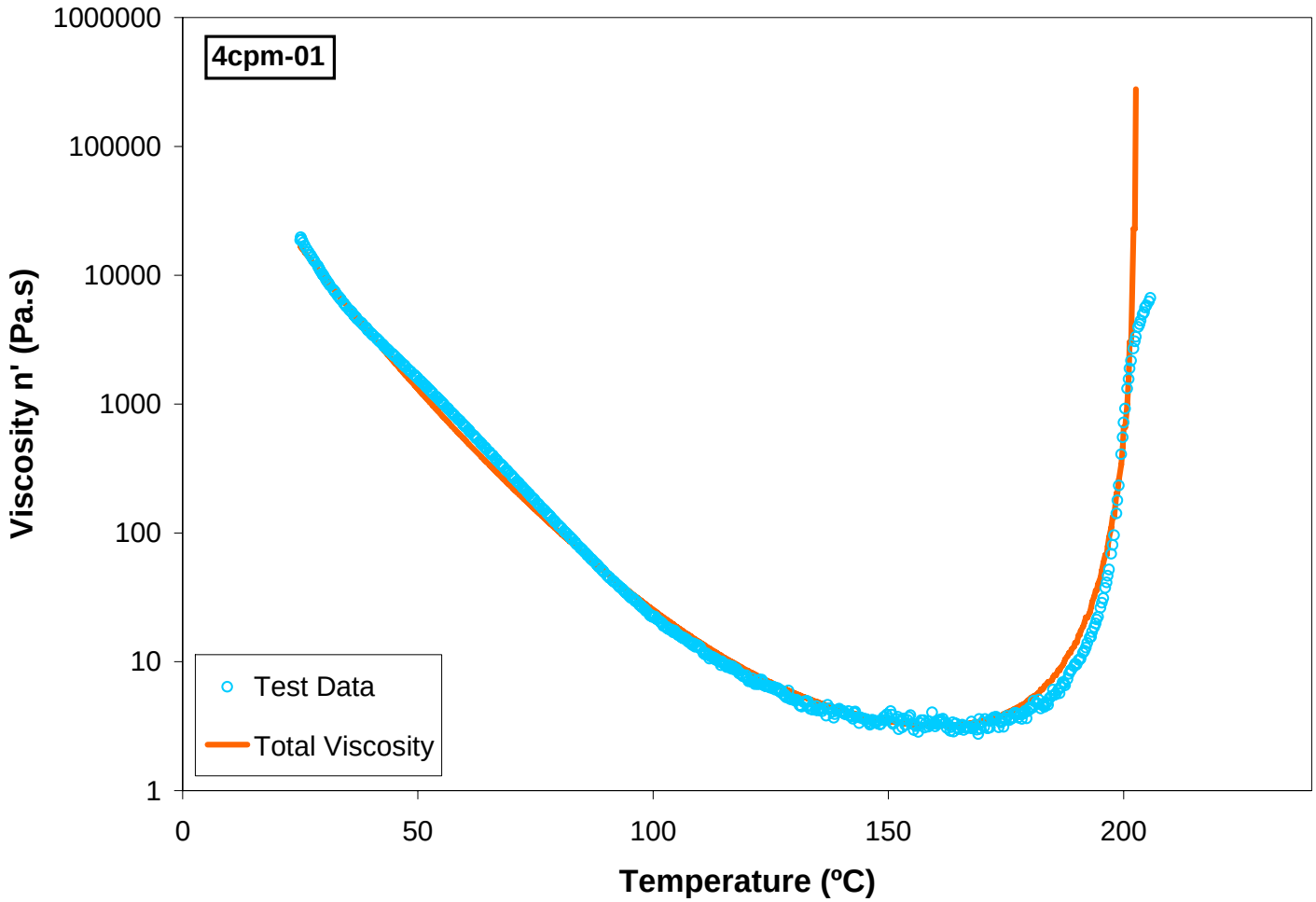
Dynamic Tests – 3cpm-1



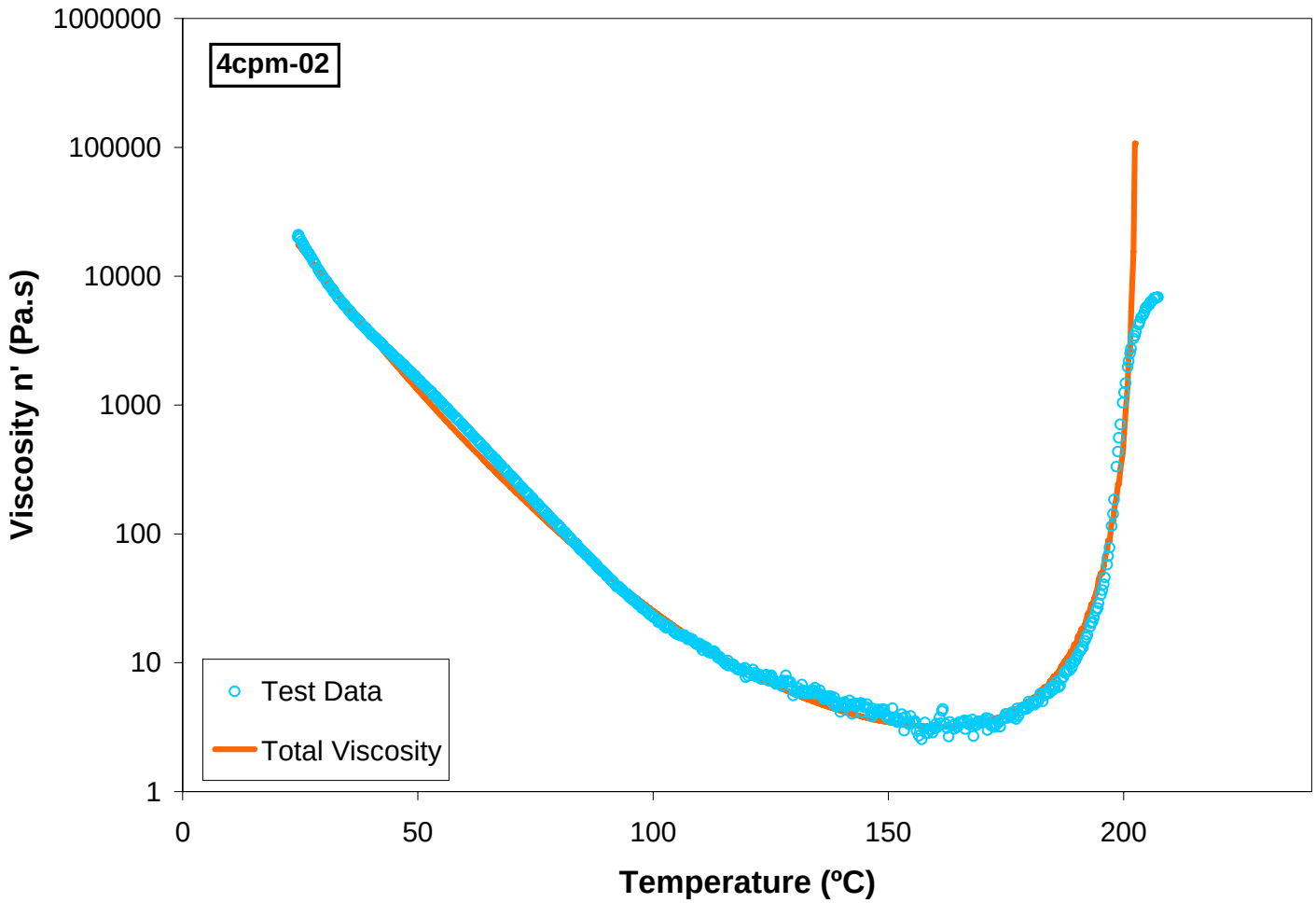
Dynamic Tests – 3cpm-2



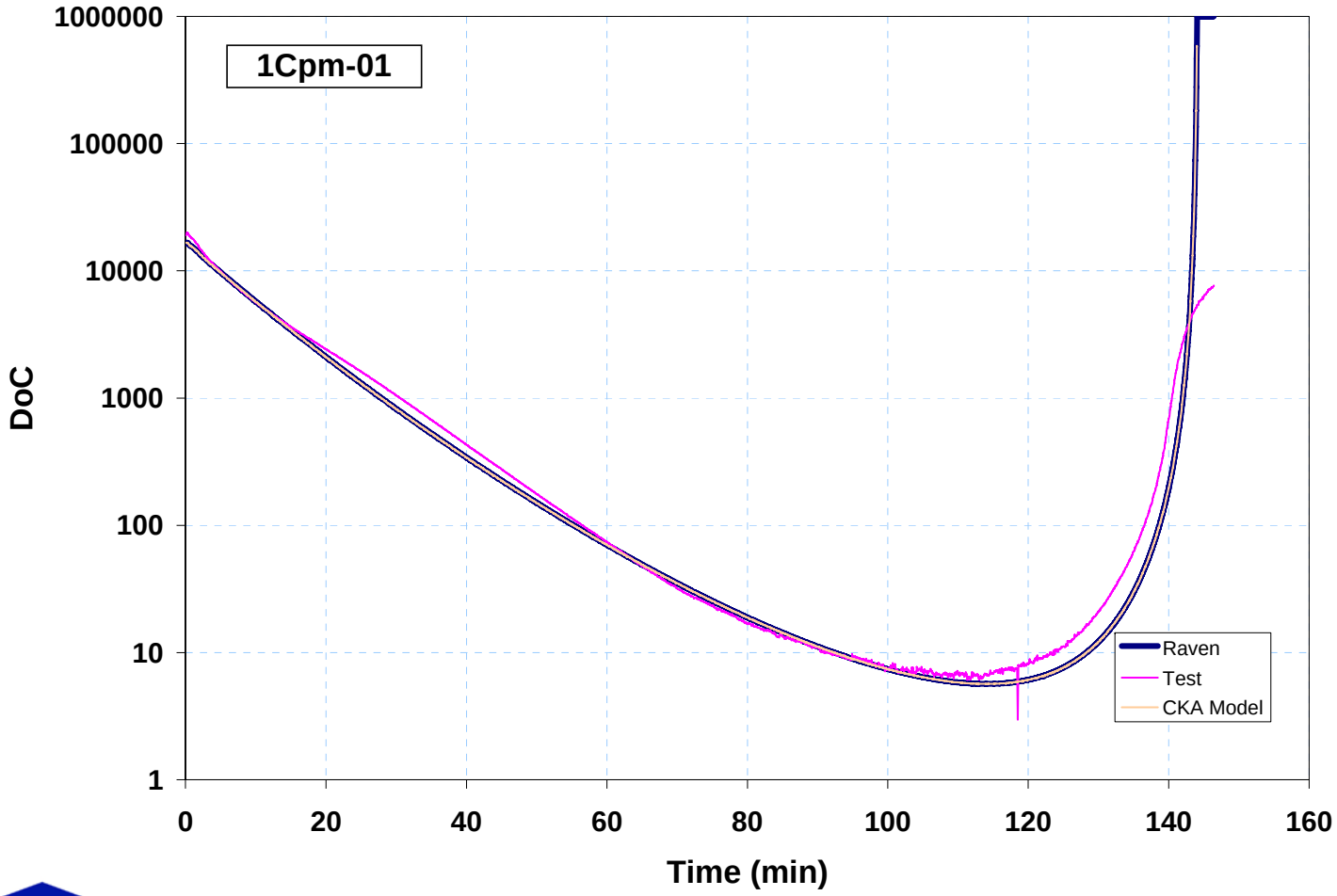
Dynamic Tests – 4cpm-1



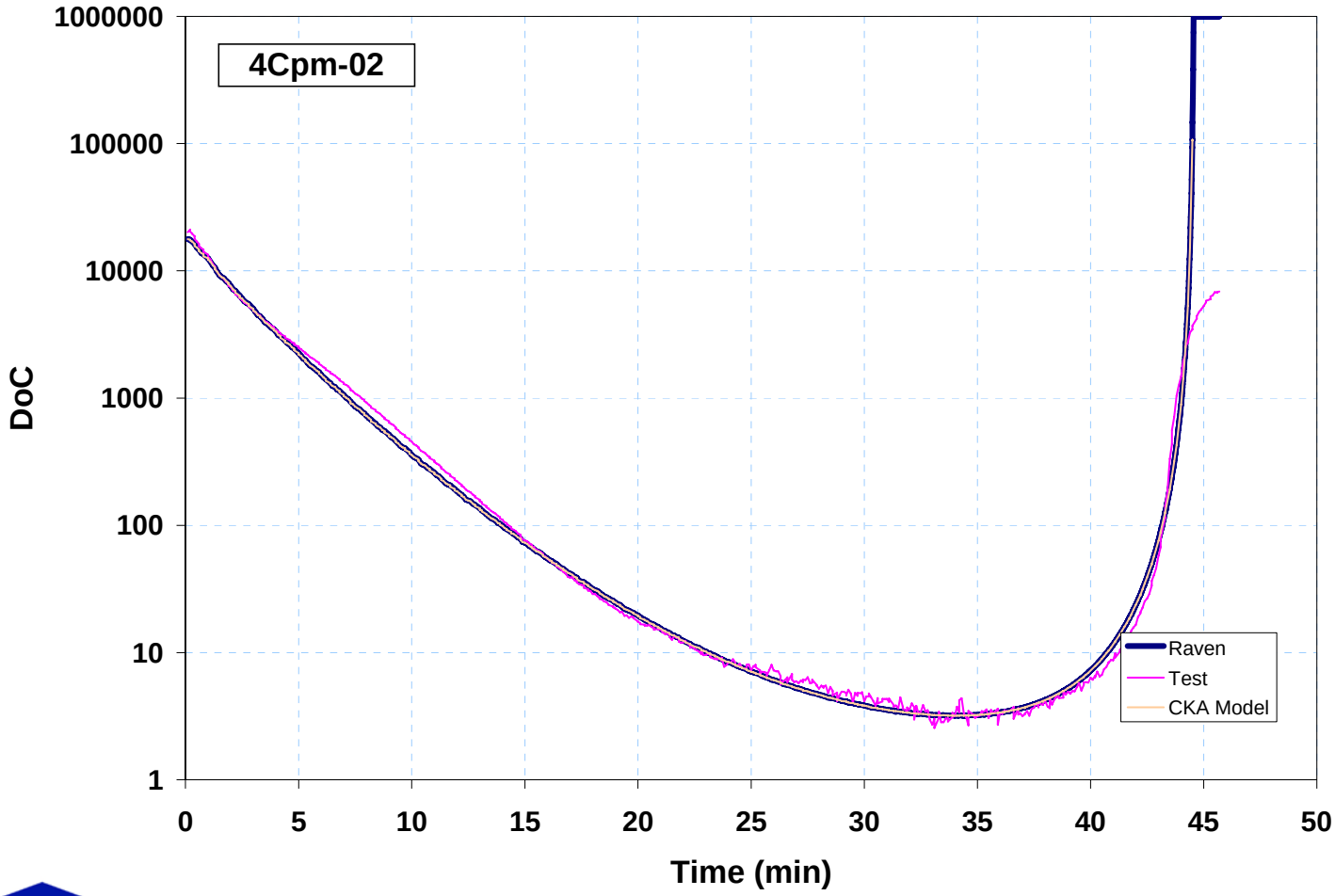
Dynamic Tests – 4cpm-2



Material Model Verification – 1Cpm-1



Material Model Verification – 4Cpm-2



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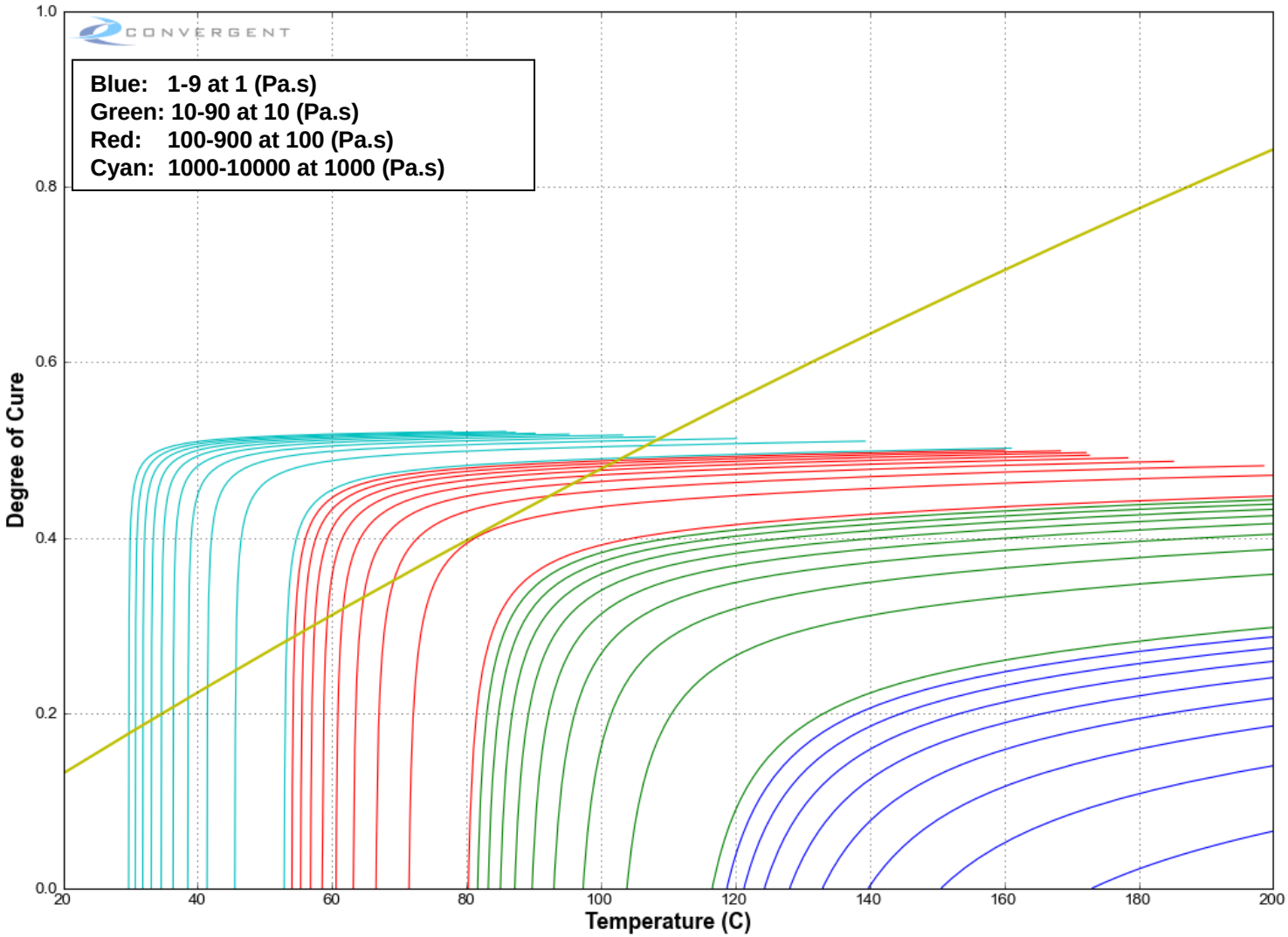
Resin Viscosity



Heat Capacity (Cp) Model

Nomenclature

Cp	Heat Capacity (J/gC)

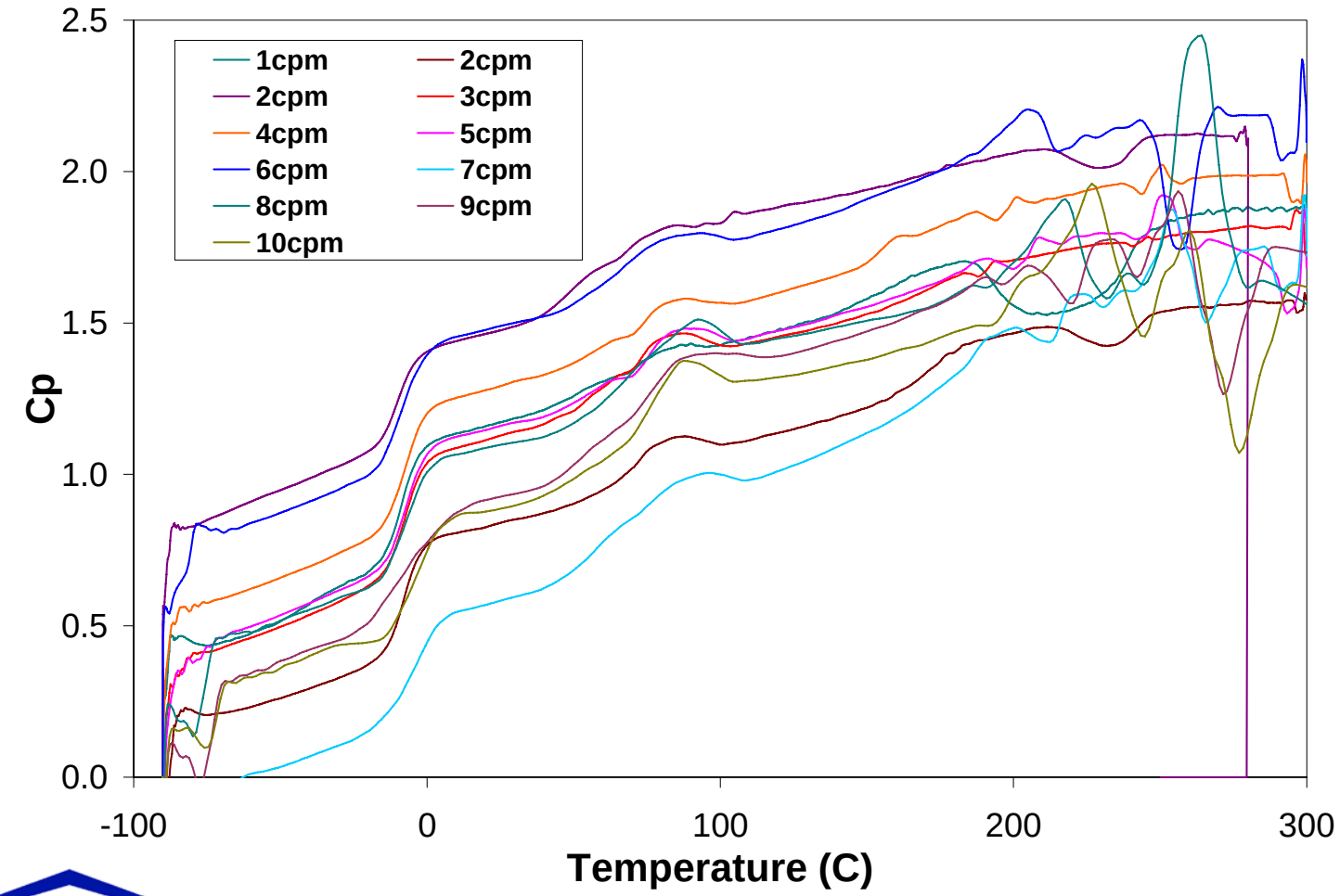
Cp Measurement

- The DSC tests were temperature modulated to obtain a measure of the material's heat capacity, Cp.

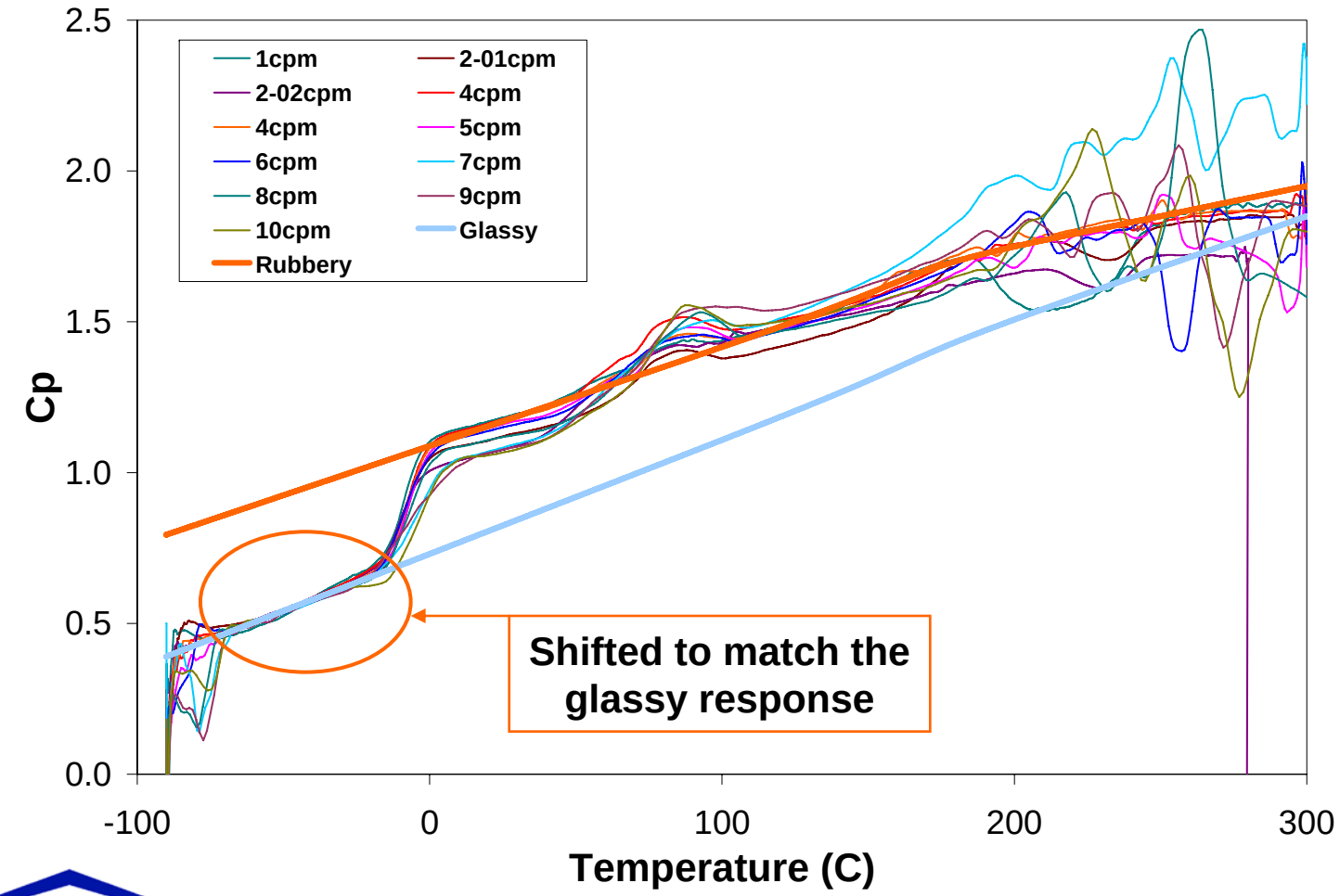
- Due to the variability associated with the measured values, some scatter was observed in the Cp response of various tests.

- The Cp responses were modified to match that of a fully cured specimen (identified as reheat tests).

Raw Data

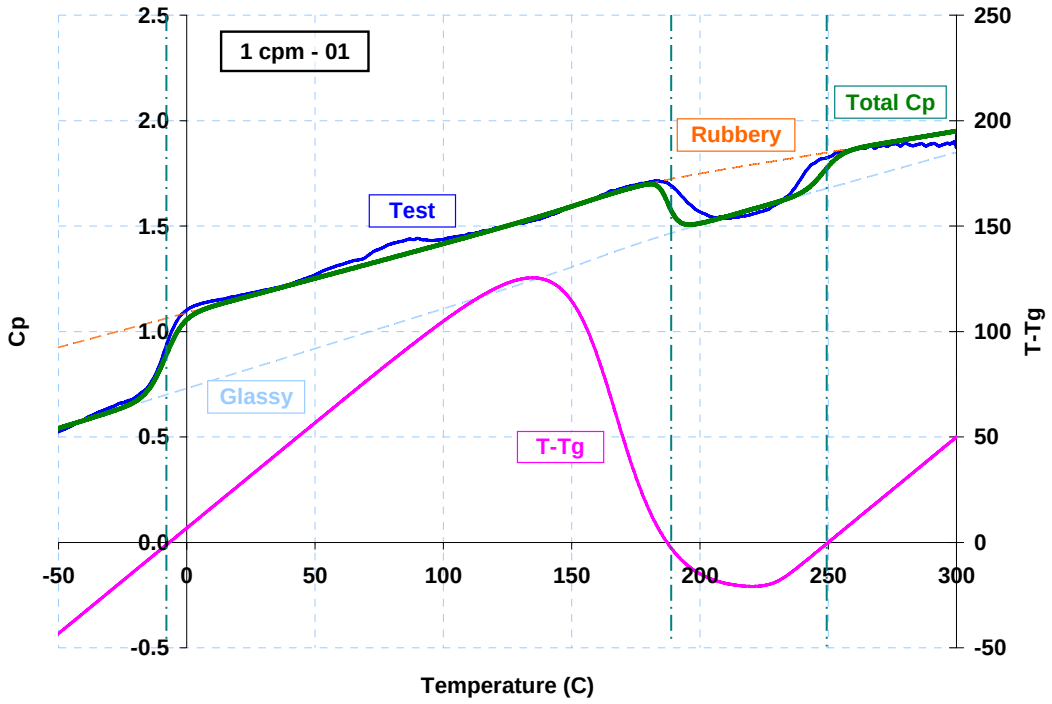

1cpm
2cpm
2cpm
3cpm
4cpm
5cpm
6cpm
7cpm
8cpm
9cpm
10cpm
Cp
2.5
2.0
1.5
1.0
0.5
0.0
-100
0
100
200
300
Temperature (C)

Raw Data – Shifted



Cp Model Basics

- Glassy and rubbery Cp were assumed to be functions of temperature and DoC
- Cp was assumed to transition between the glassy and rubbery responses with changes in T-Tg



Cp Model

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

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

Glassy or rubbery at ***x***: $C_{p_i} = (1-x)C_{p_{i0}} + xC_{p_{i\infty}}$

Total Cp response: $C_p = C_{p_r} + \dfrac{C_{p_g} - C_{p_r}}{1 + e^{k[(T-T_g)-\Delta T_c]}}$

Glassy

$s_{g0} = 0.003775$ $s_{g\infty} = 0.0034$

$c_{g0} = 0.73$ $c_{g\infty} = 0.83$

Rubbery

$s_{r0} = 0.00327$ $s_{g\infty} = 0.002$

$c_{r0} = 1.088$ $c_{g\infty} = 1.35$

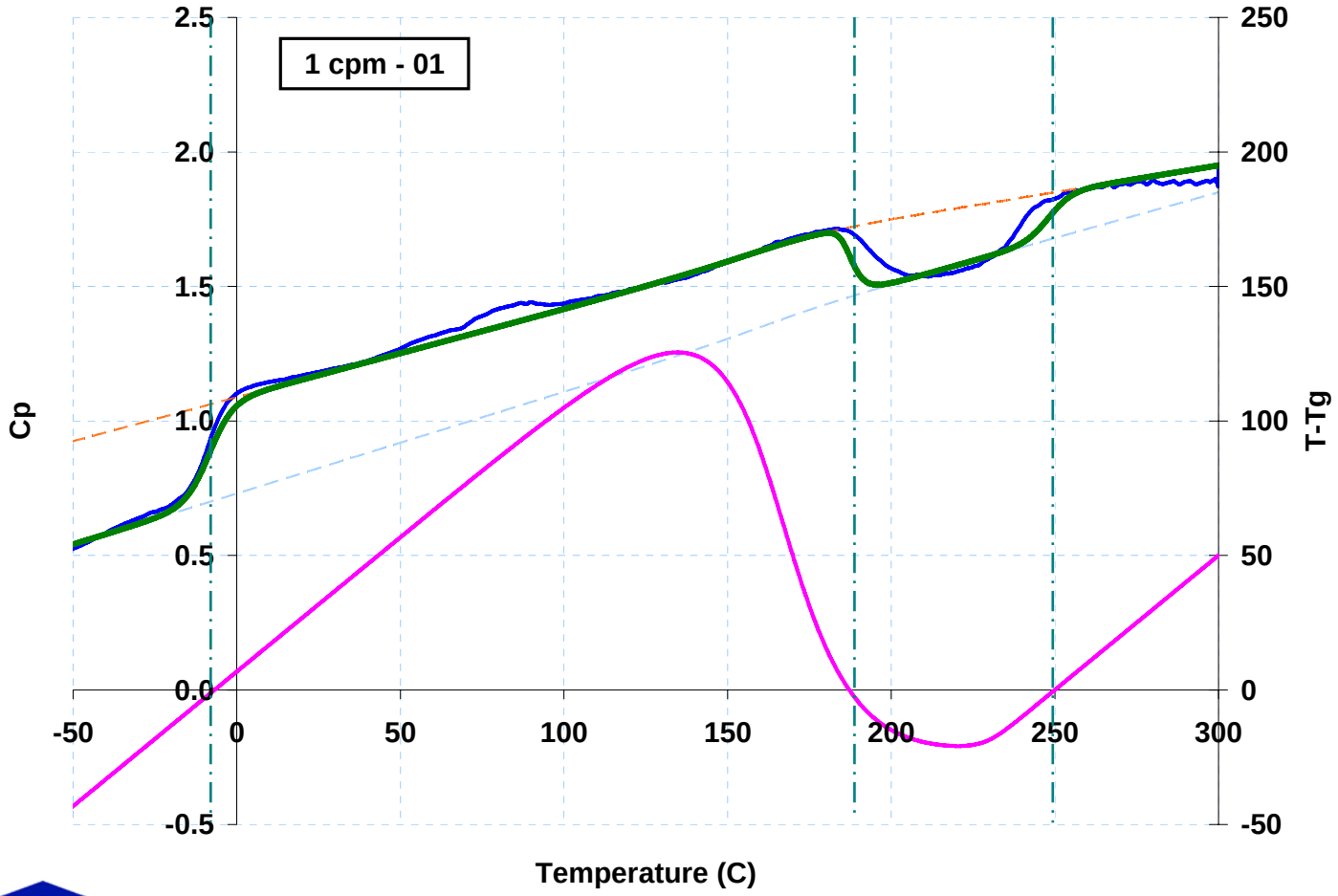
Other parameters

$k = 0.278$

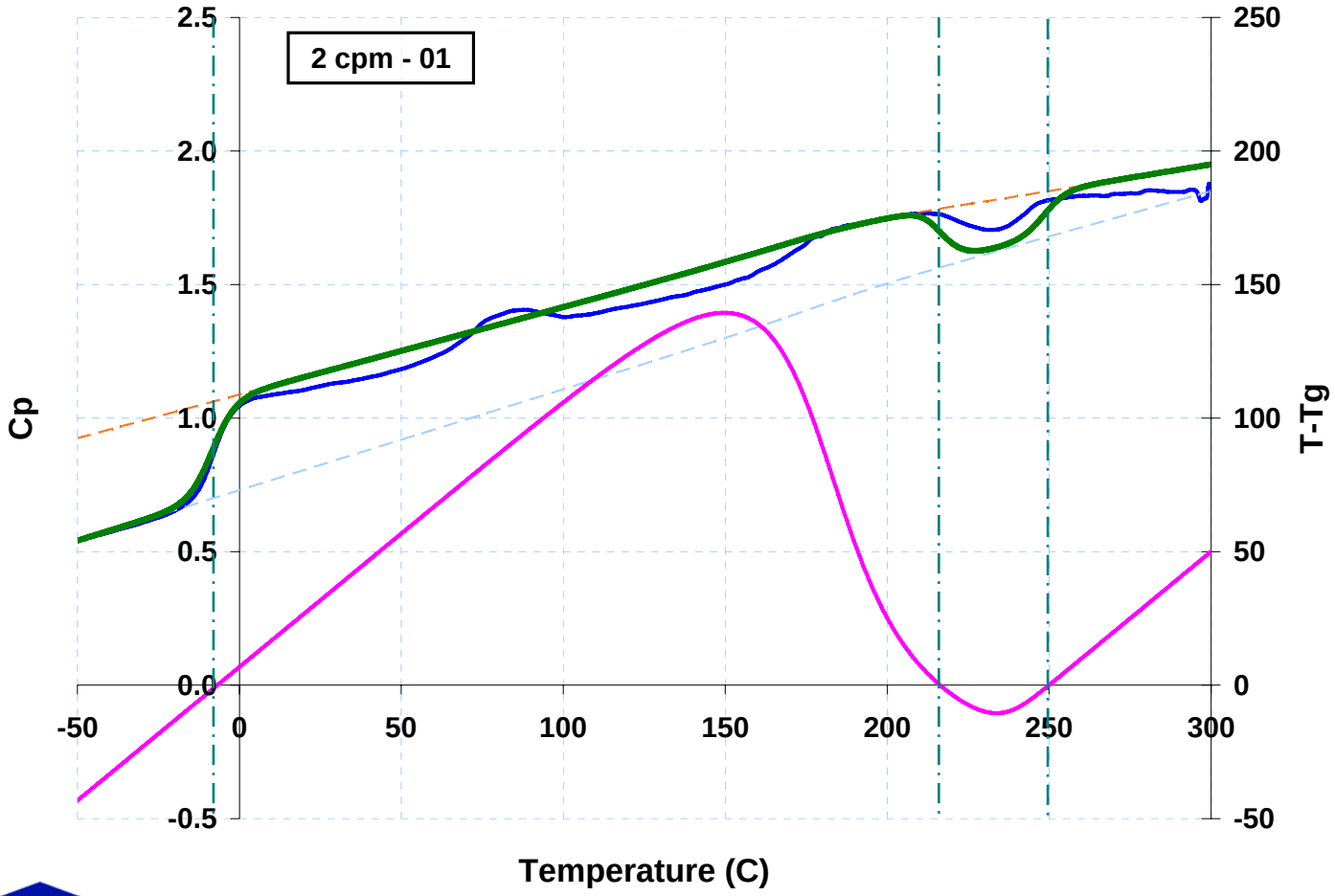
$\Delta T_c = -1.5$

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

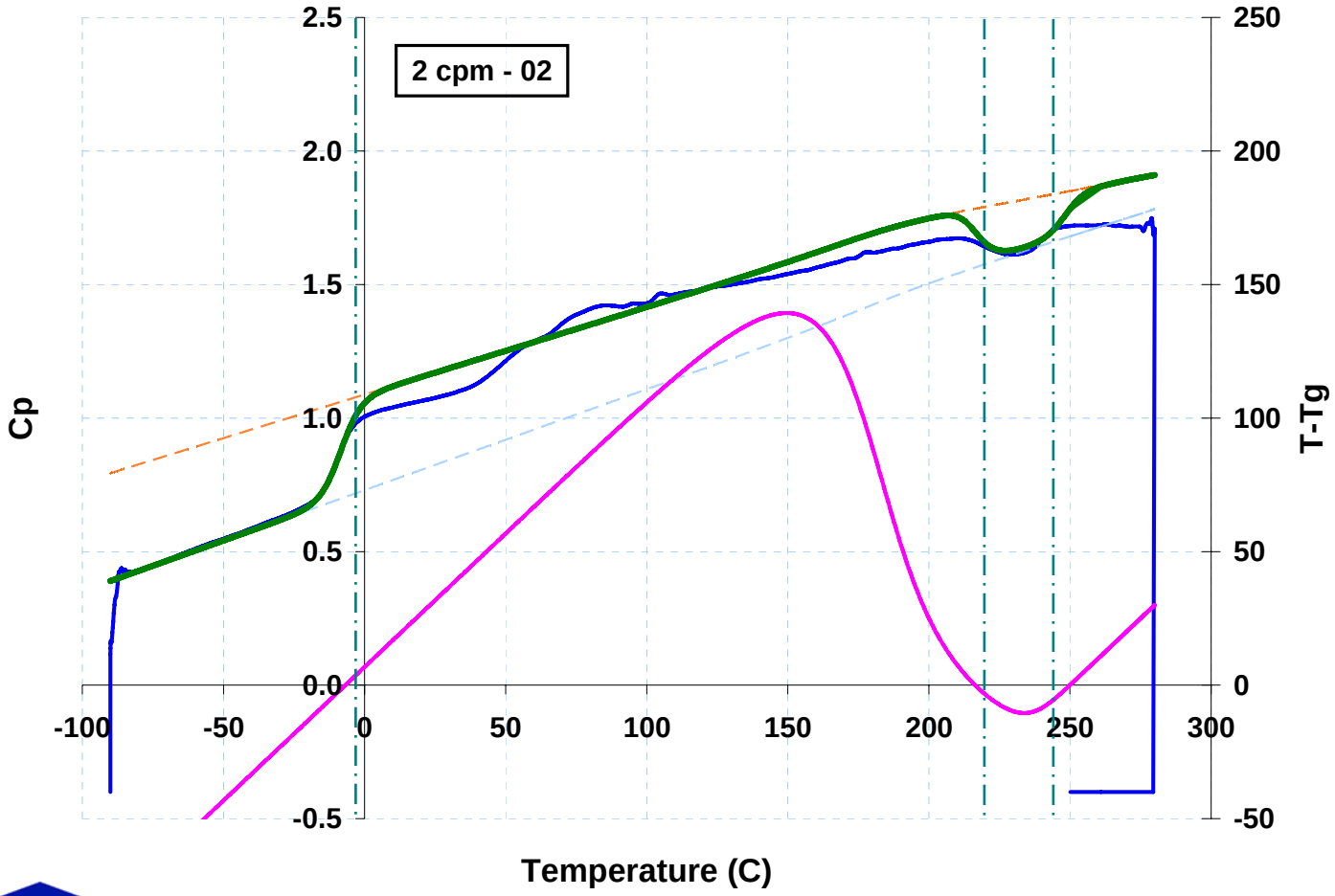
Dynamic Tests – 1cpm



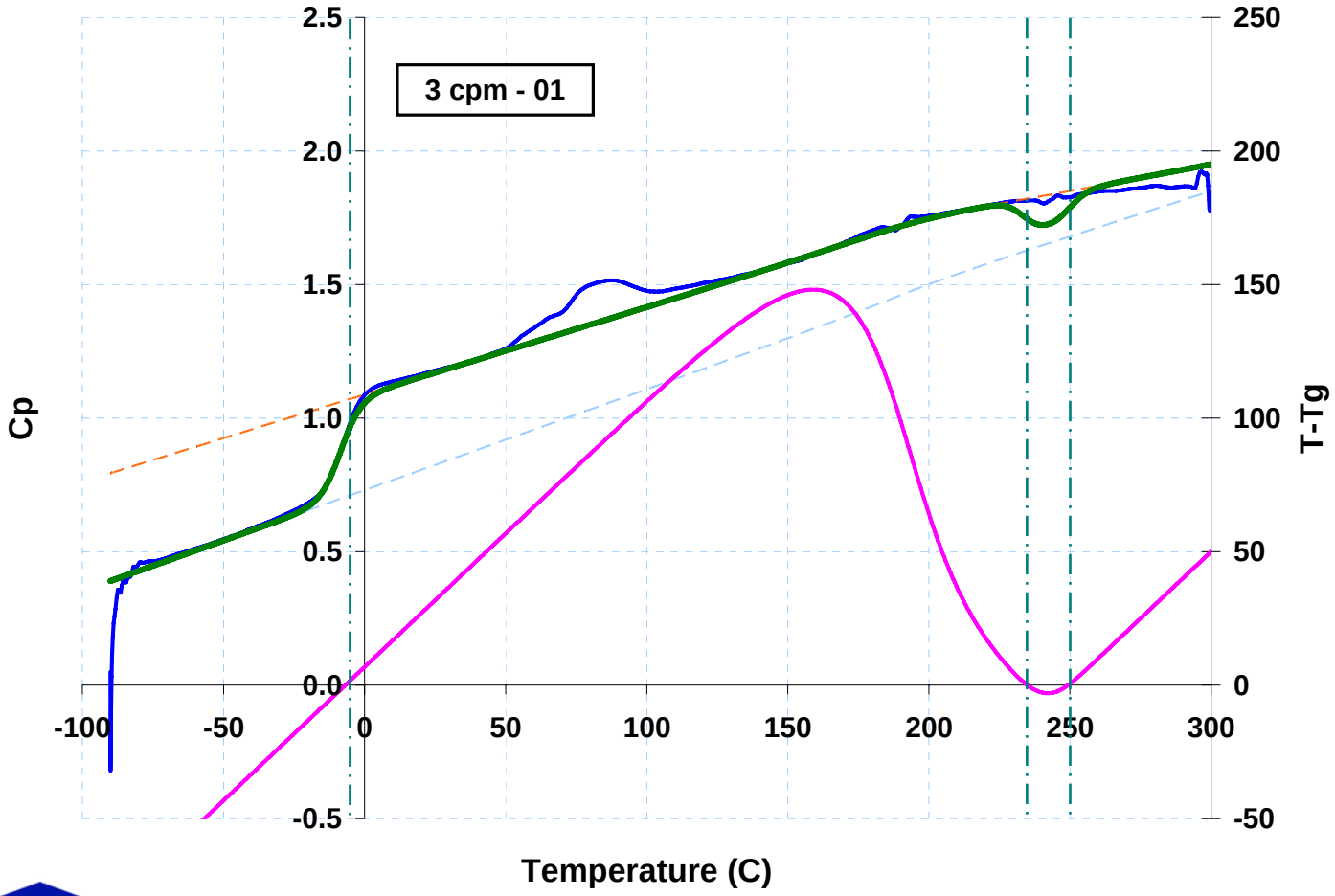
Dynamic Tests – 2cpm-1



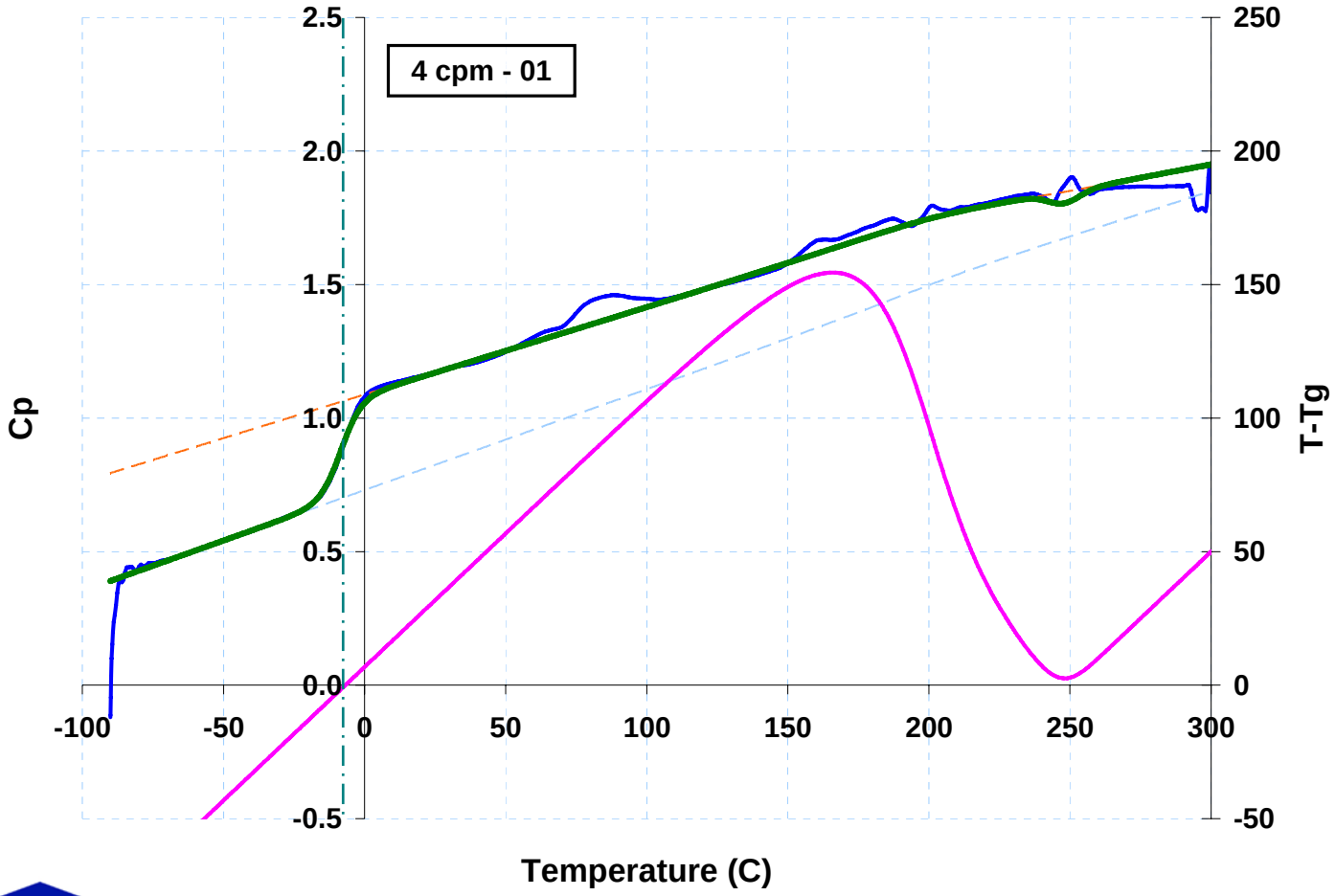
Dynamic Tests – 2cpm-2



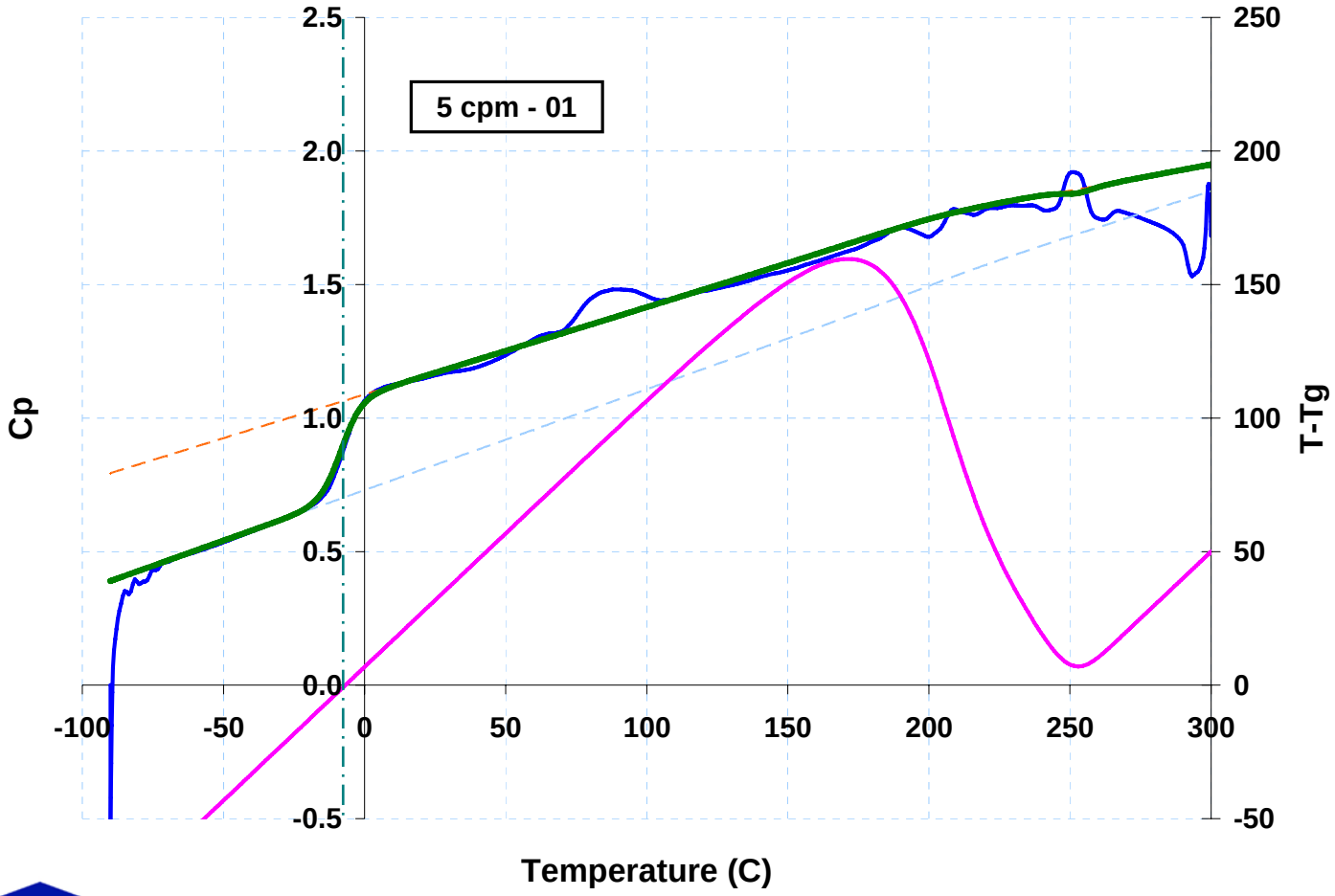
Dynamic Tests – 3cpm



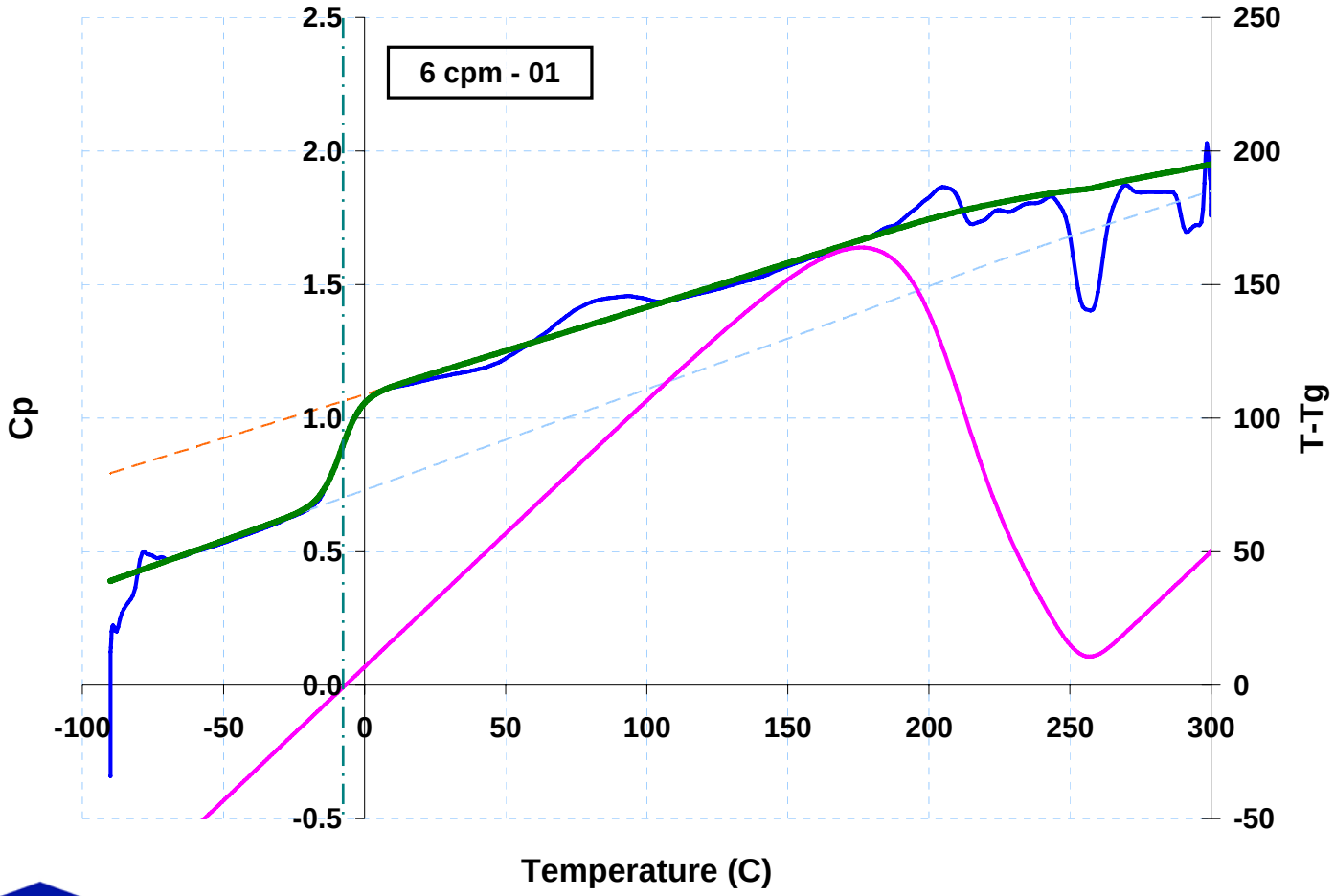
Dynamic Tests – 4cpm



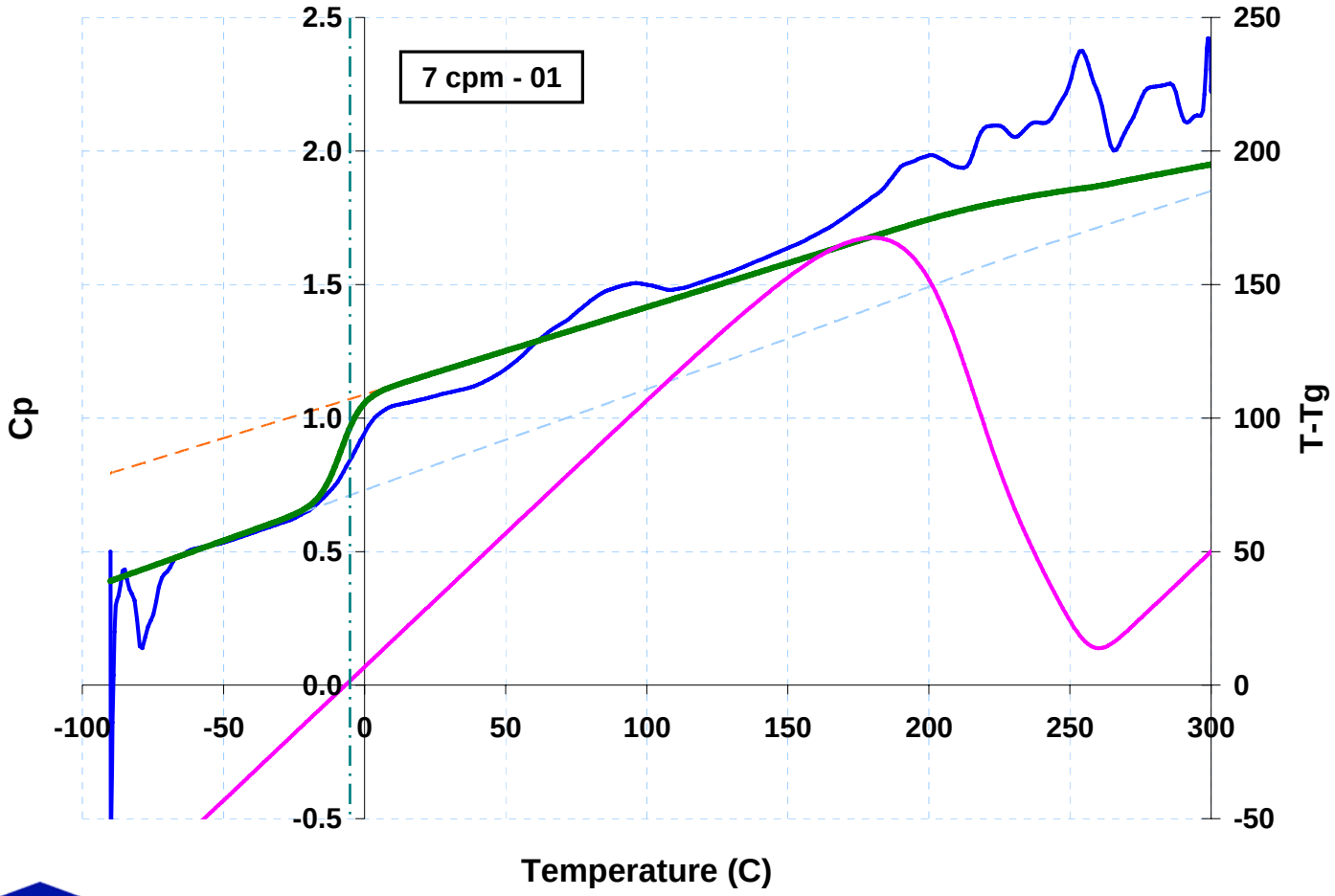
Dynamic Tests – 5cpm



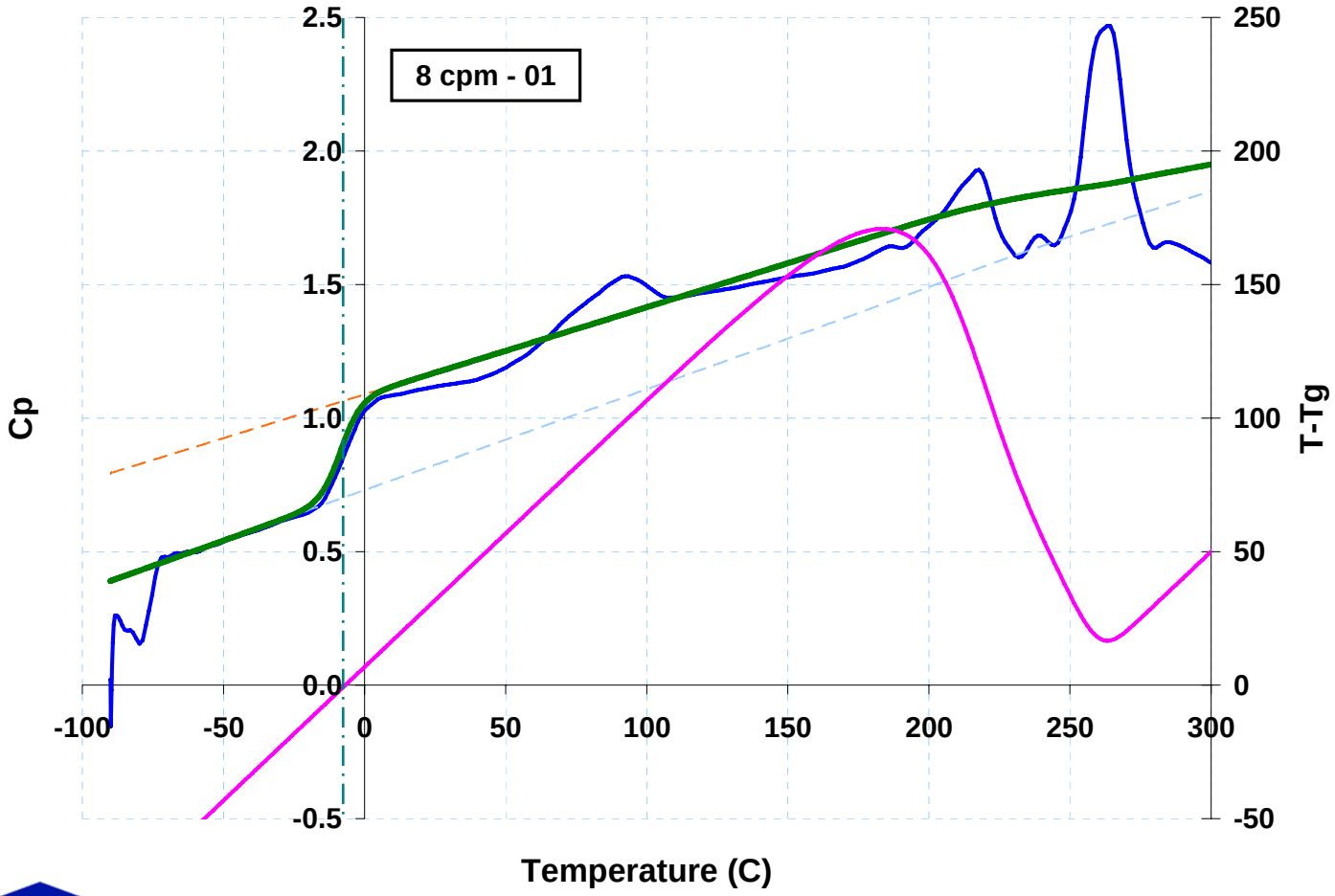
Dynamic Tests – 6cpm



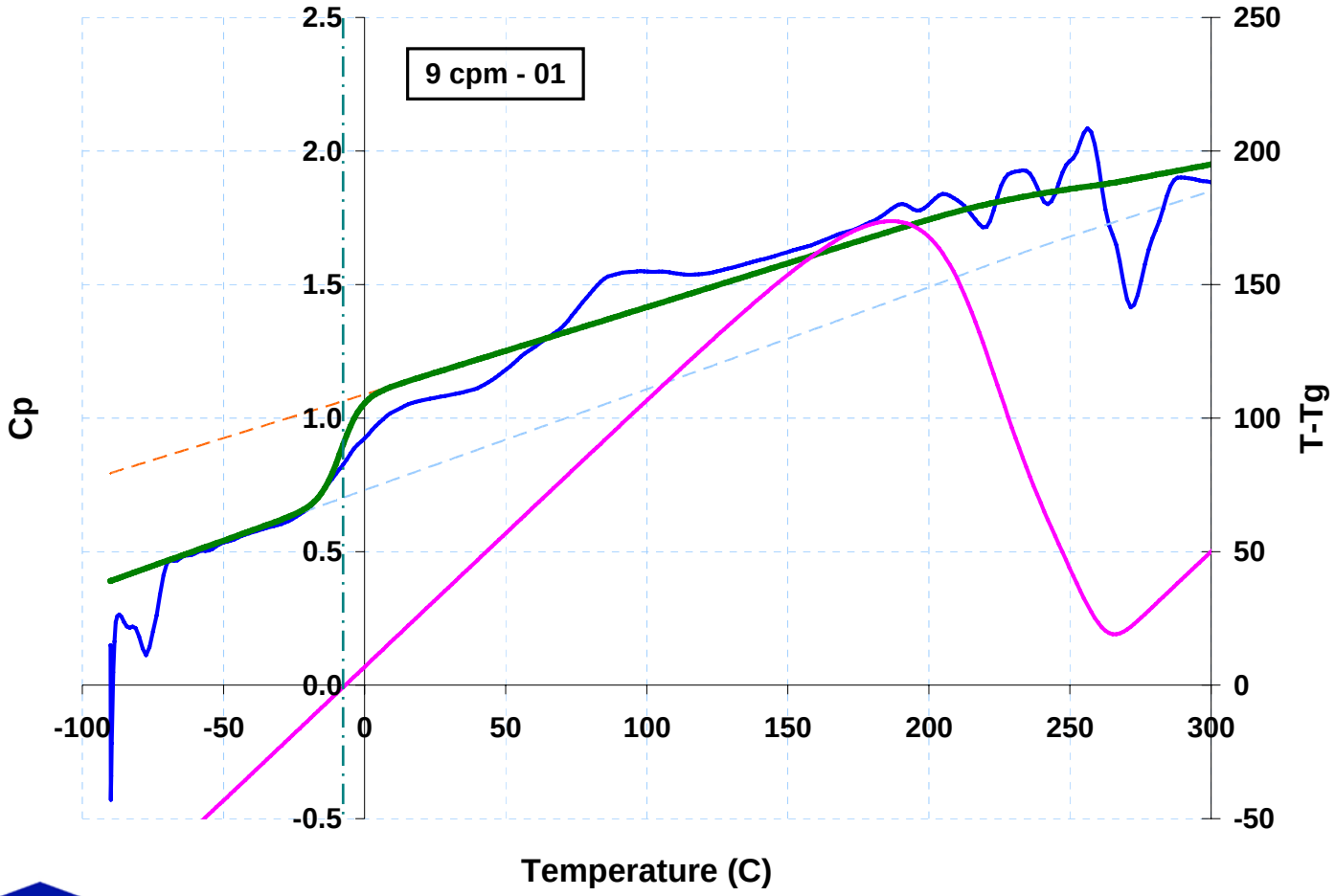
Dynamic Tests – 7cpm



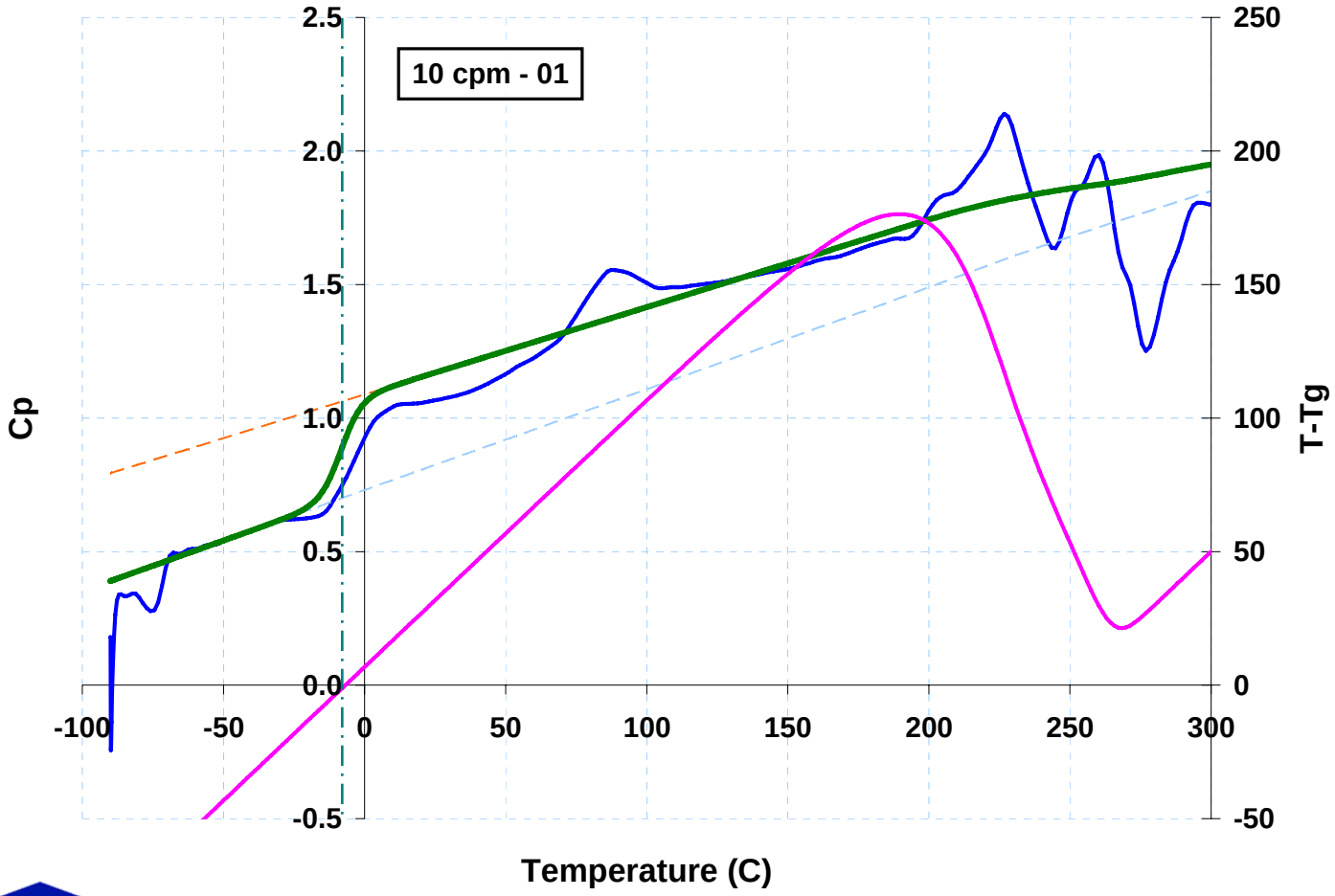
Dynamic Tests – 8cpm



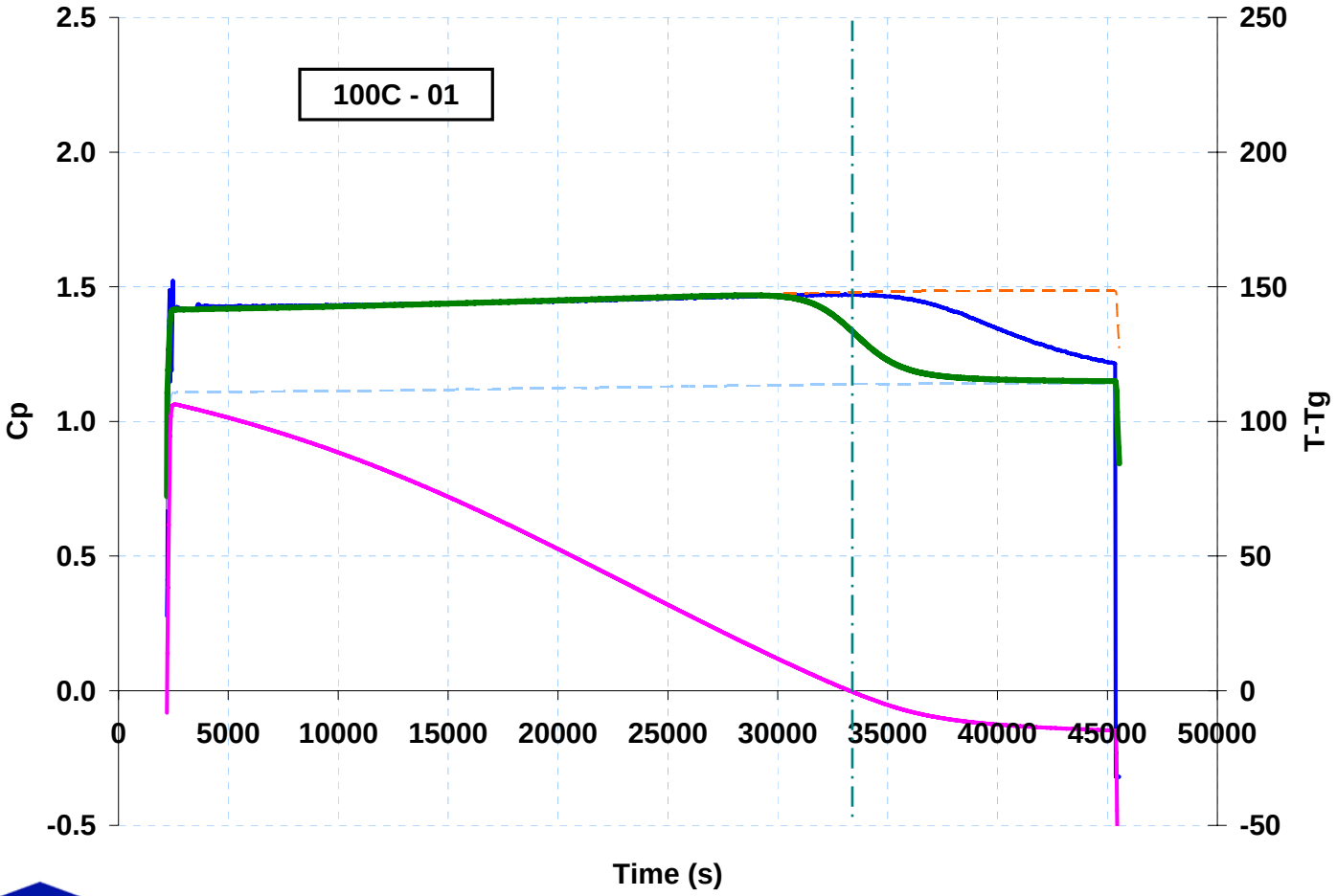
Dynamic Tests – 9cpm



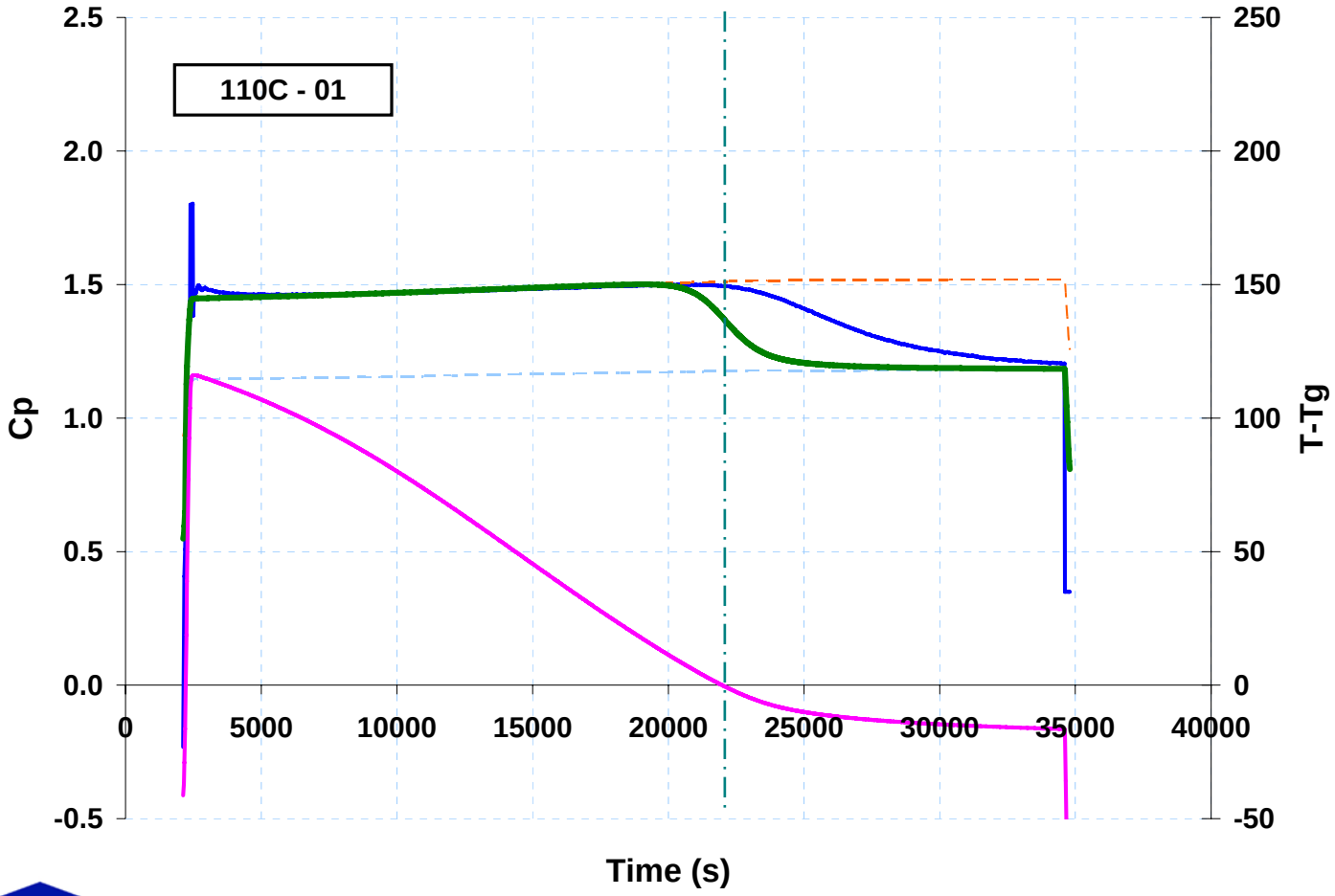
Dynamic Tests – 10cpm



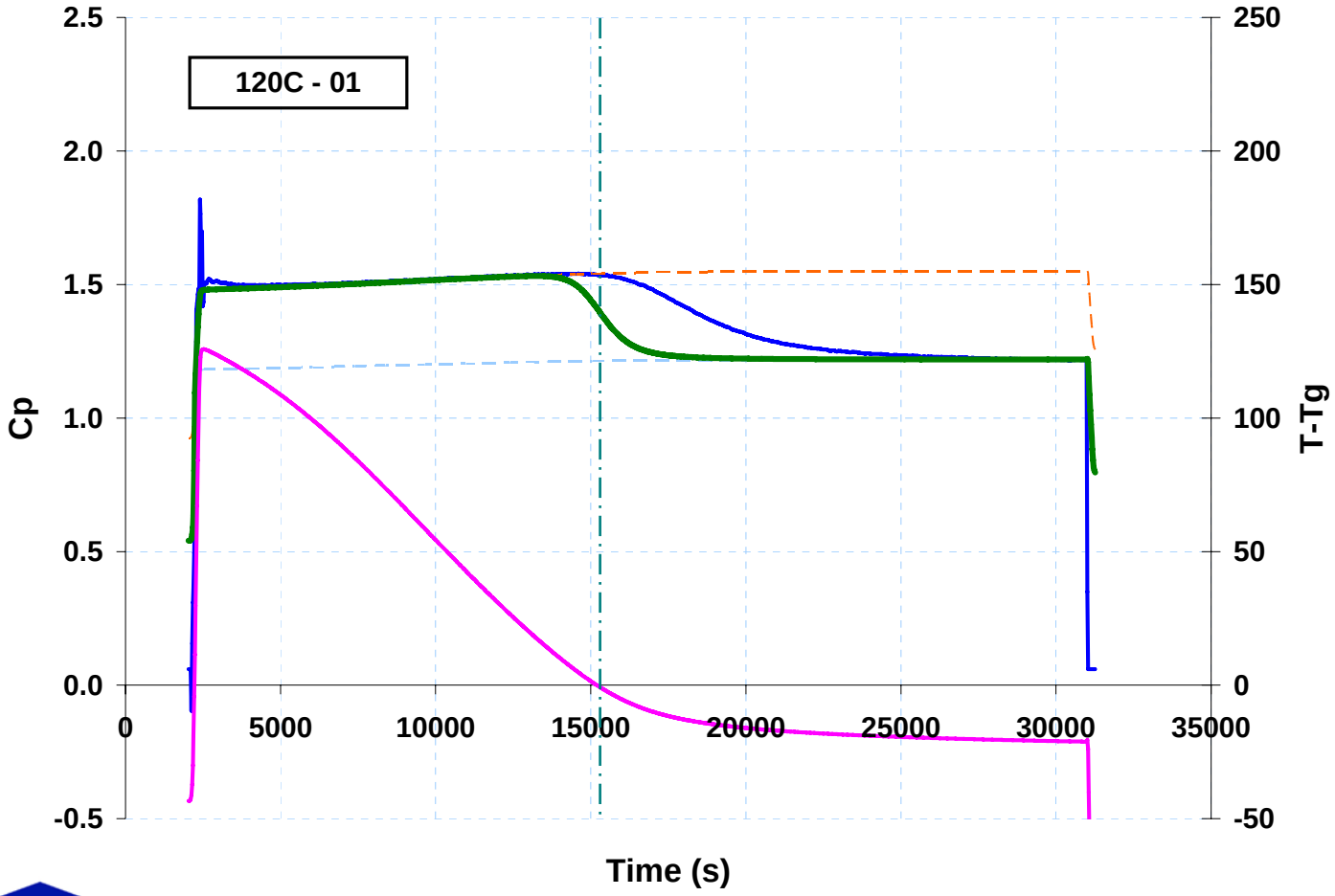
Isothermal Tests – 100C



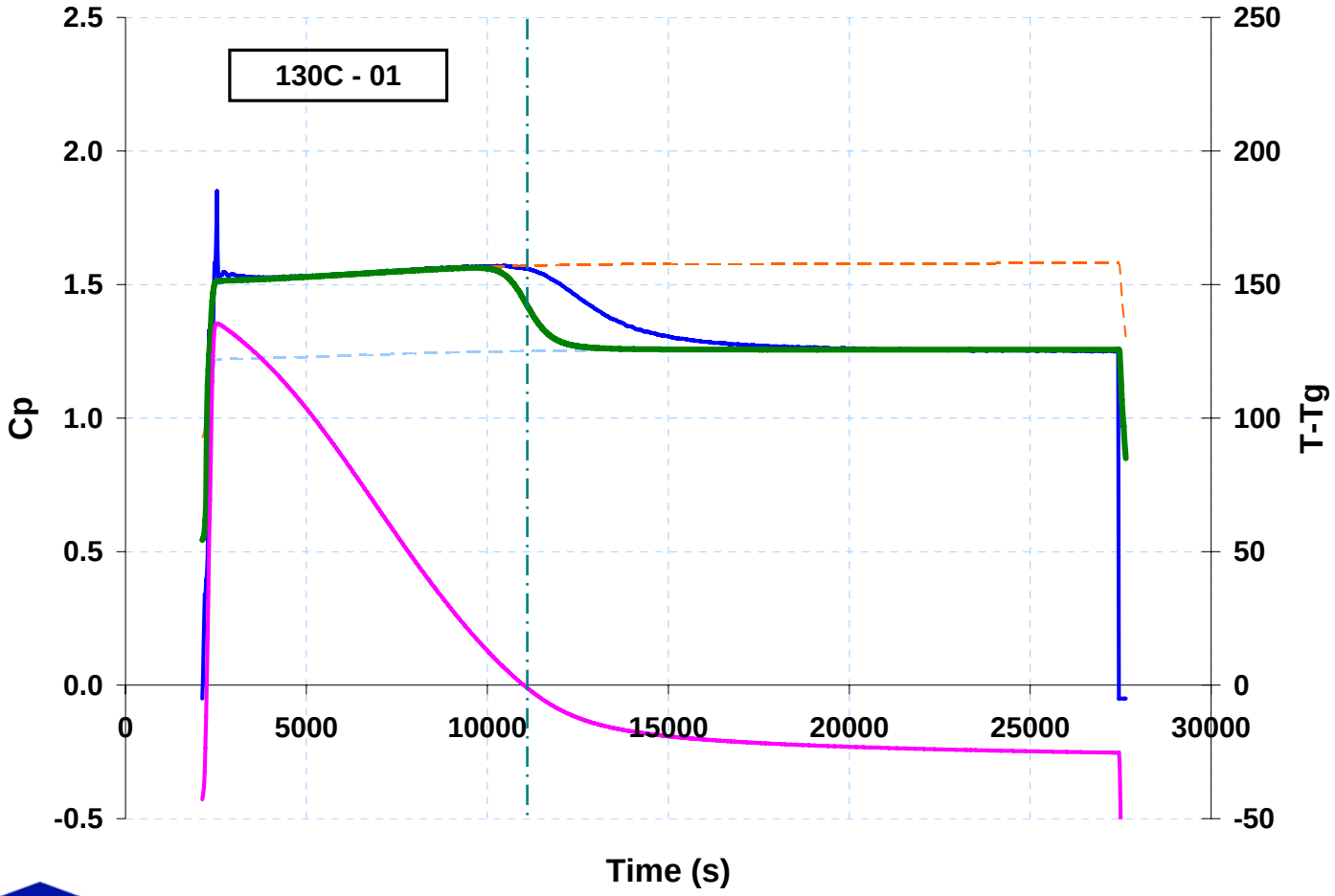
Isothermal Tests – 110C



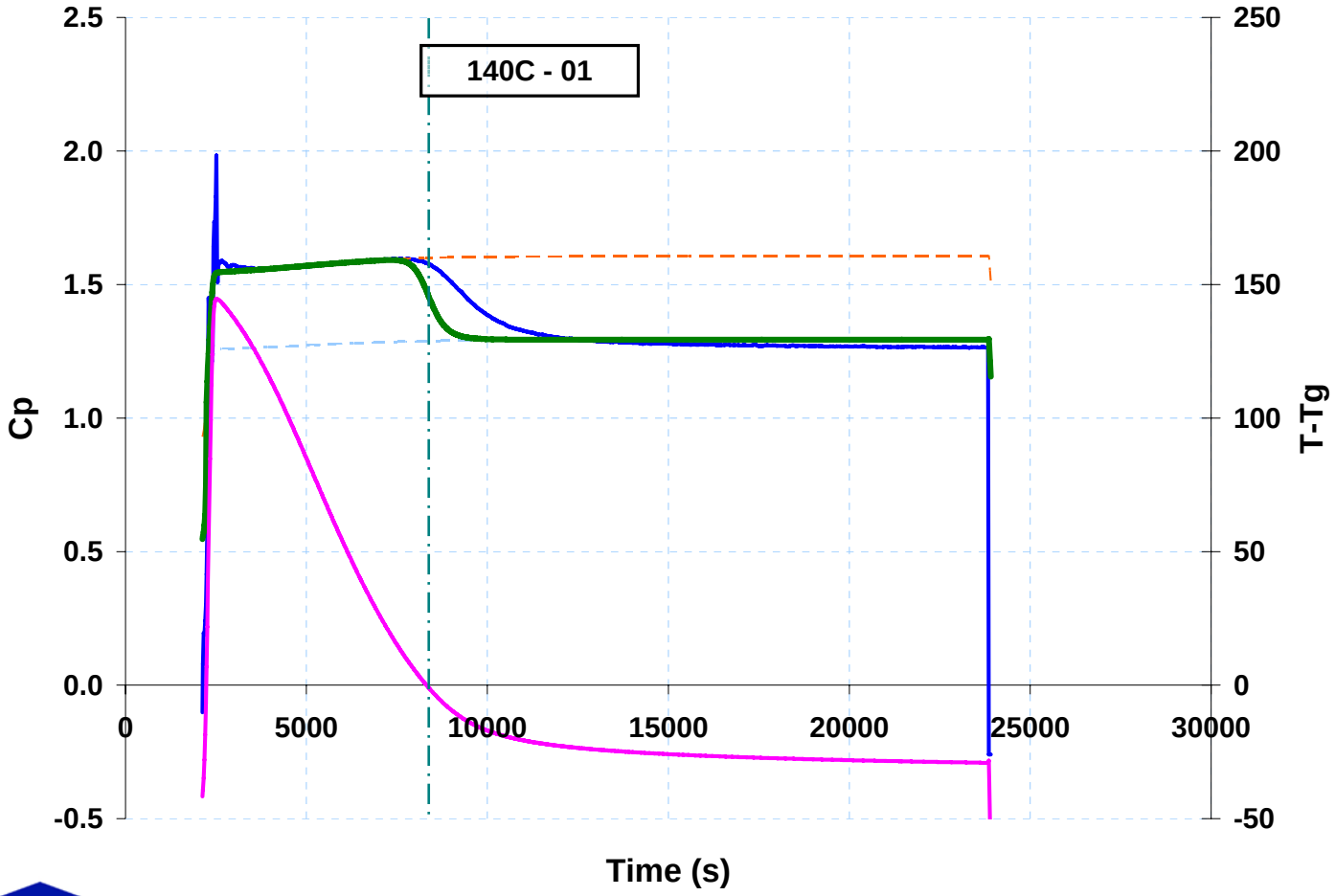
Isothermal Tests – 120C



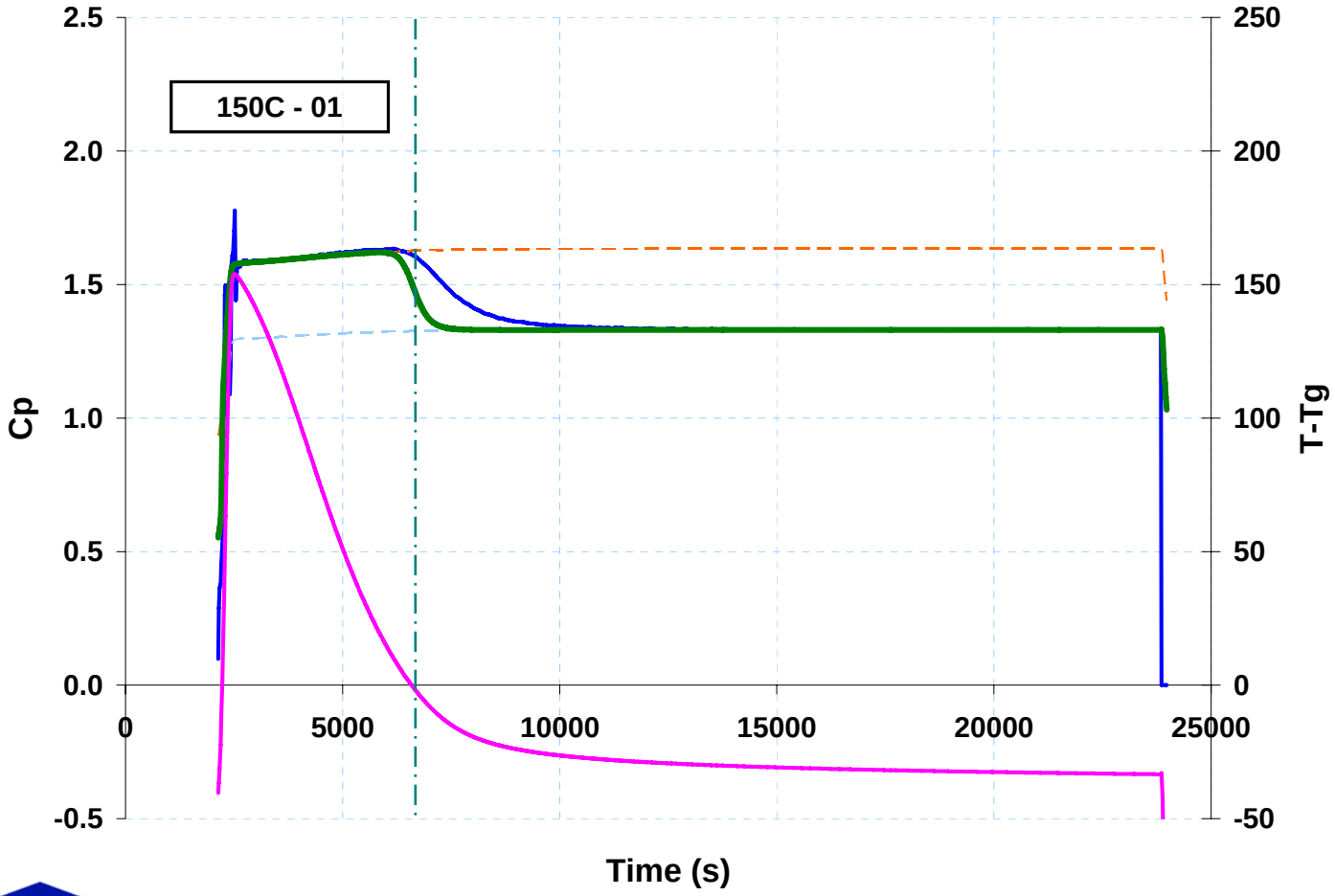
Isothermal Tests – 130C



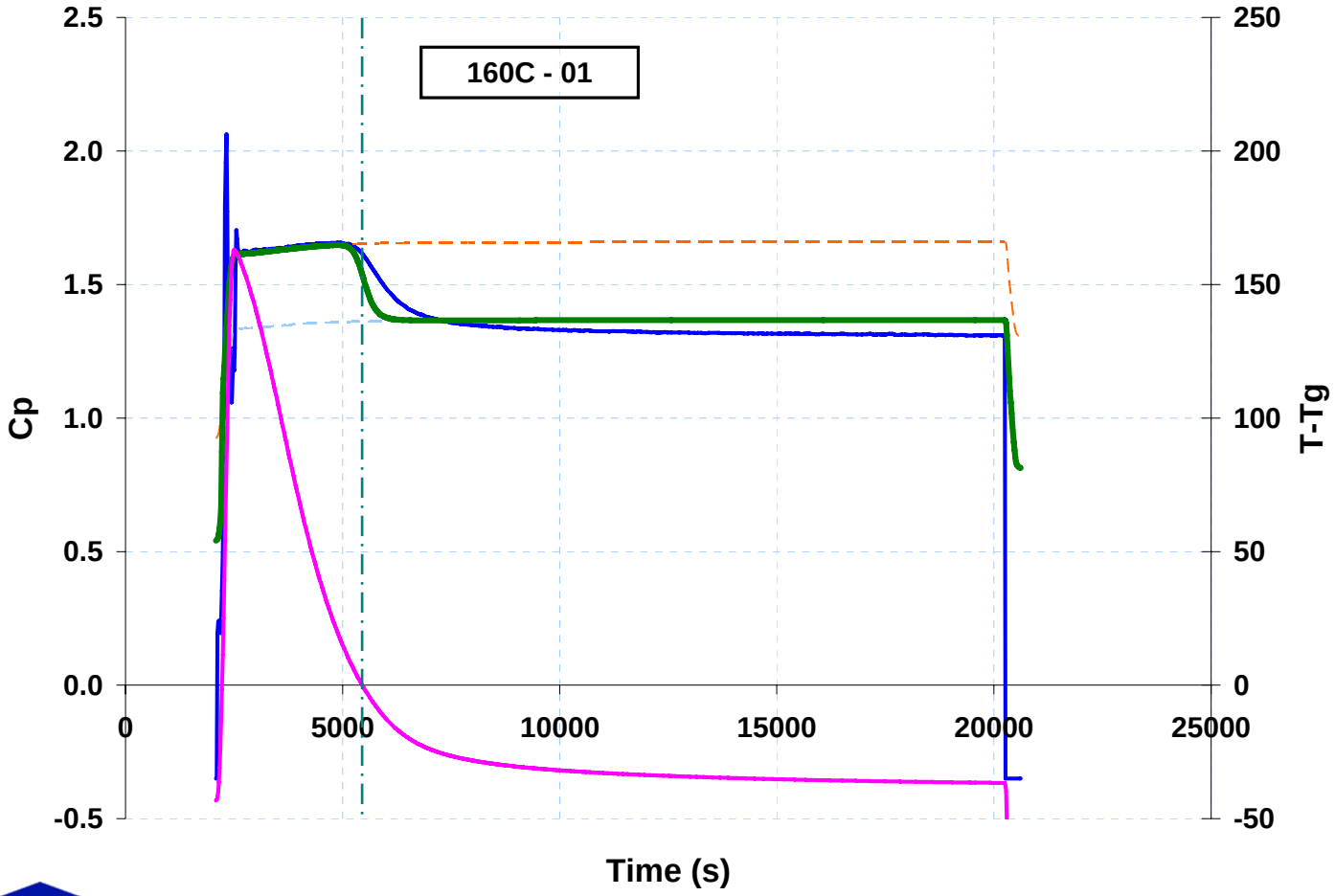
Isothermal Tests – 140C



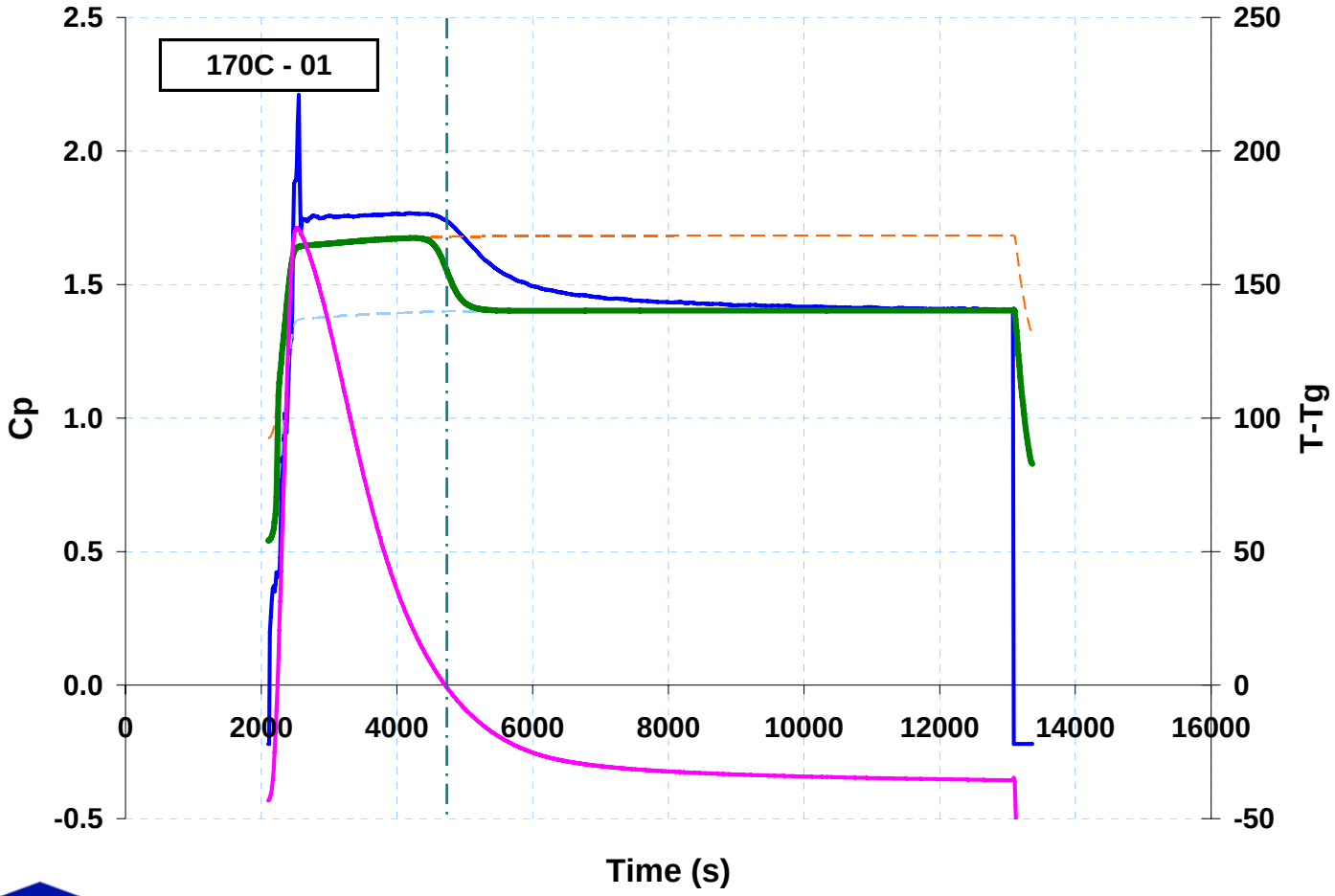
Isothermal Tests – 150C



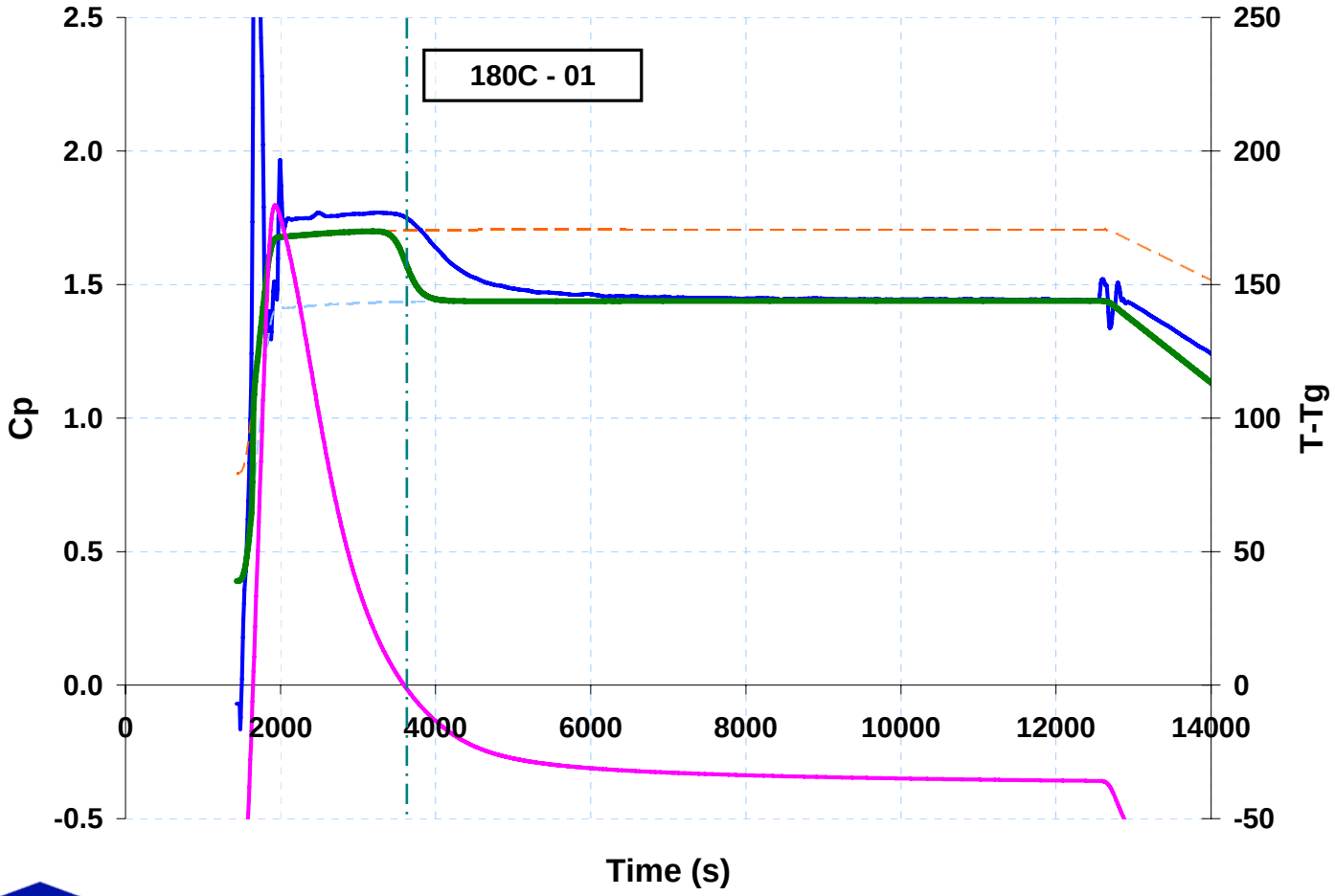
Isothermal Tests – 160C



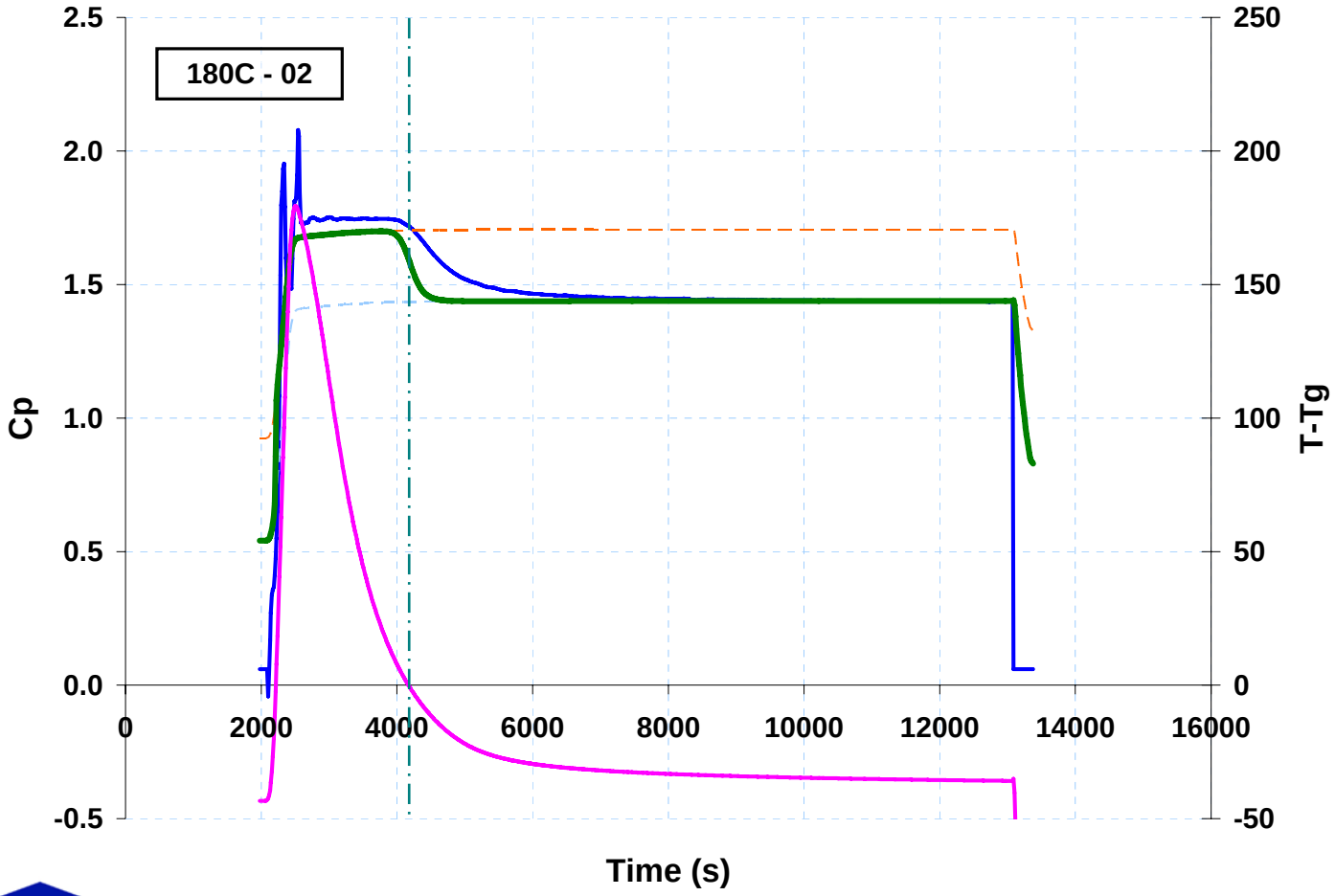
Isothermal Tests – 170C



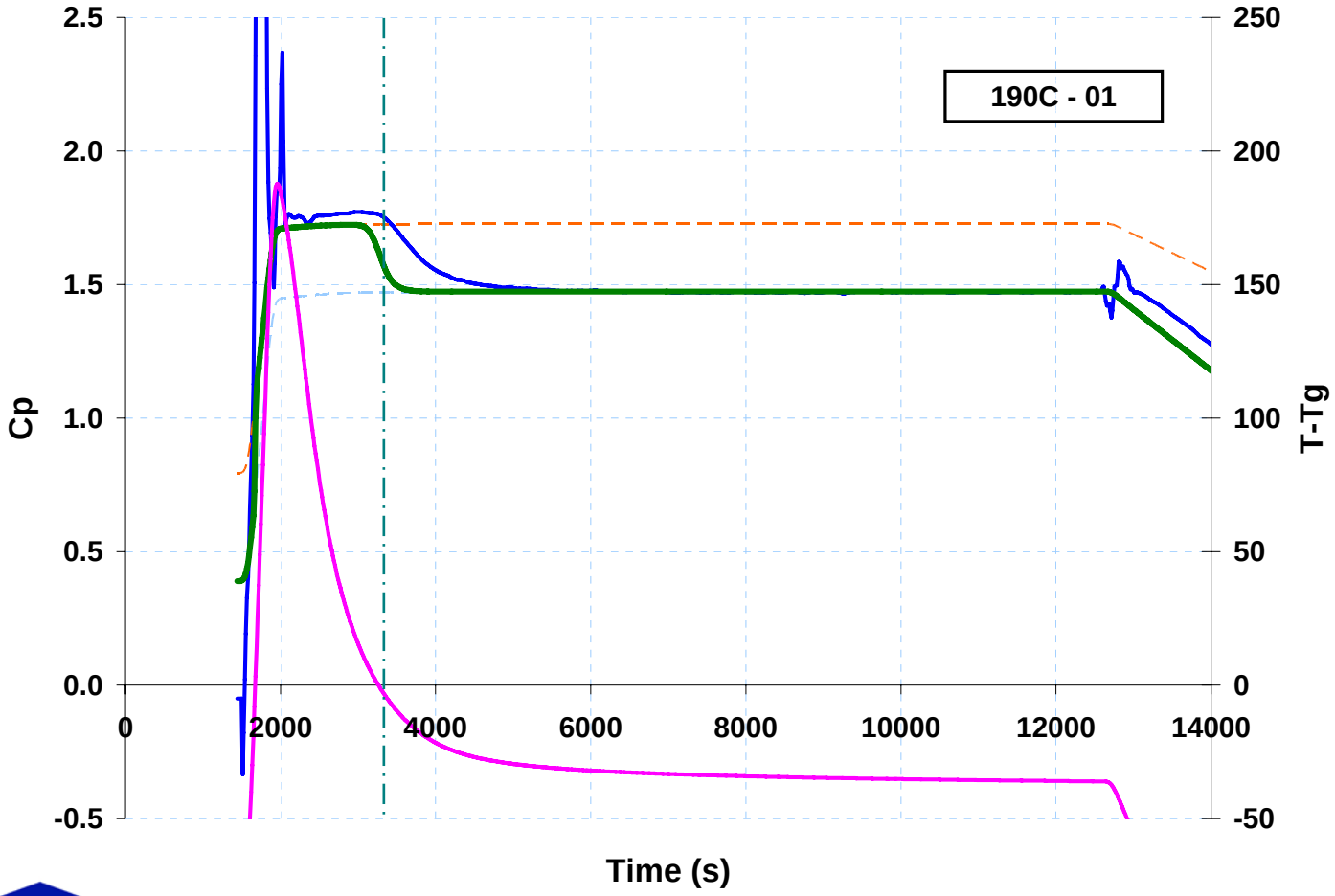
Isothermal Tests – 180C-1



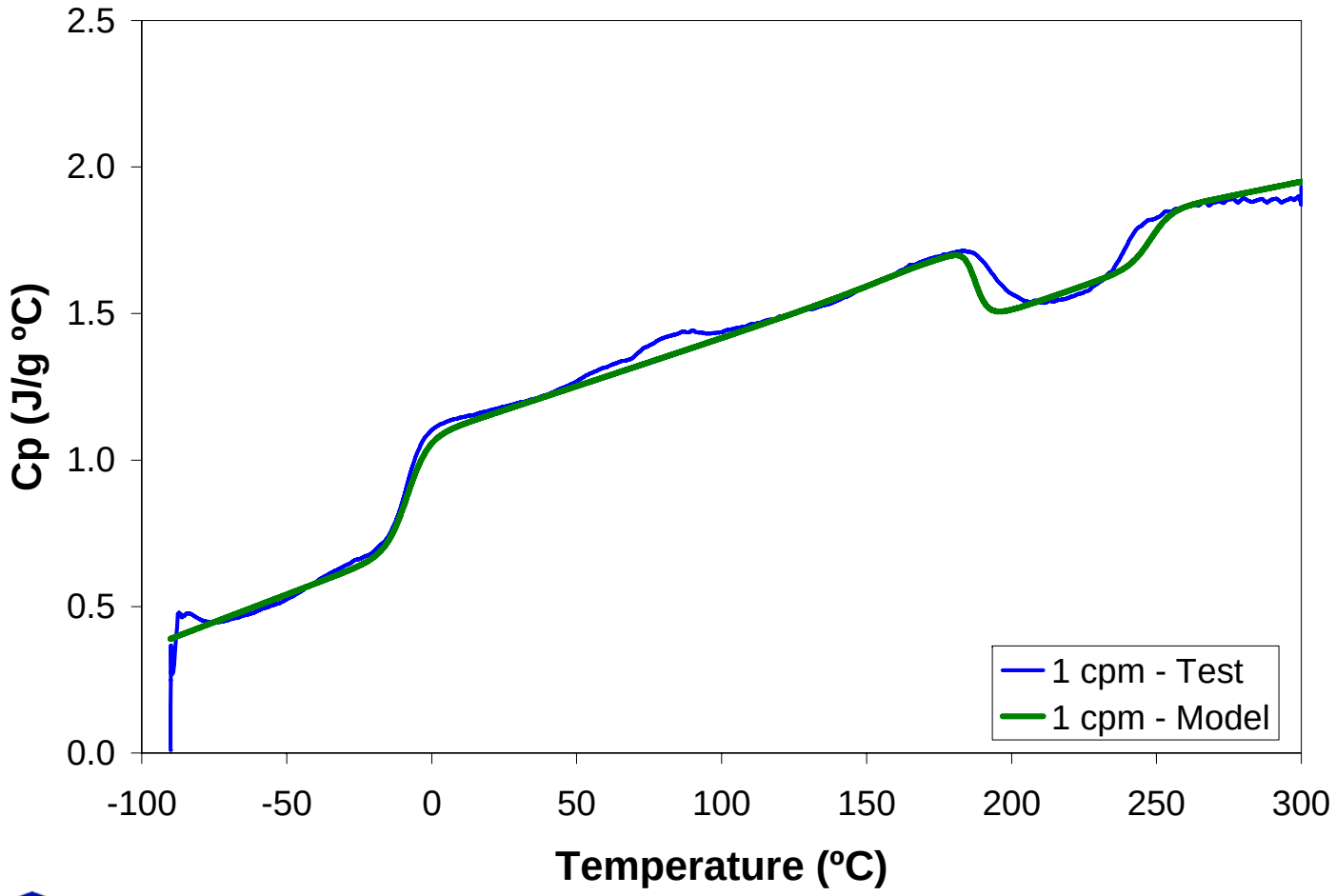
Isothermal Tests – 180C-2



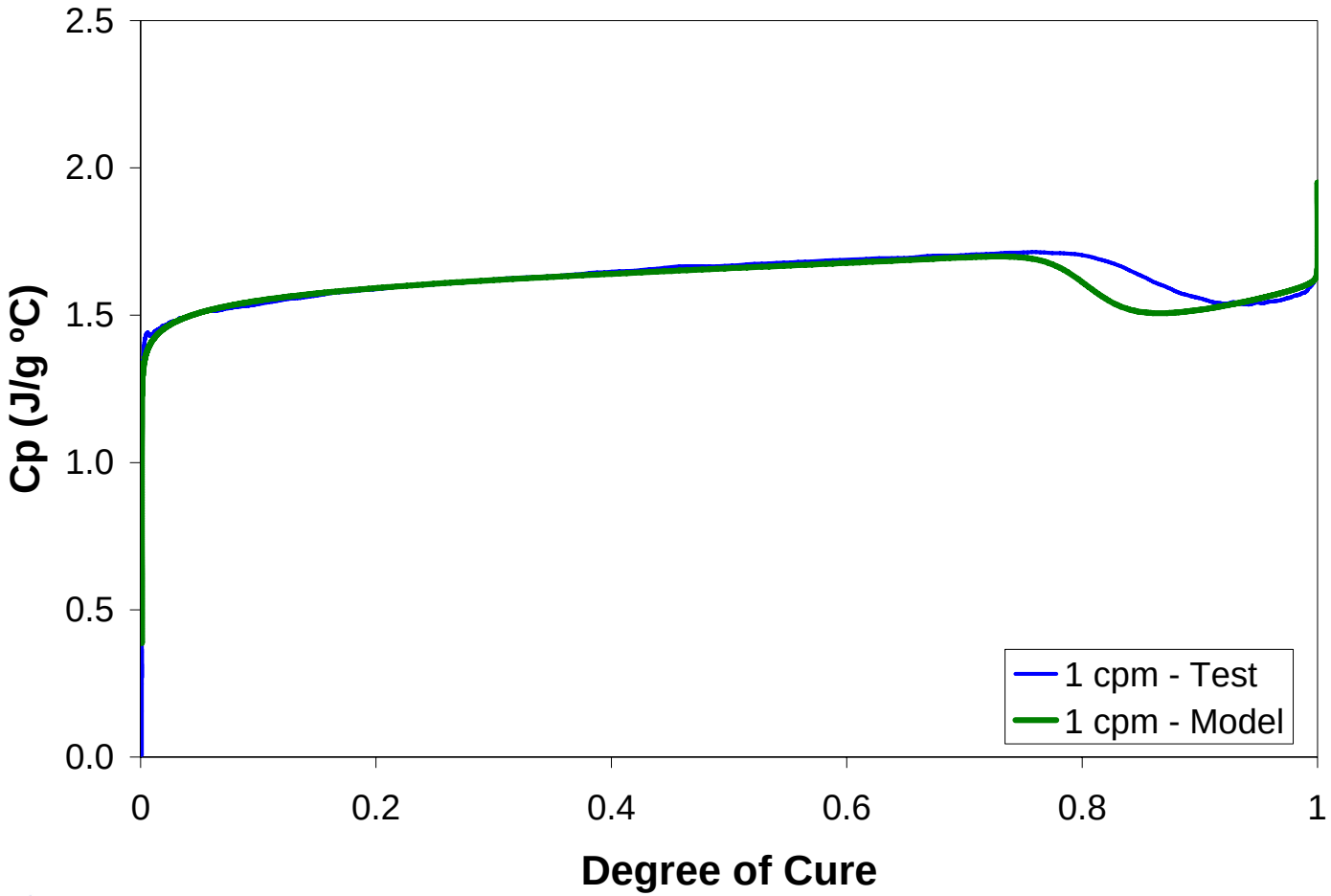
Isothermal Tests – 190C



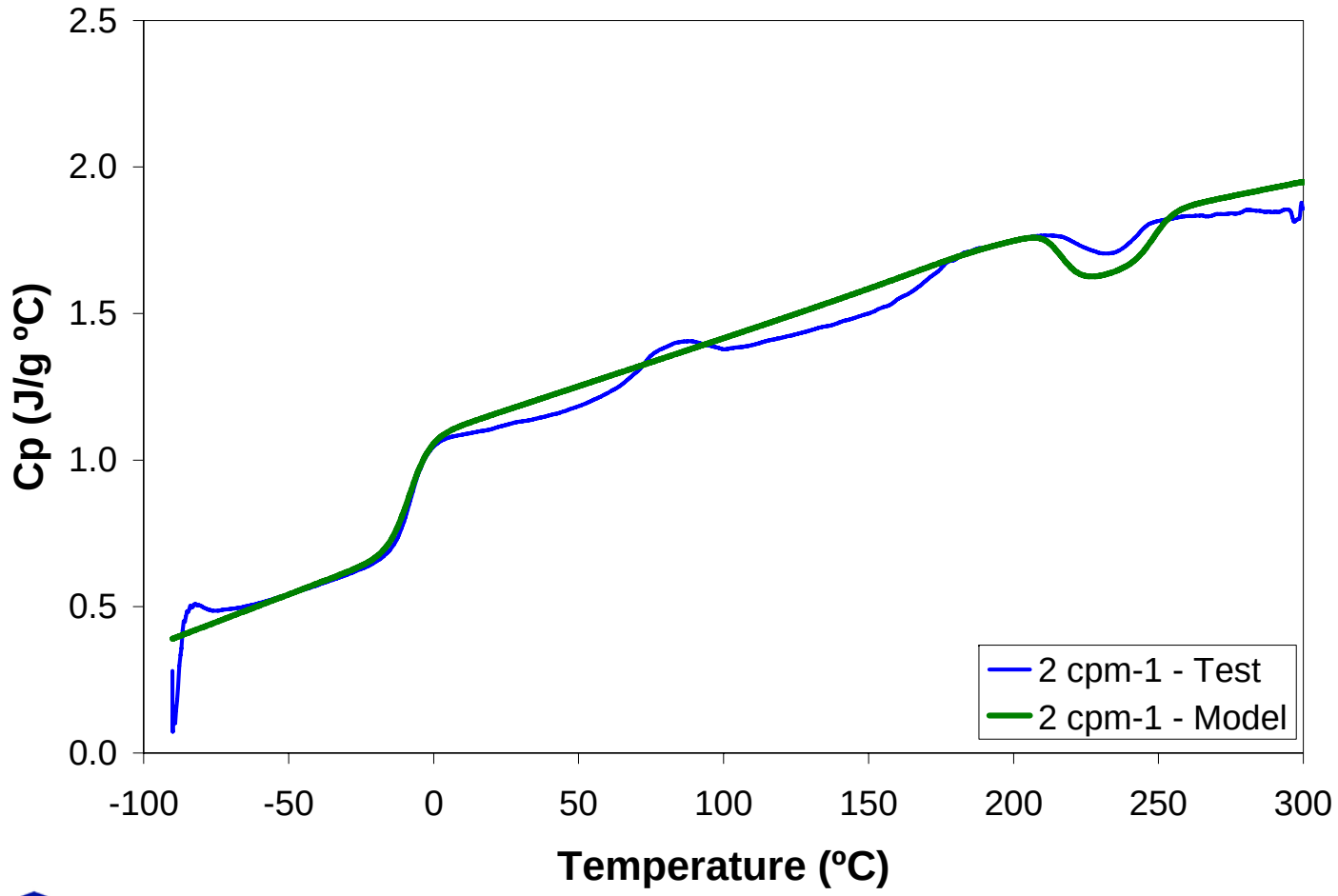
Dynamic Tests – 1Cpm



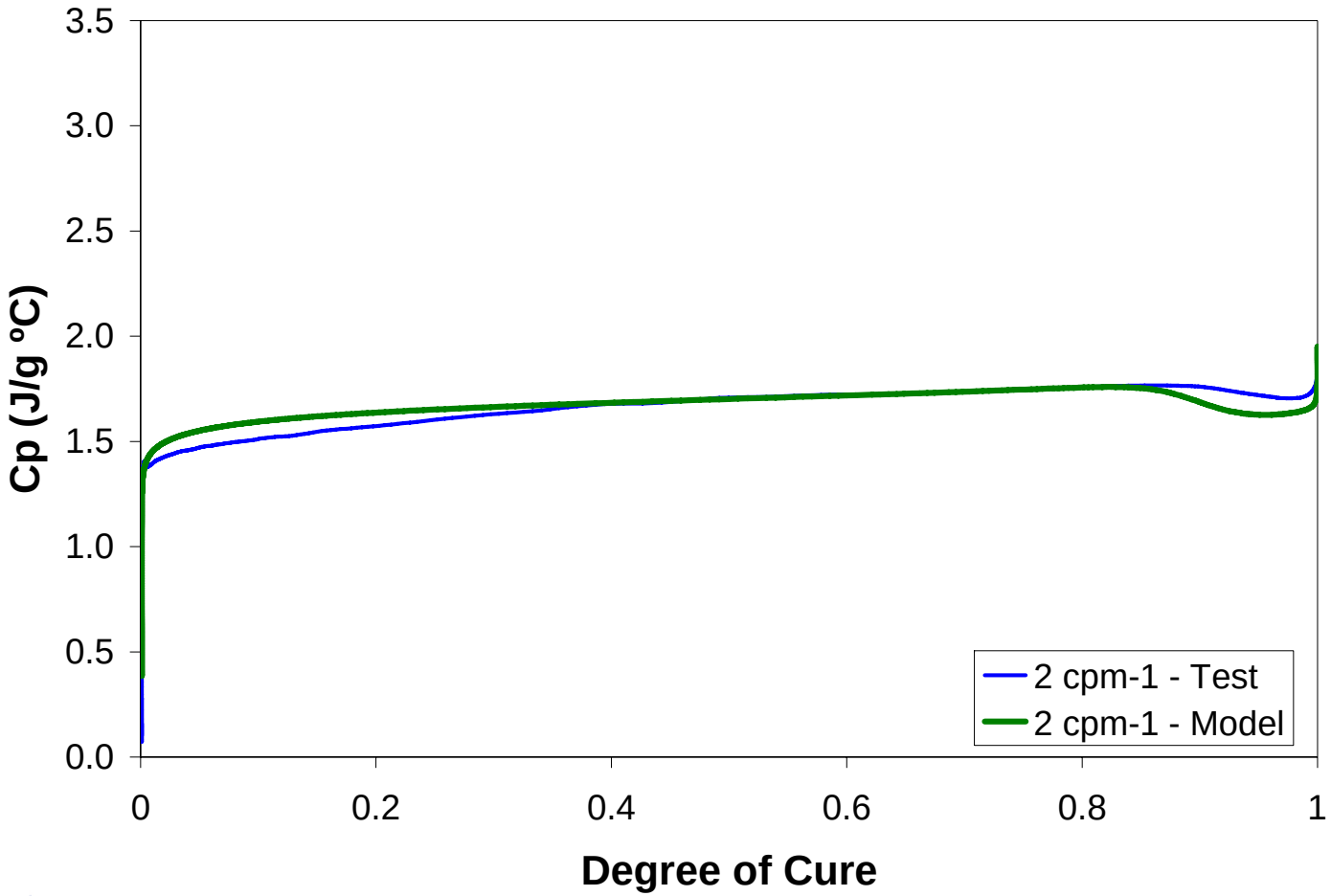
Dynamic Tests – 1Cpm



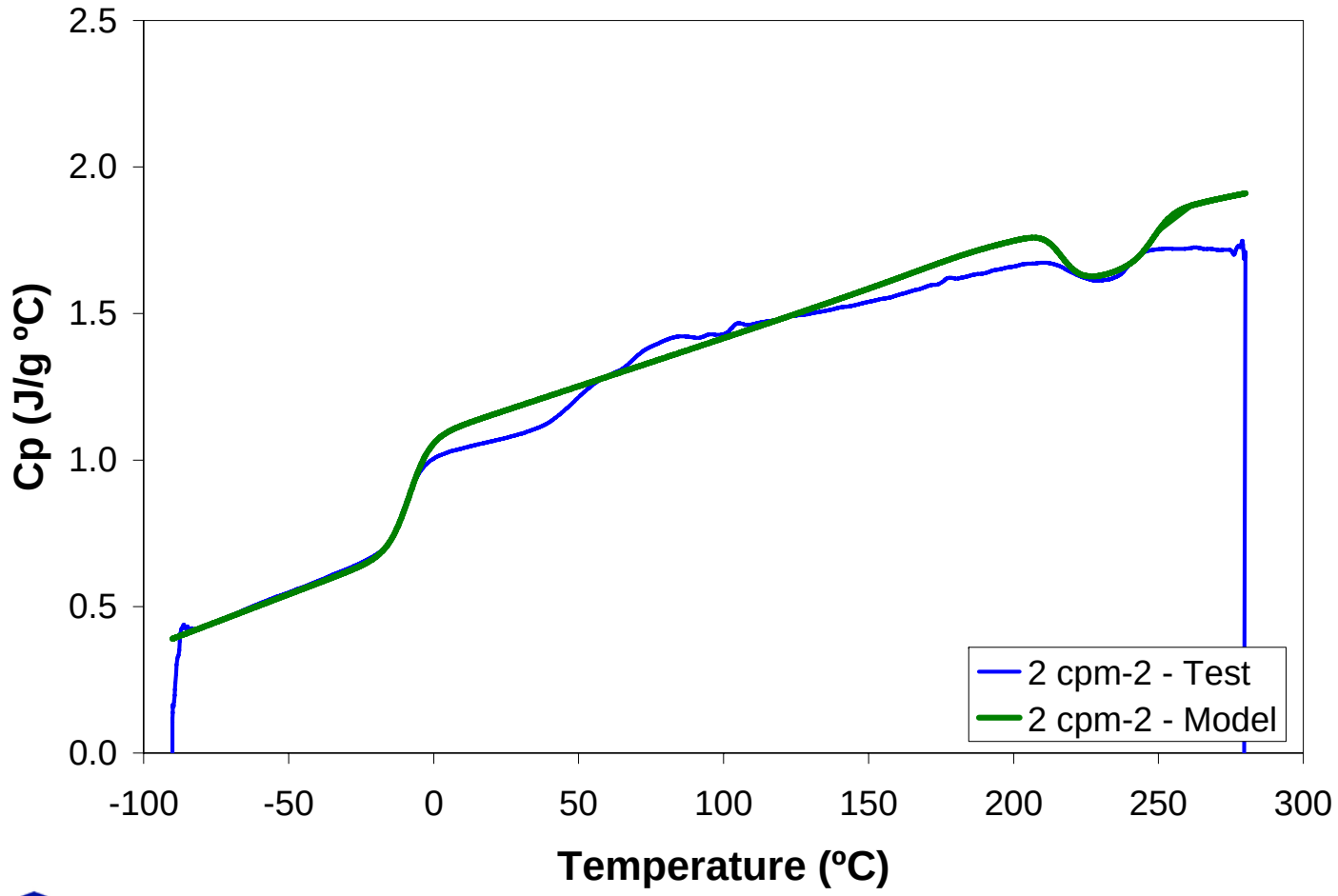
Dynamic Tests – 2Cpm-1



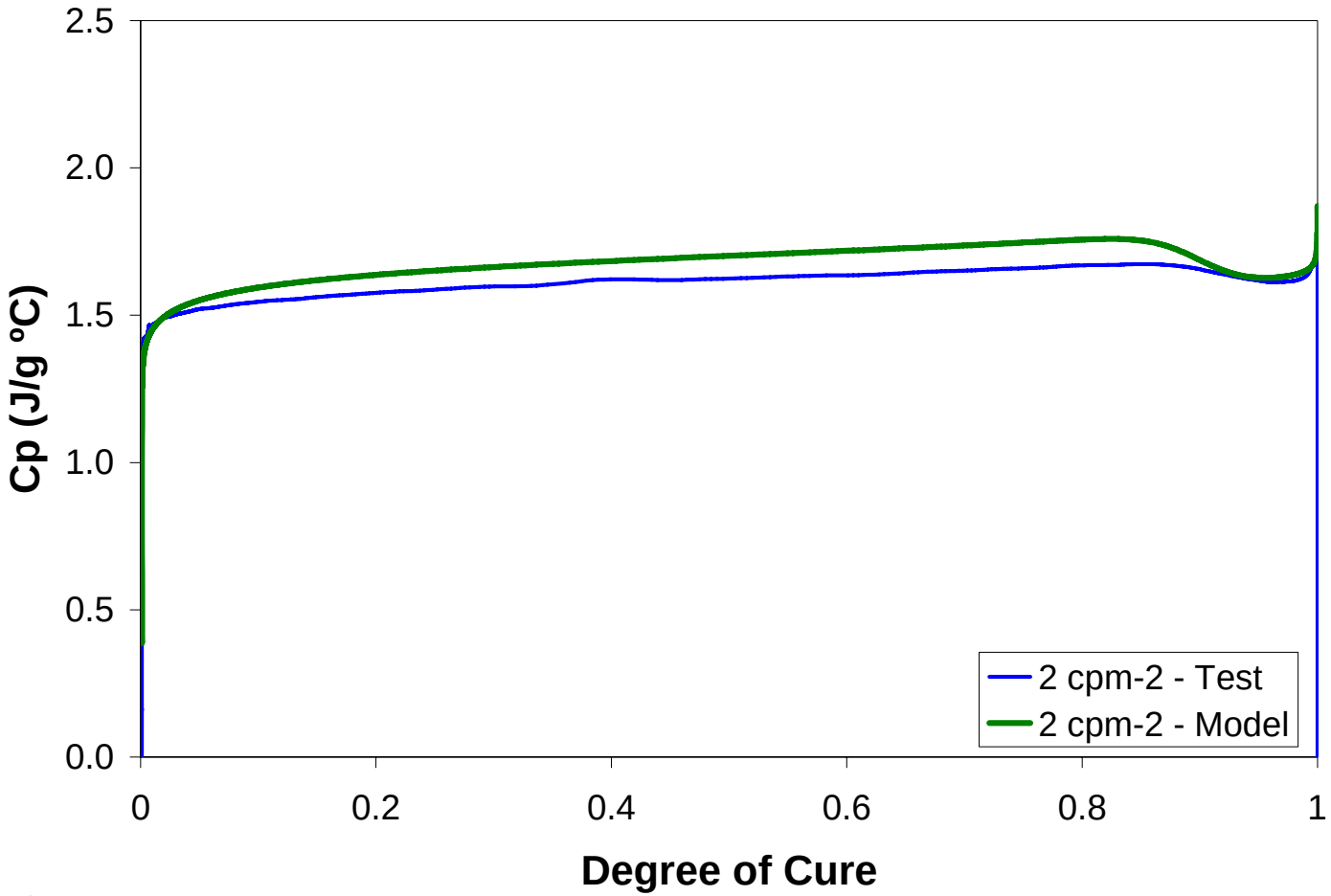
Dynamic Tests – 2Cpm-1



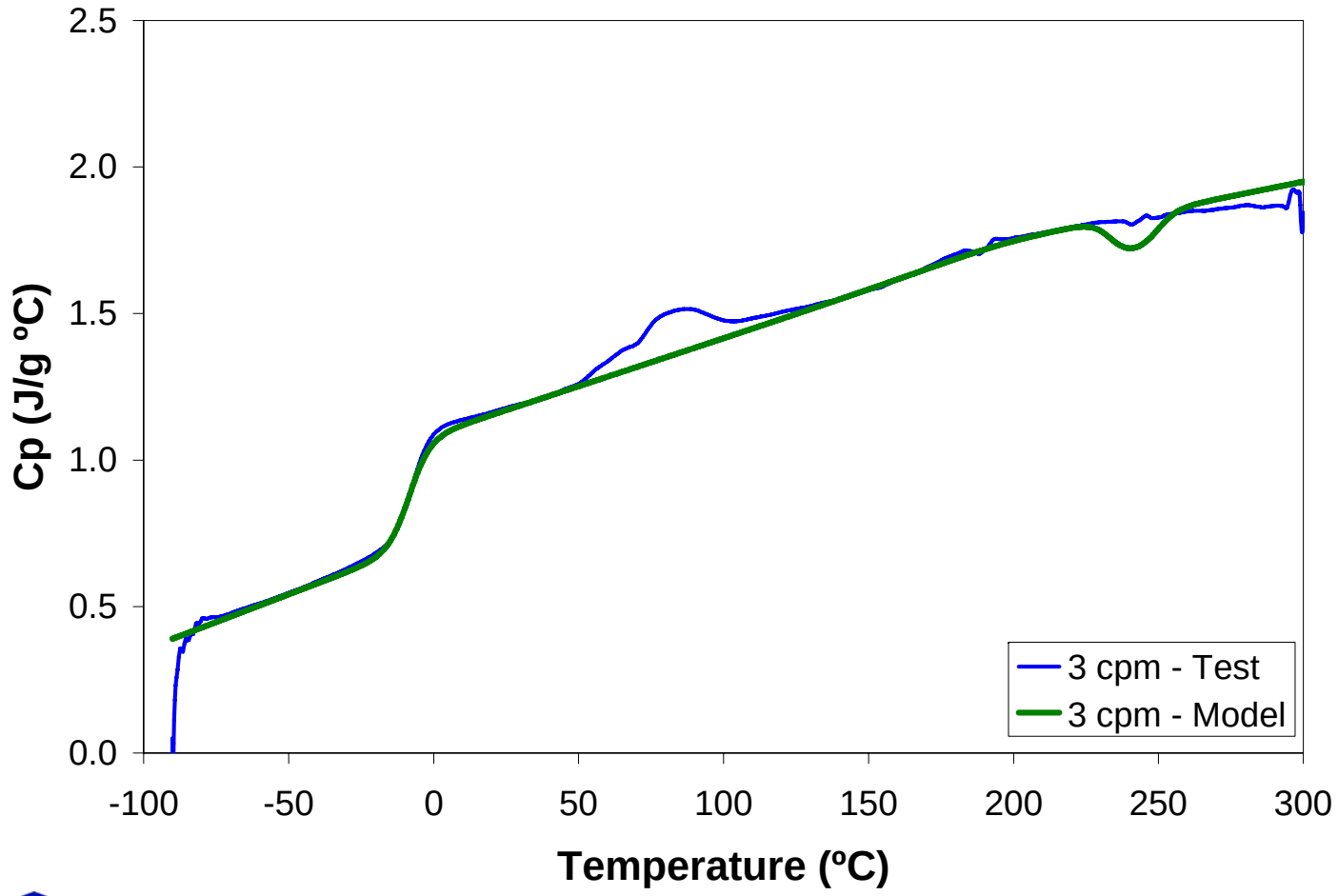
Dynamic Tests – 2Cpm-1



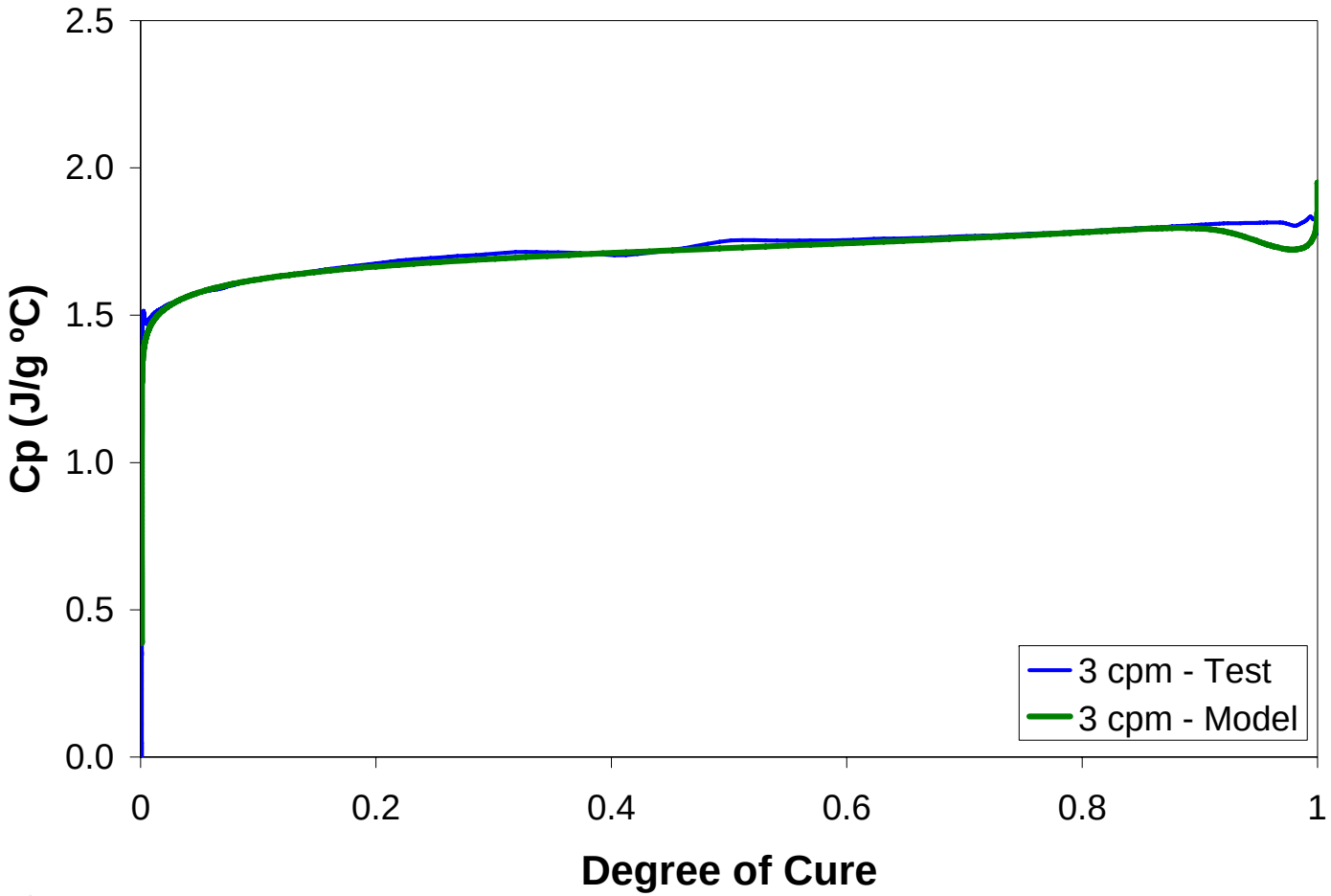
Dynamic Tests – 2Cpm-1



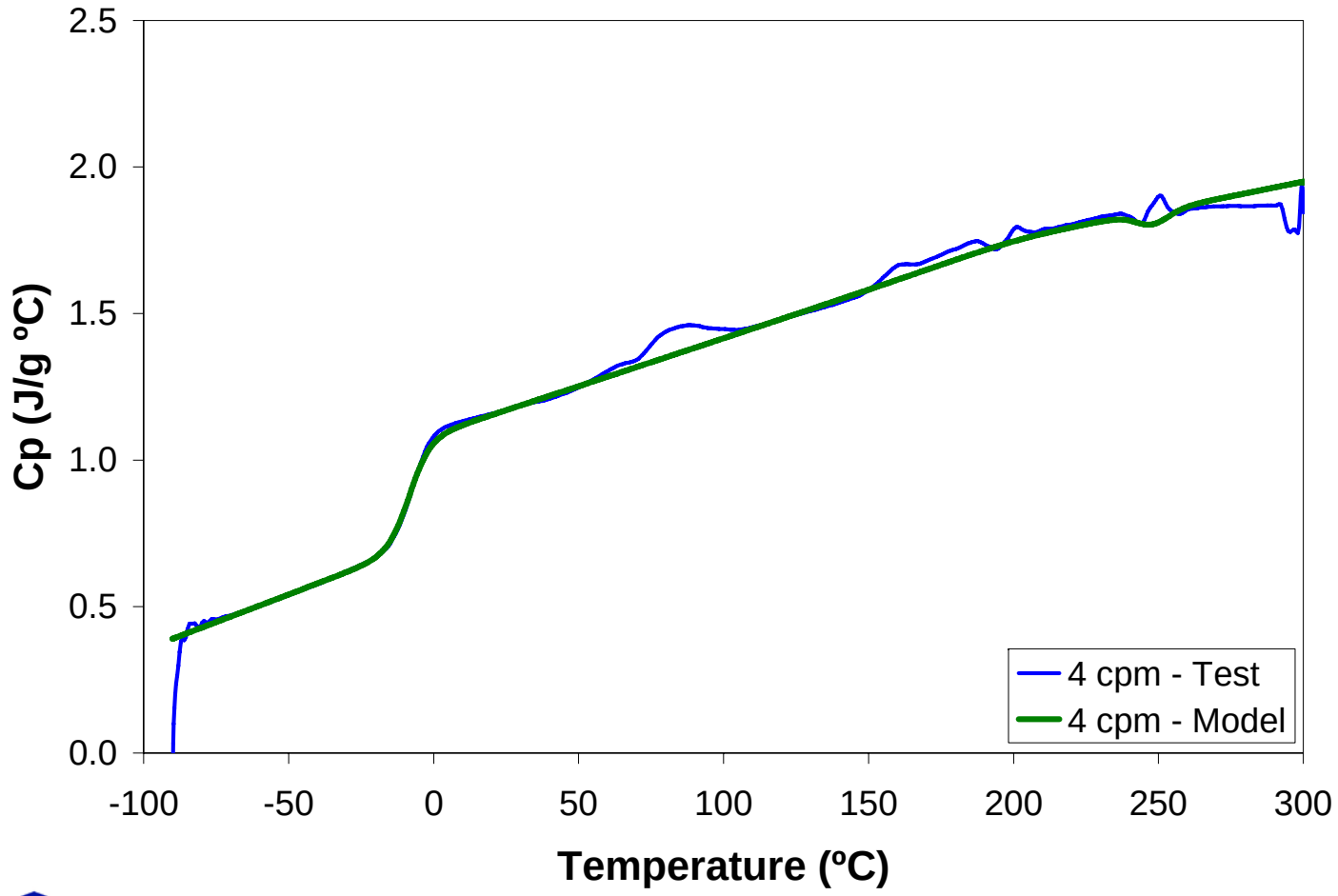
Dynamic Tests – 3Cpm



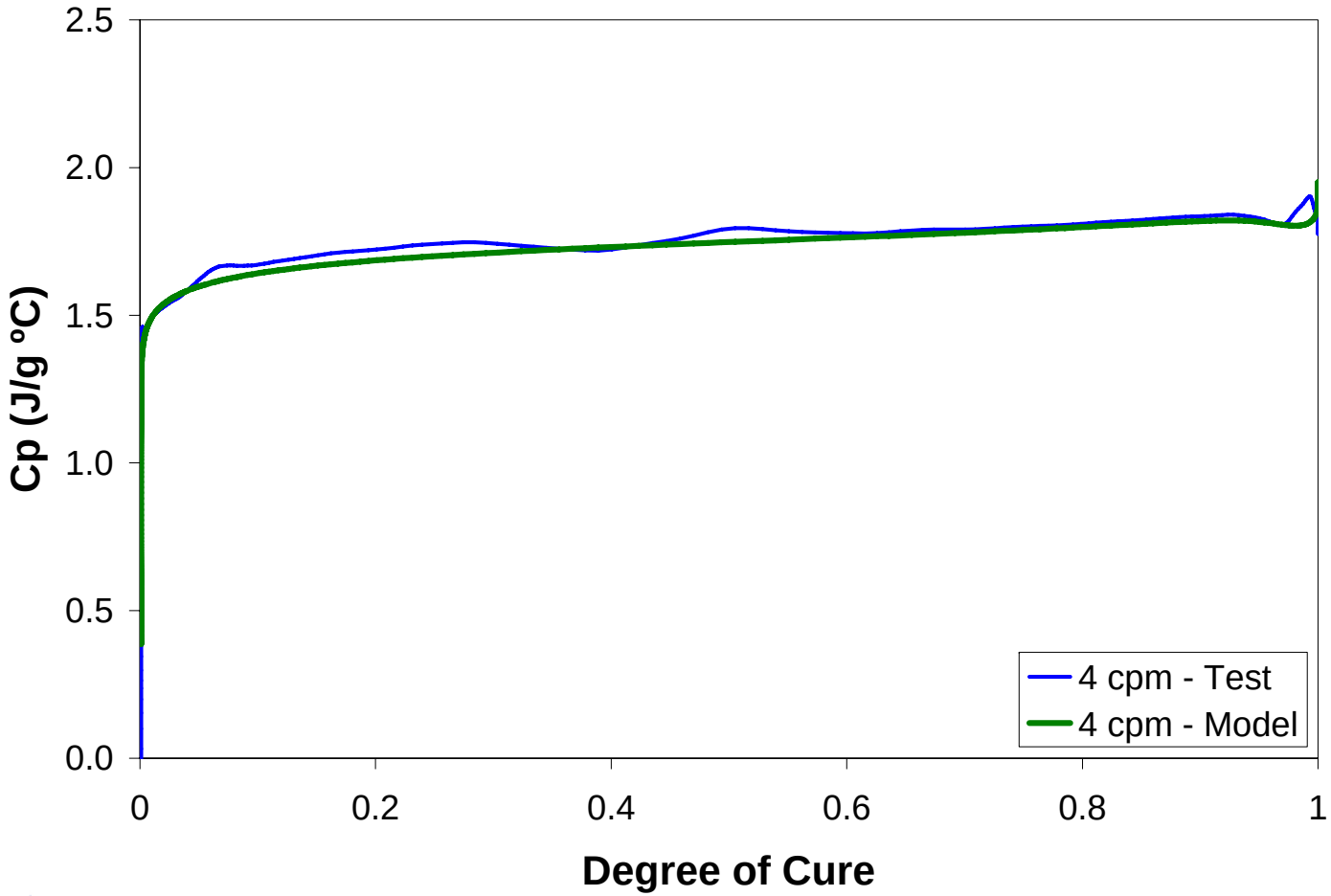
Dynamic Tests – 3Cpm



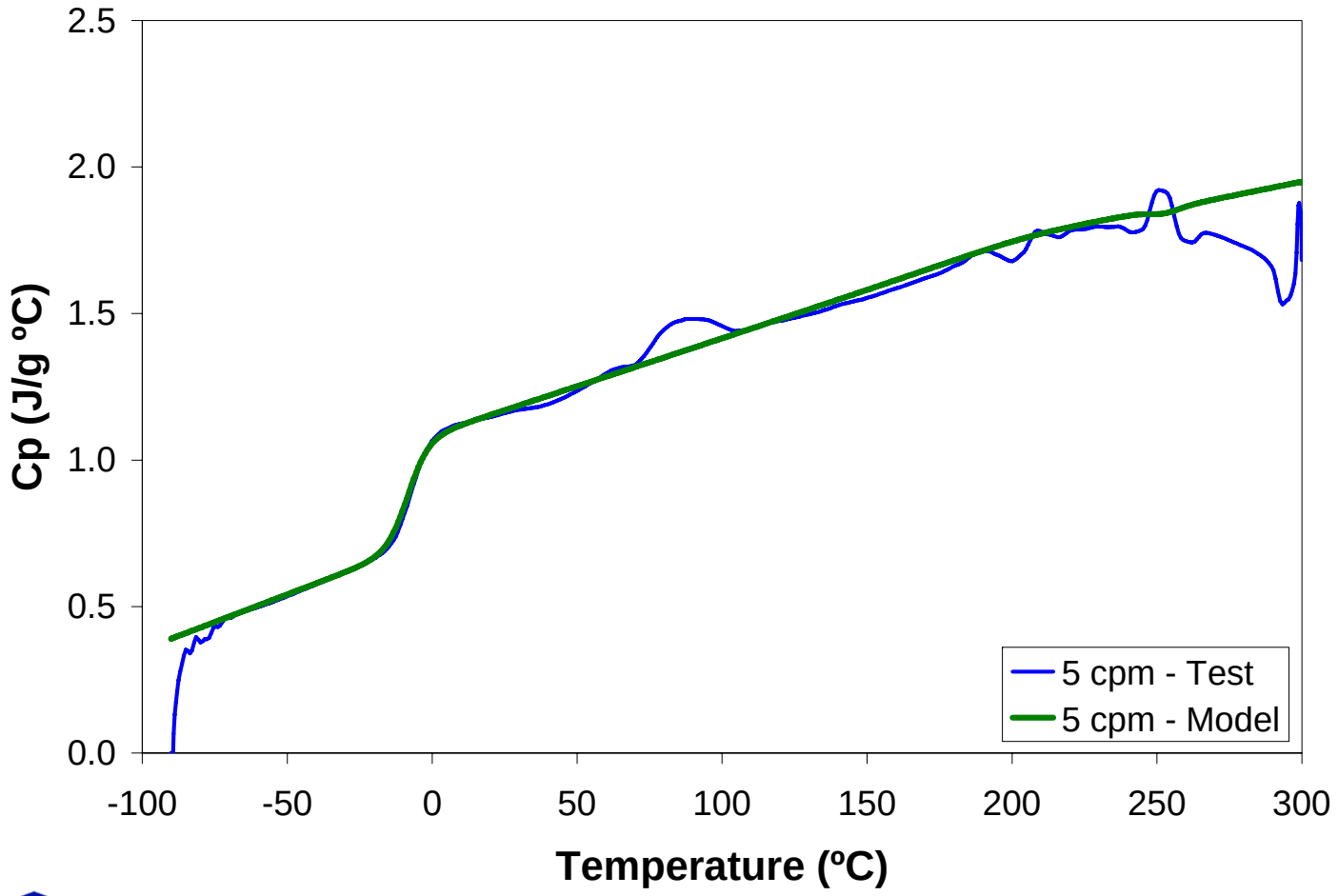
Dynamic Tests – 4Cpm



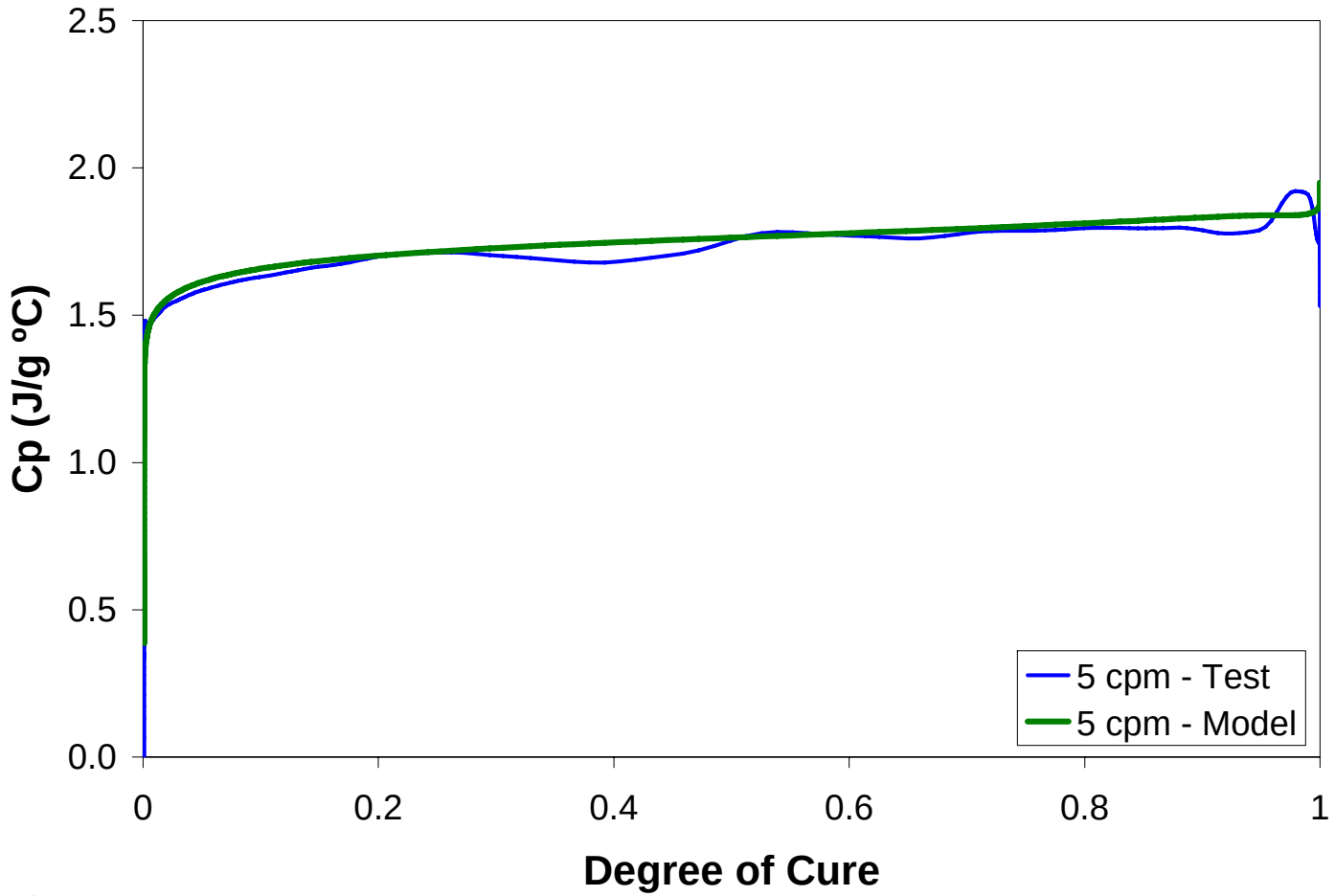
Dynamic Tests – 4Cpm



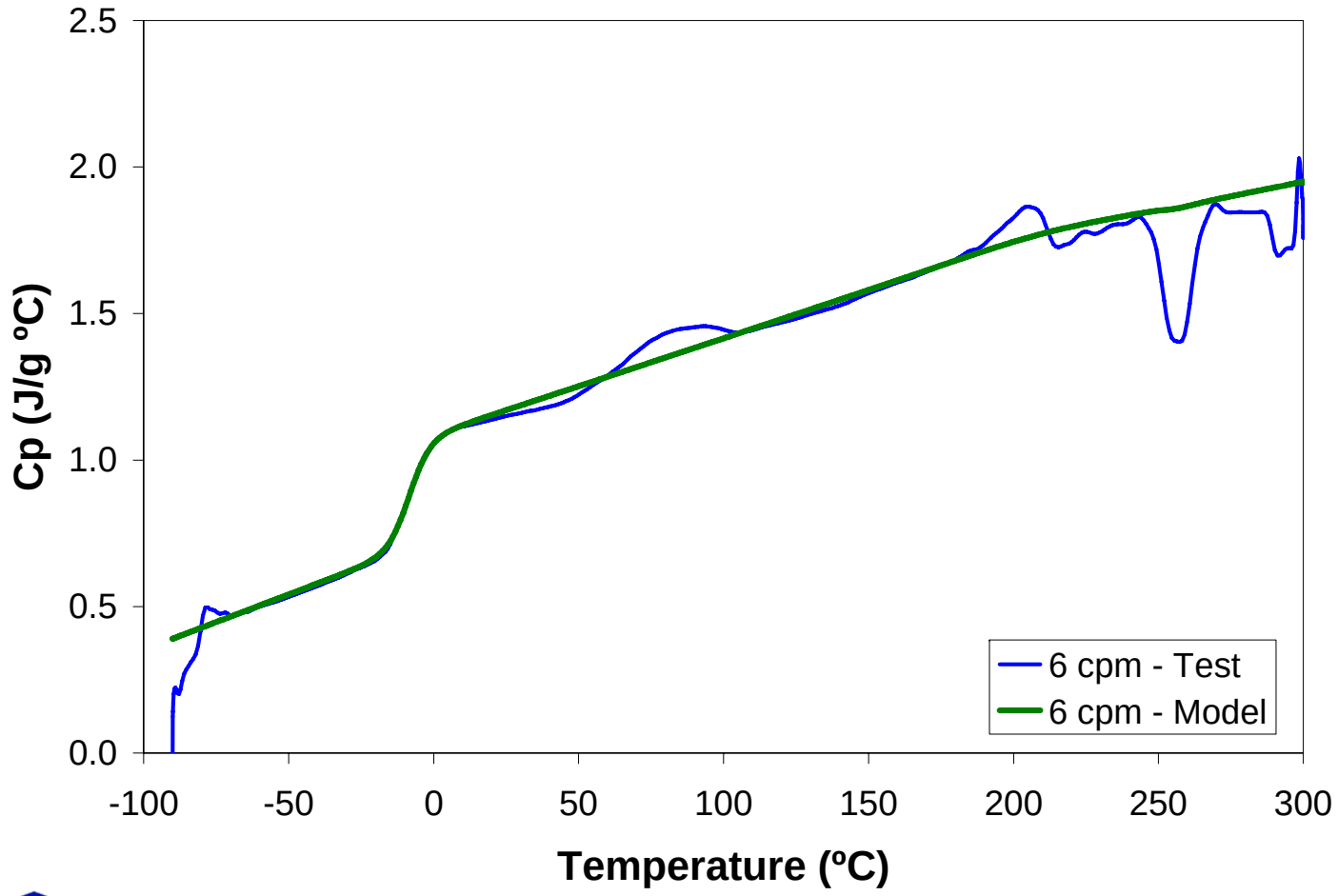
Dynamic Tests – 5Cpm



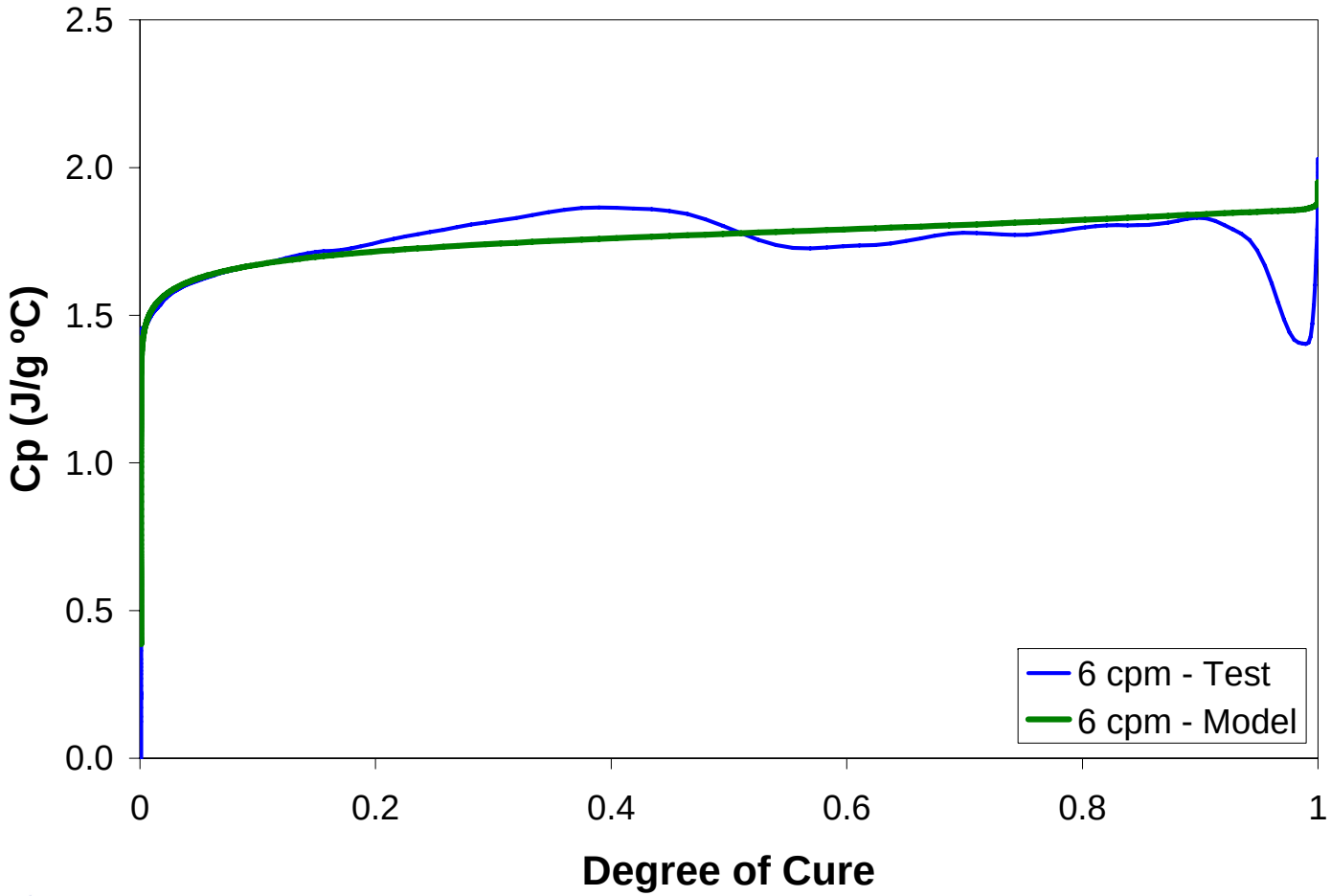
Dynamic Tests – 5Cpm



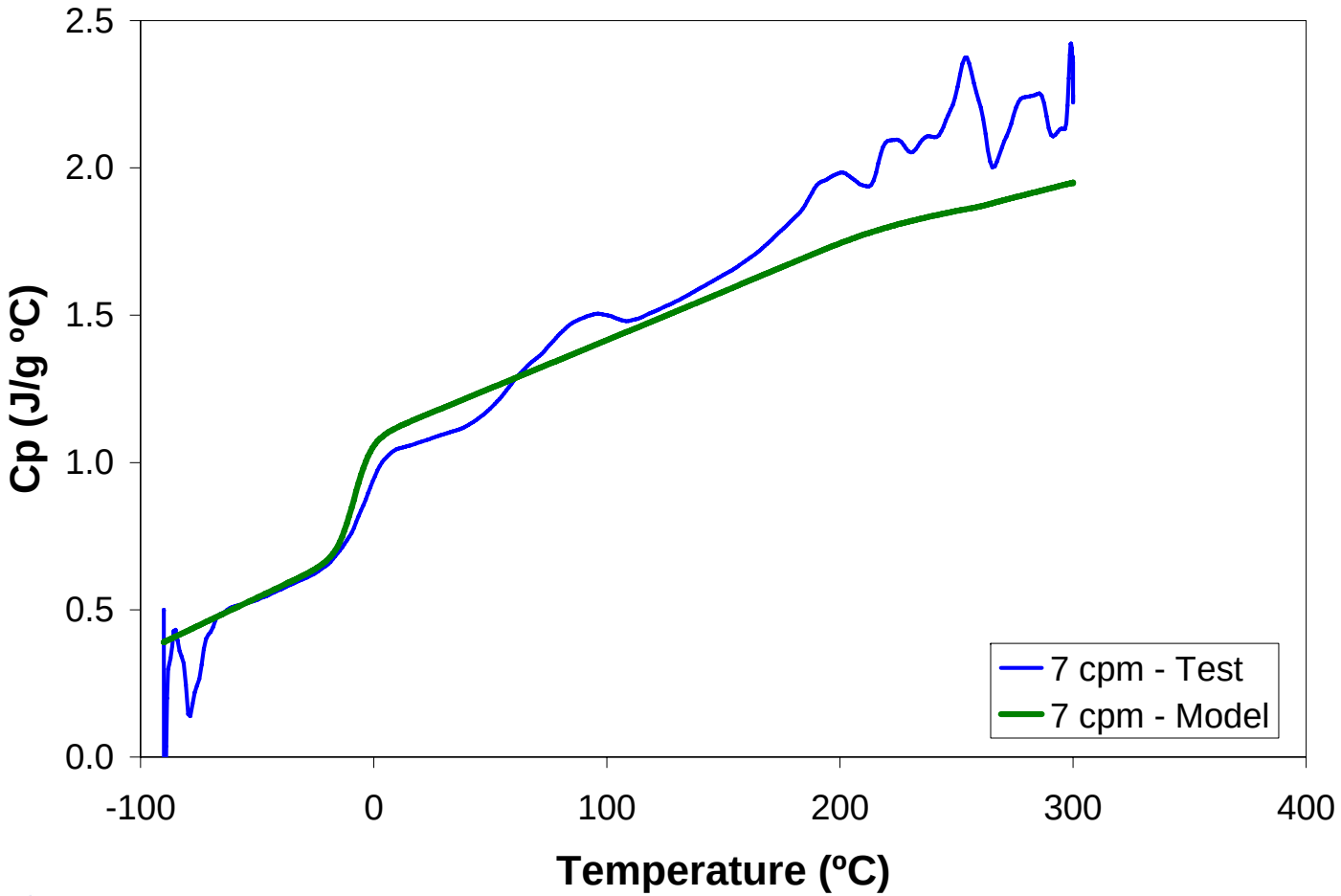
Dynamic Tests – 6Cpm



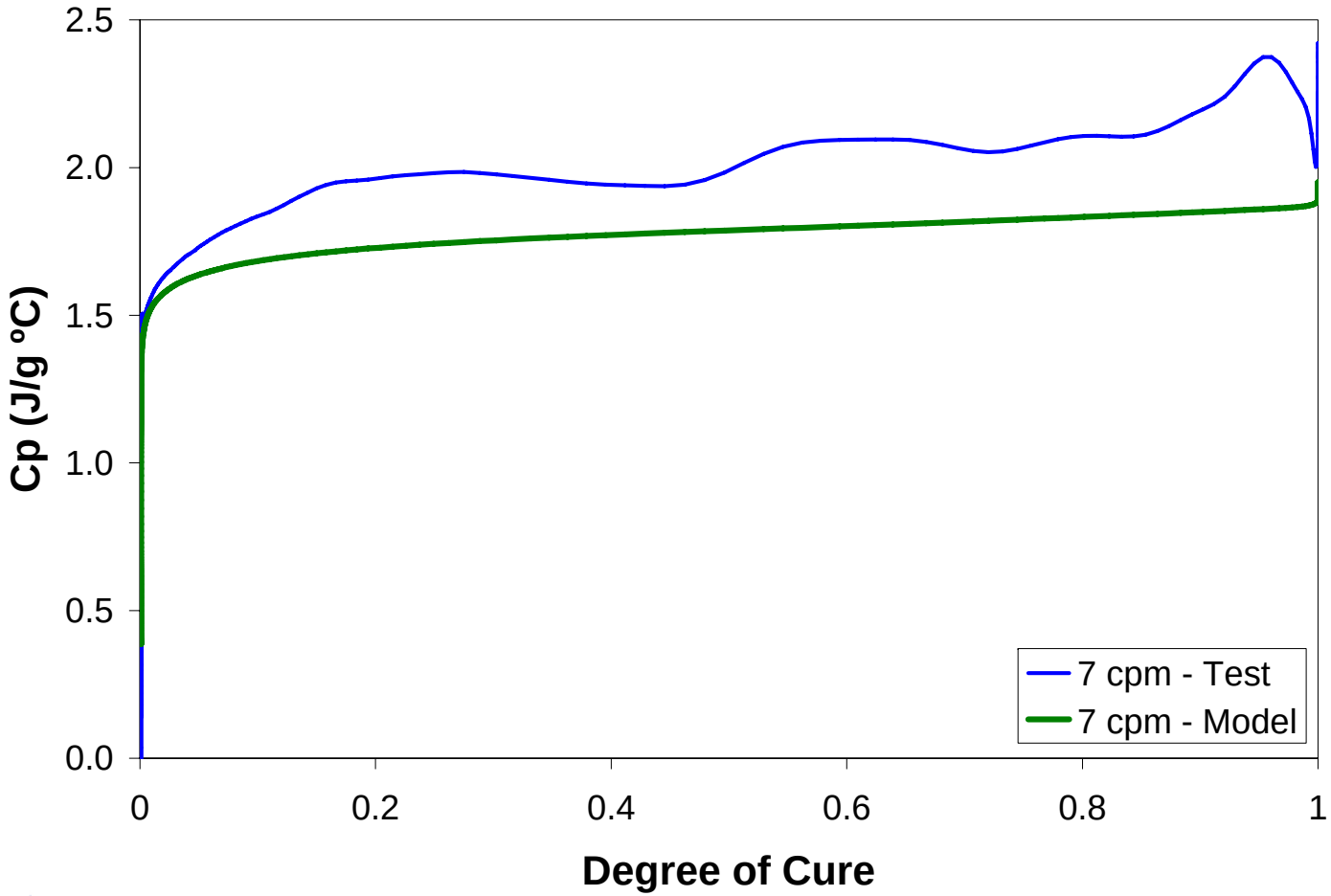
Dynamic Tests – 6Cpm



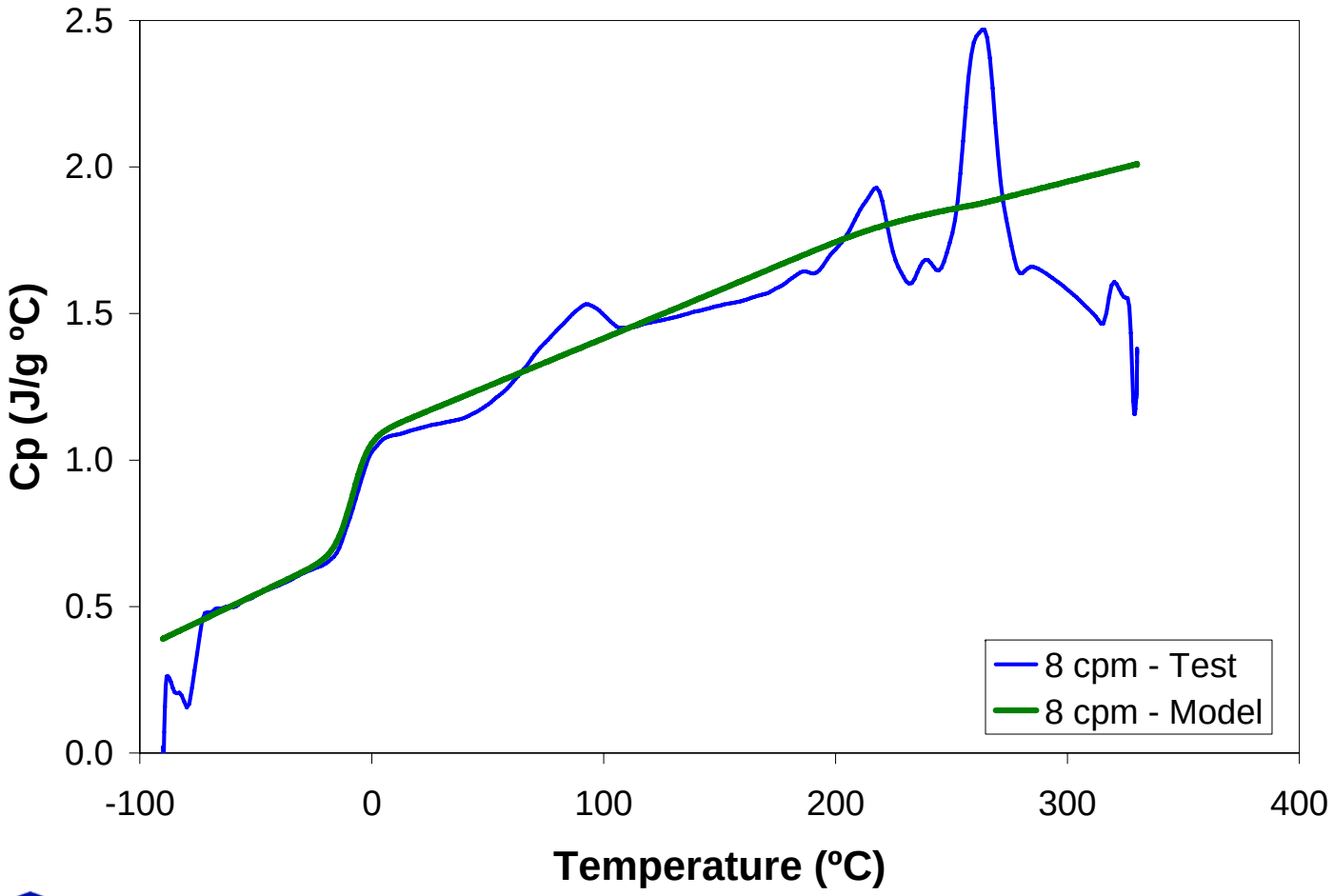
Dynamic Tests – 7Cpm



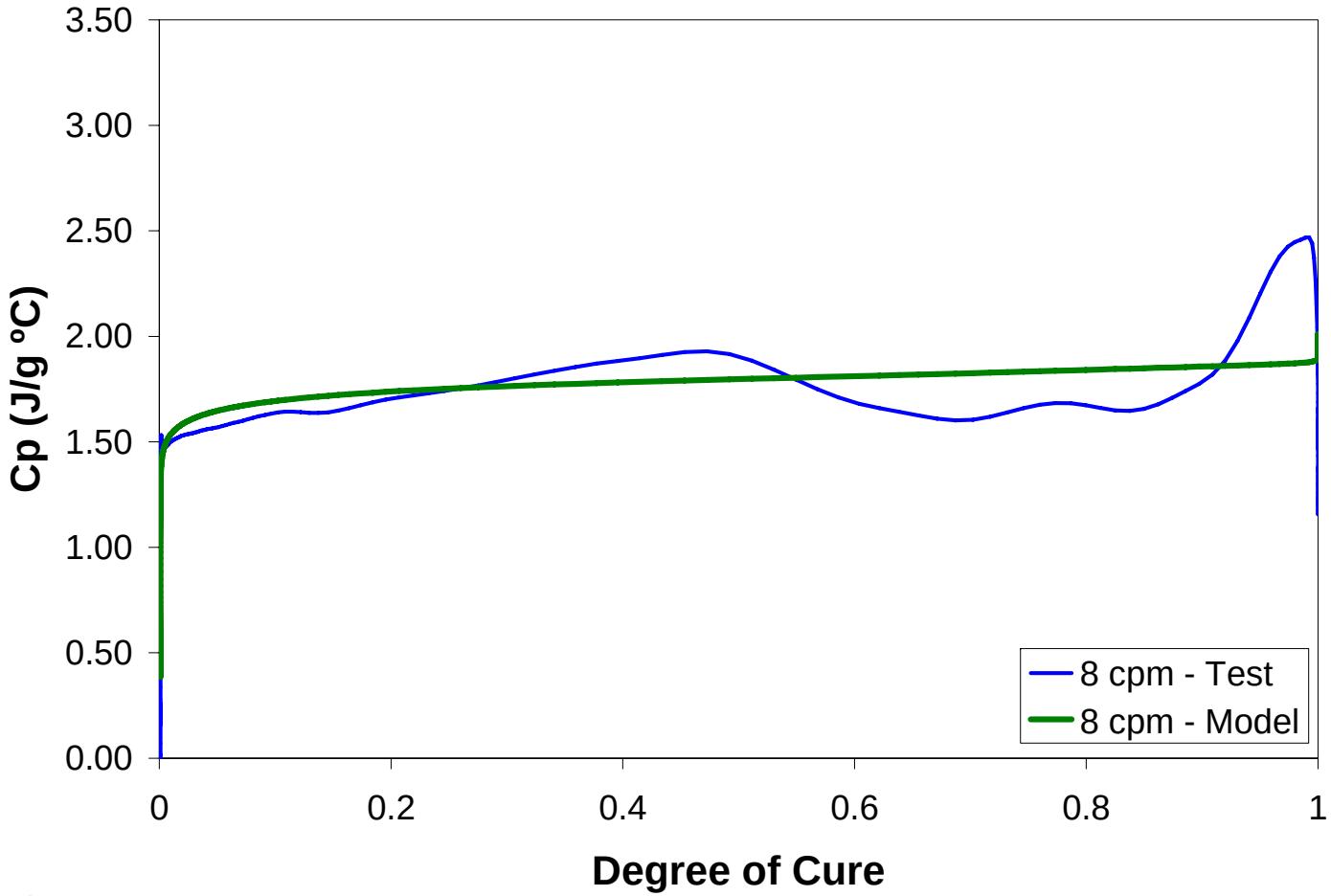
Dynamic Tests – 7Cpm



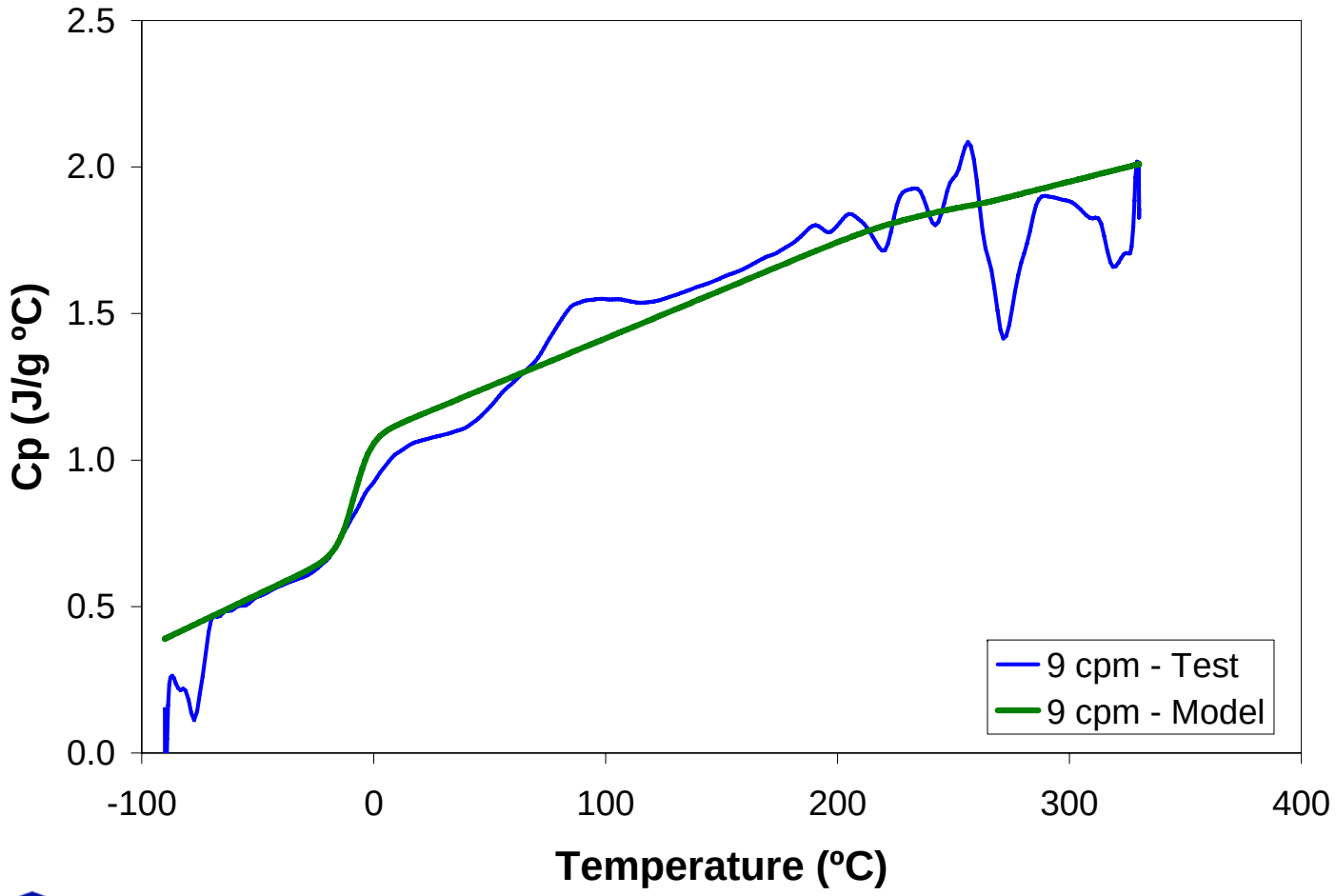
Dynamic Tests – 8Cpm



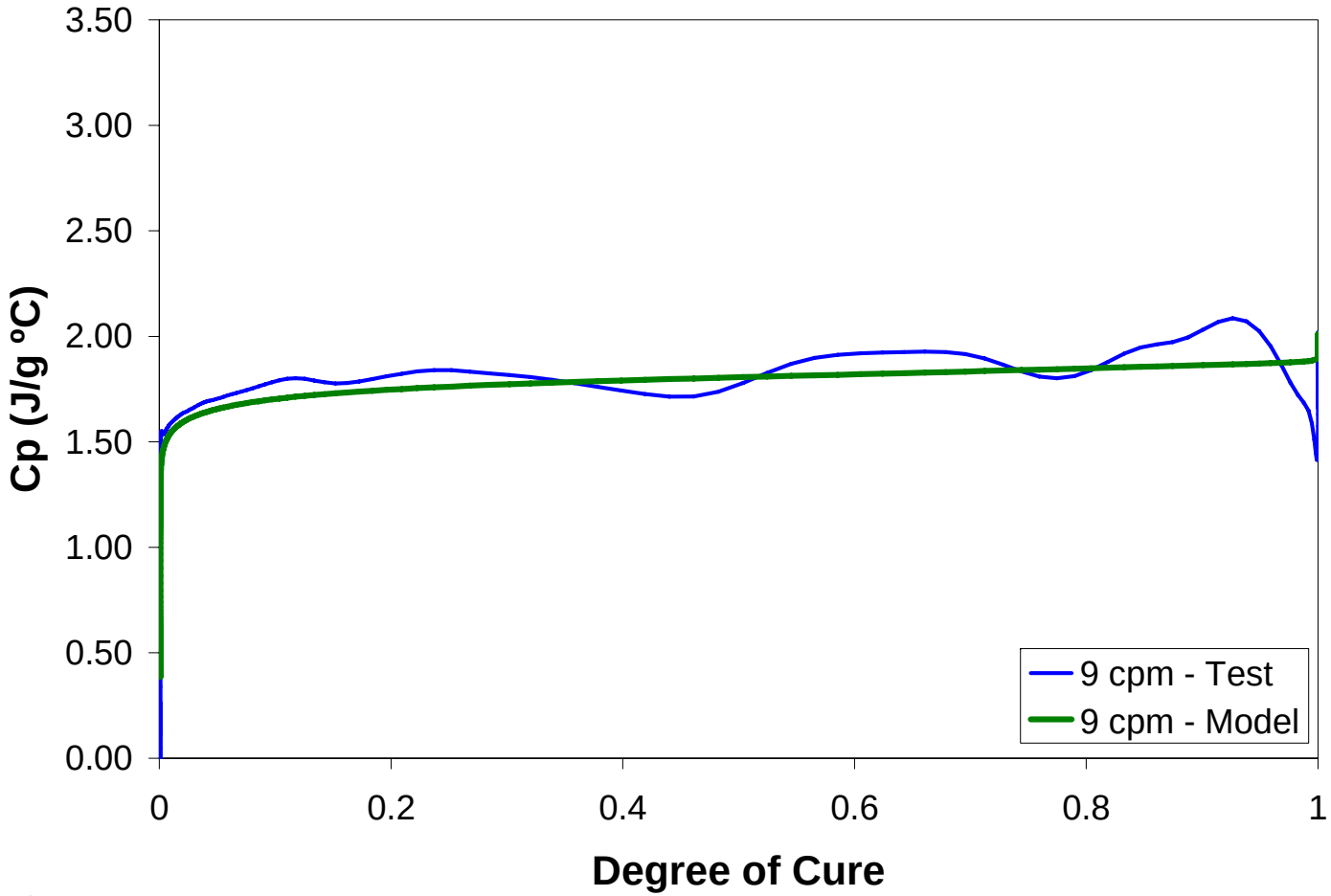
Dynamic Tests – 8Cpm



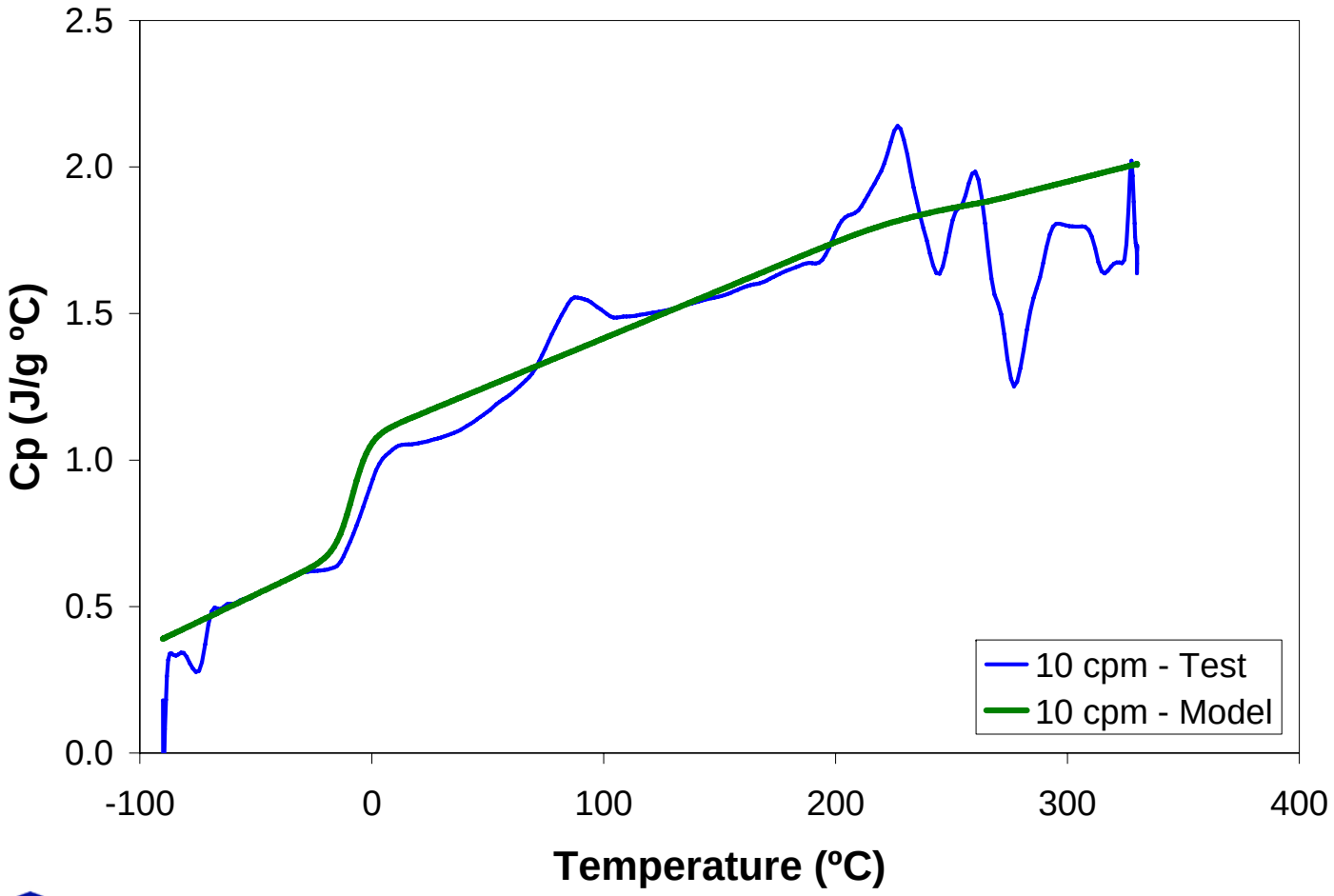
Dynamic Tests – 9Cpm



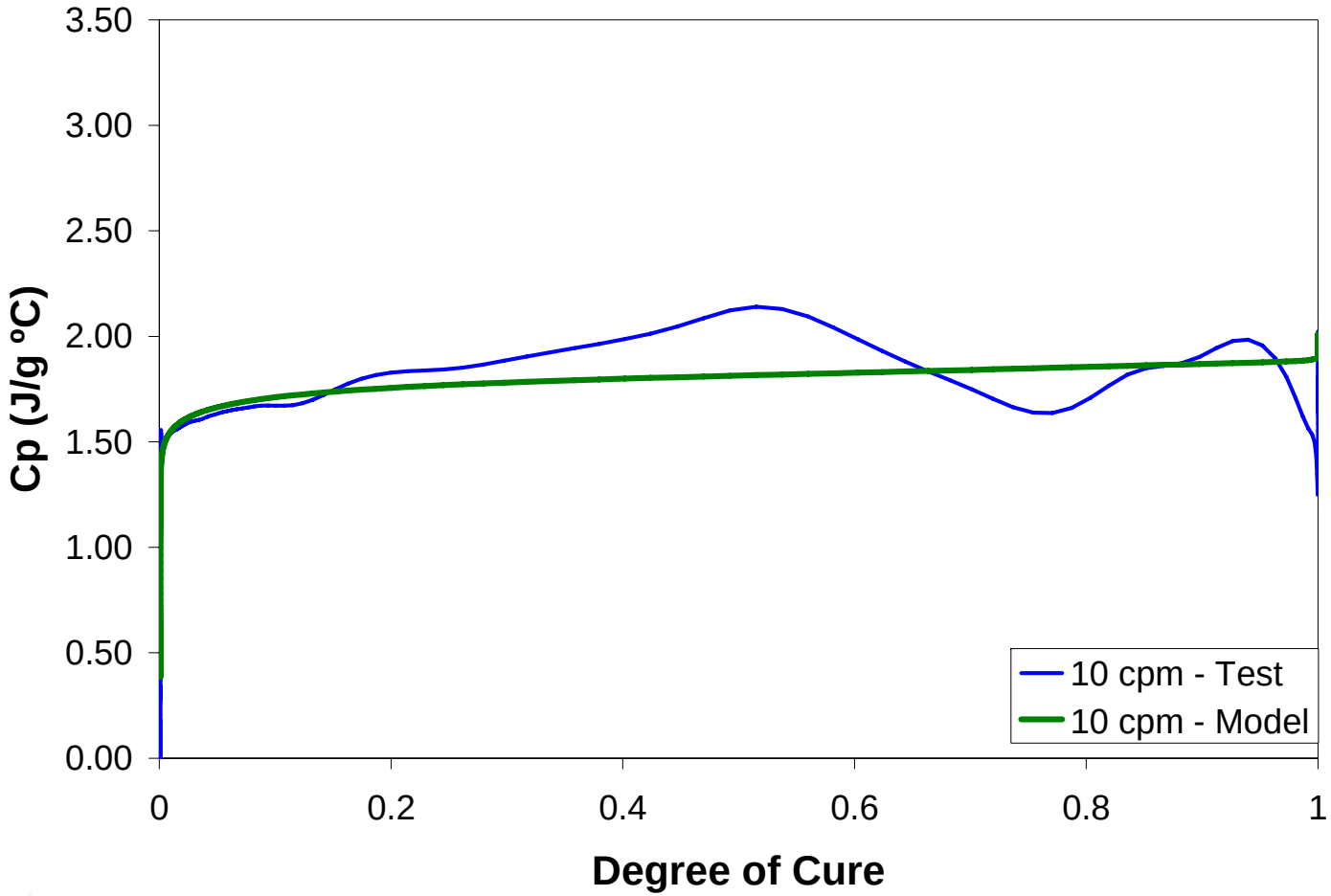
Dynamic Tests – 9Cpm



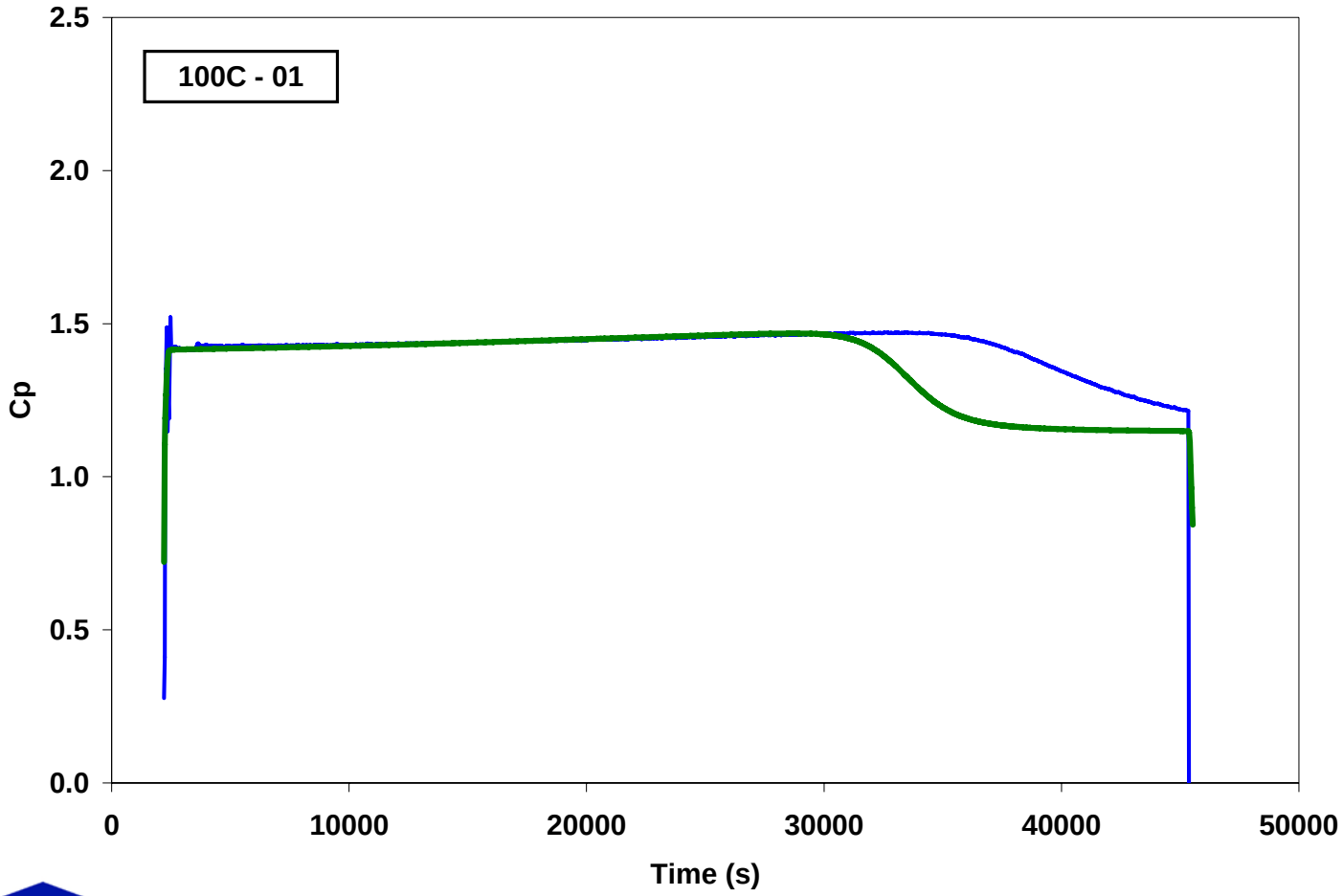
Dynamic Tests – 10Cpm



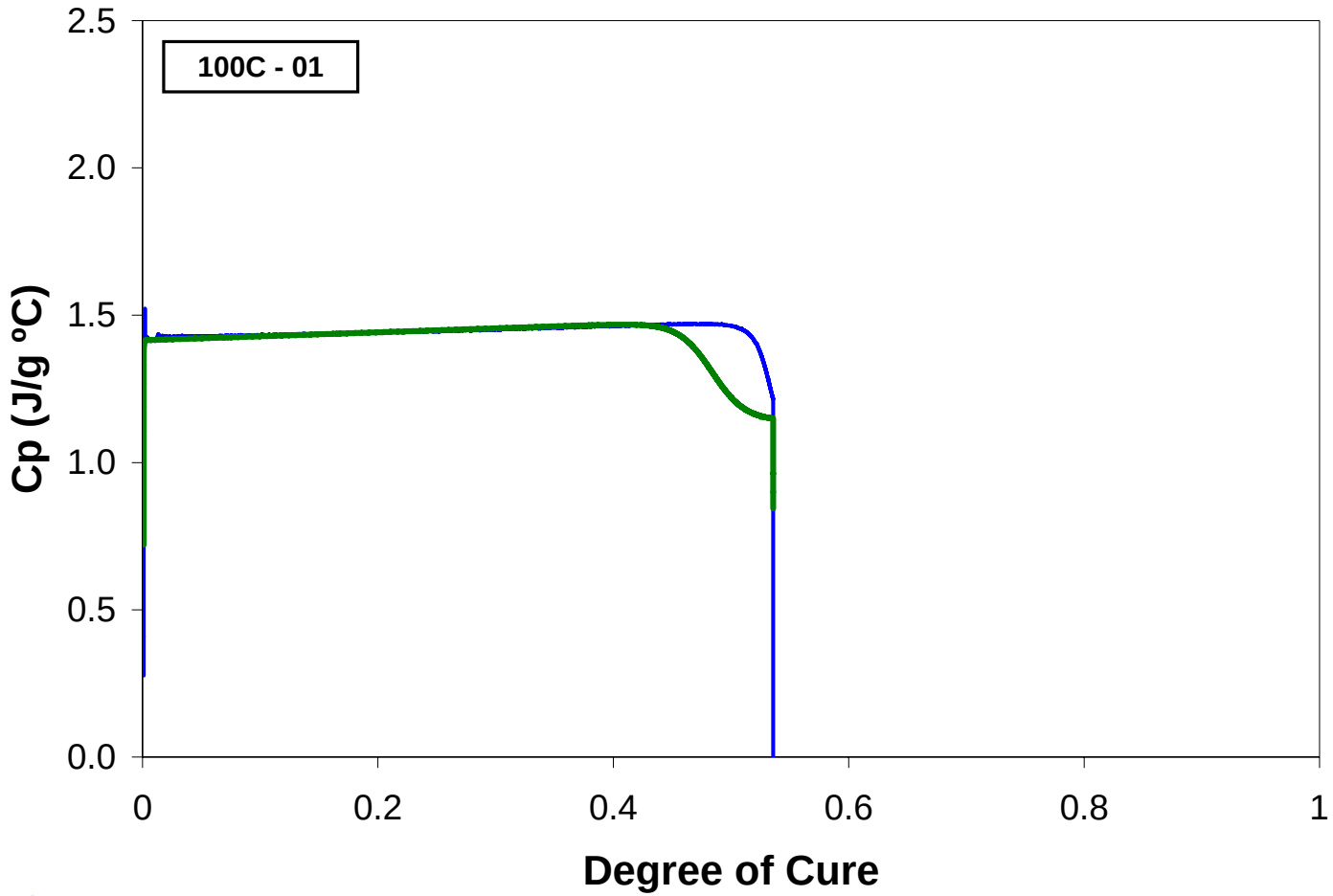
Dynamic Tests – 10Cpm



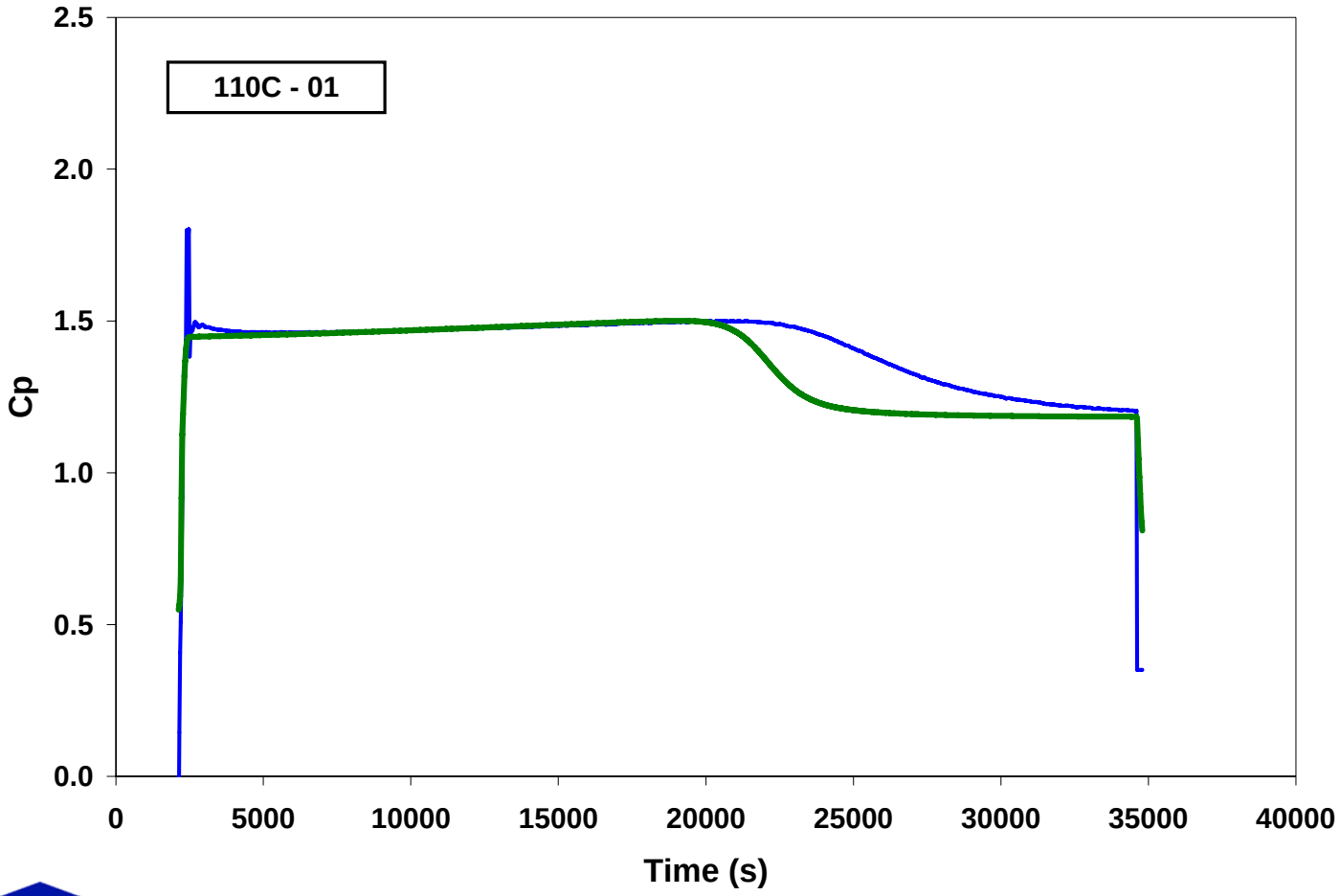
Isothermal Tests – 100C



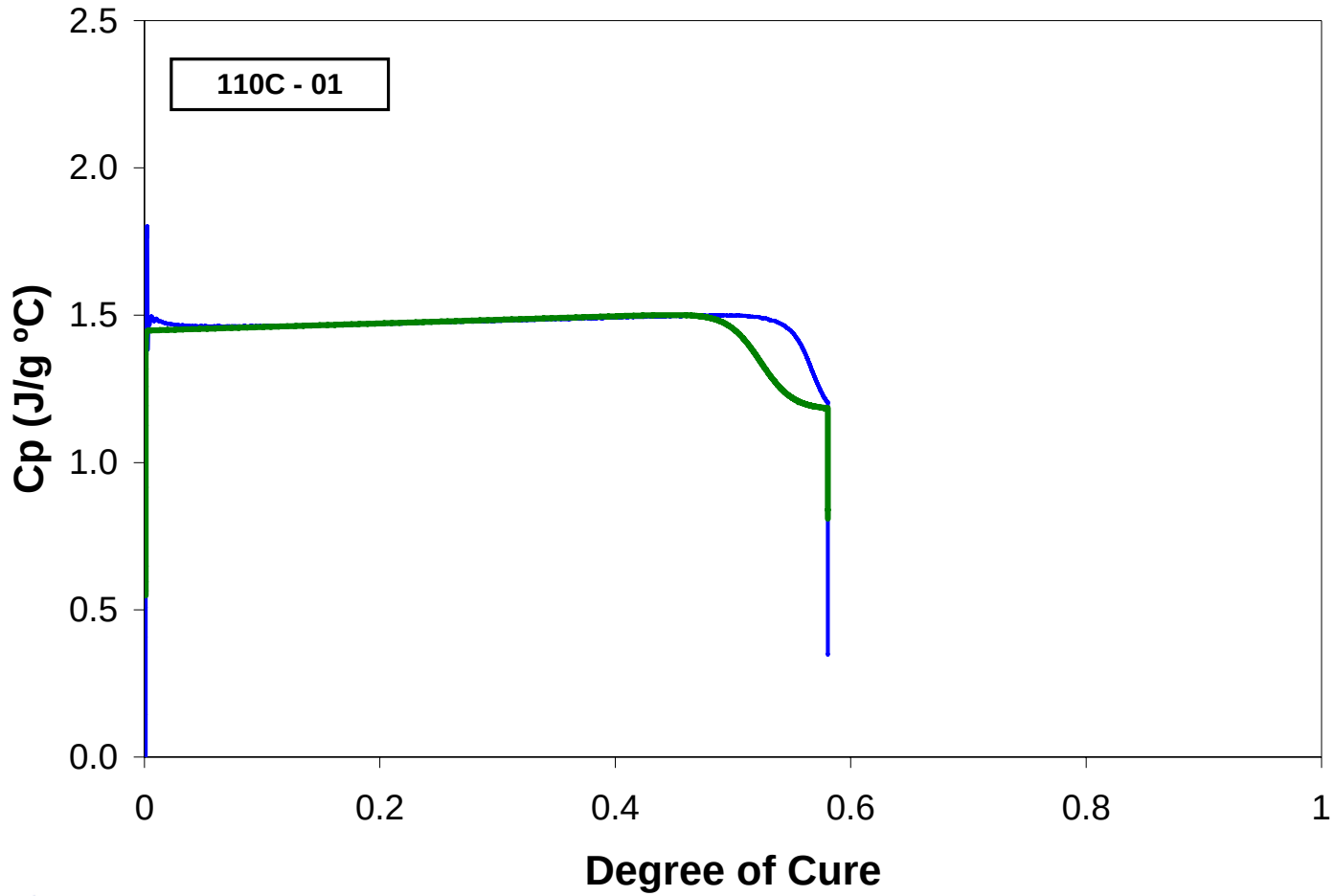
Isothermal Tests – 100C



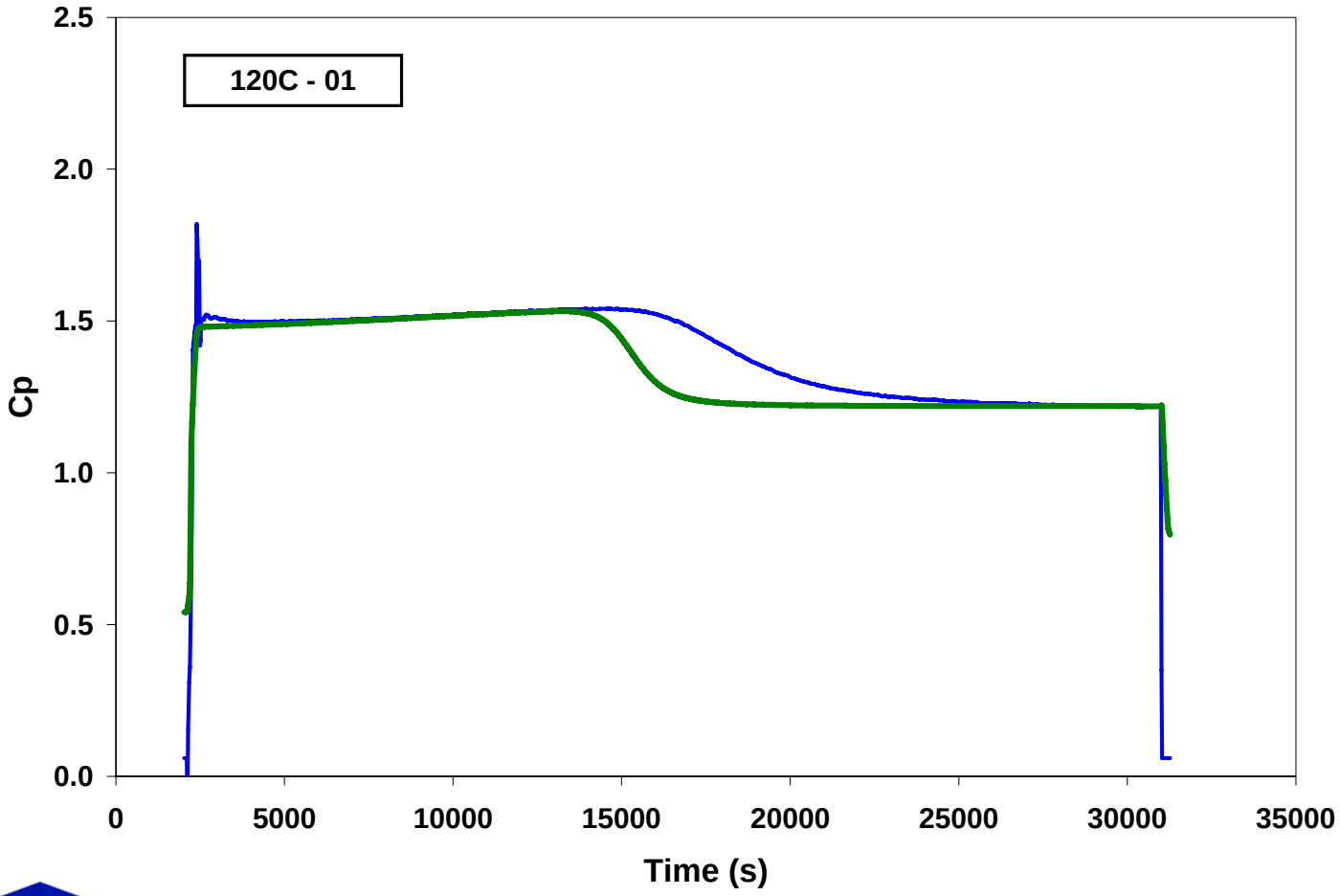
Isothermal Tests – 110C



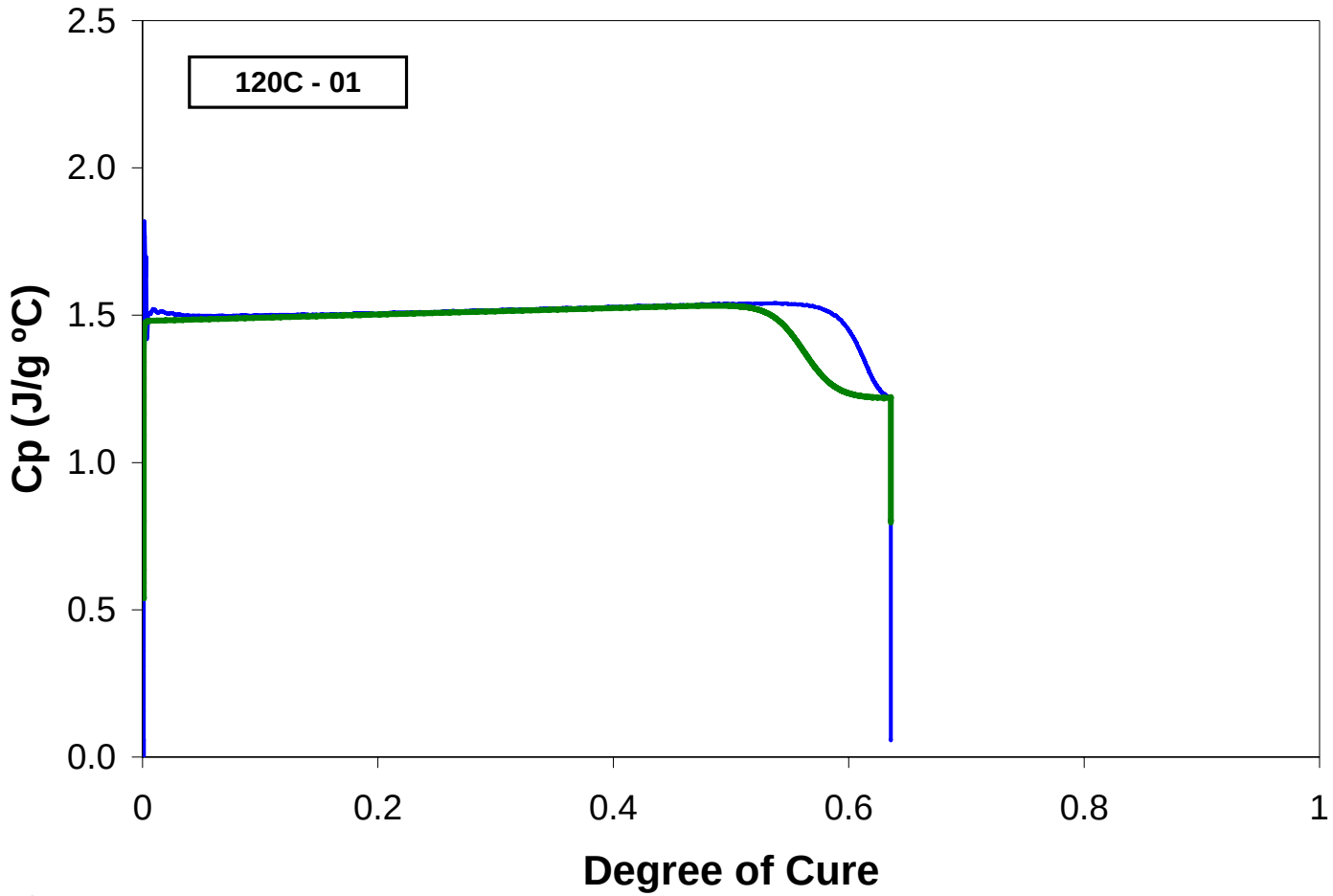
Isothermal Tests – 110C



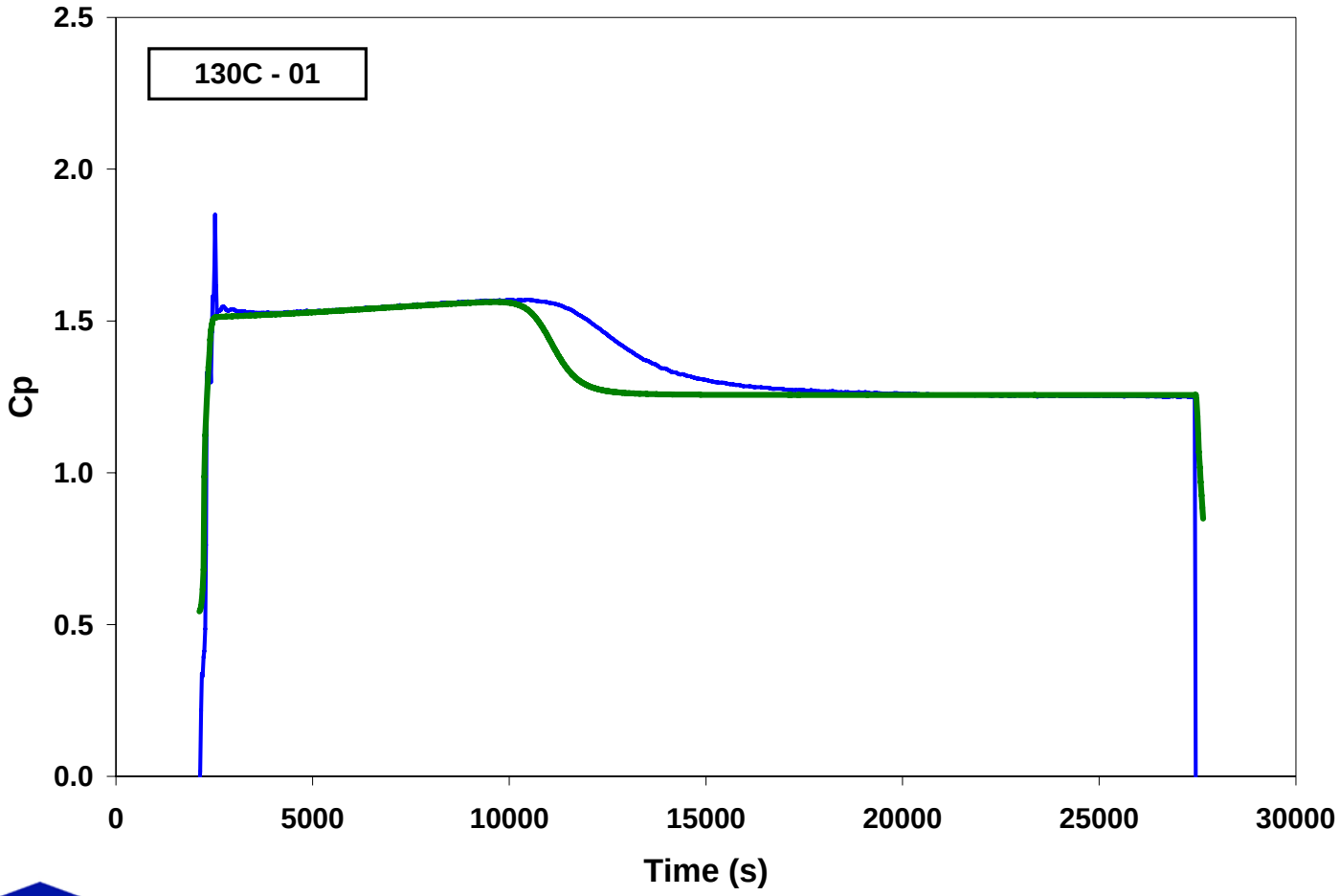
Isothermal Tests – 120C



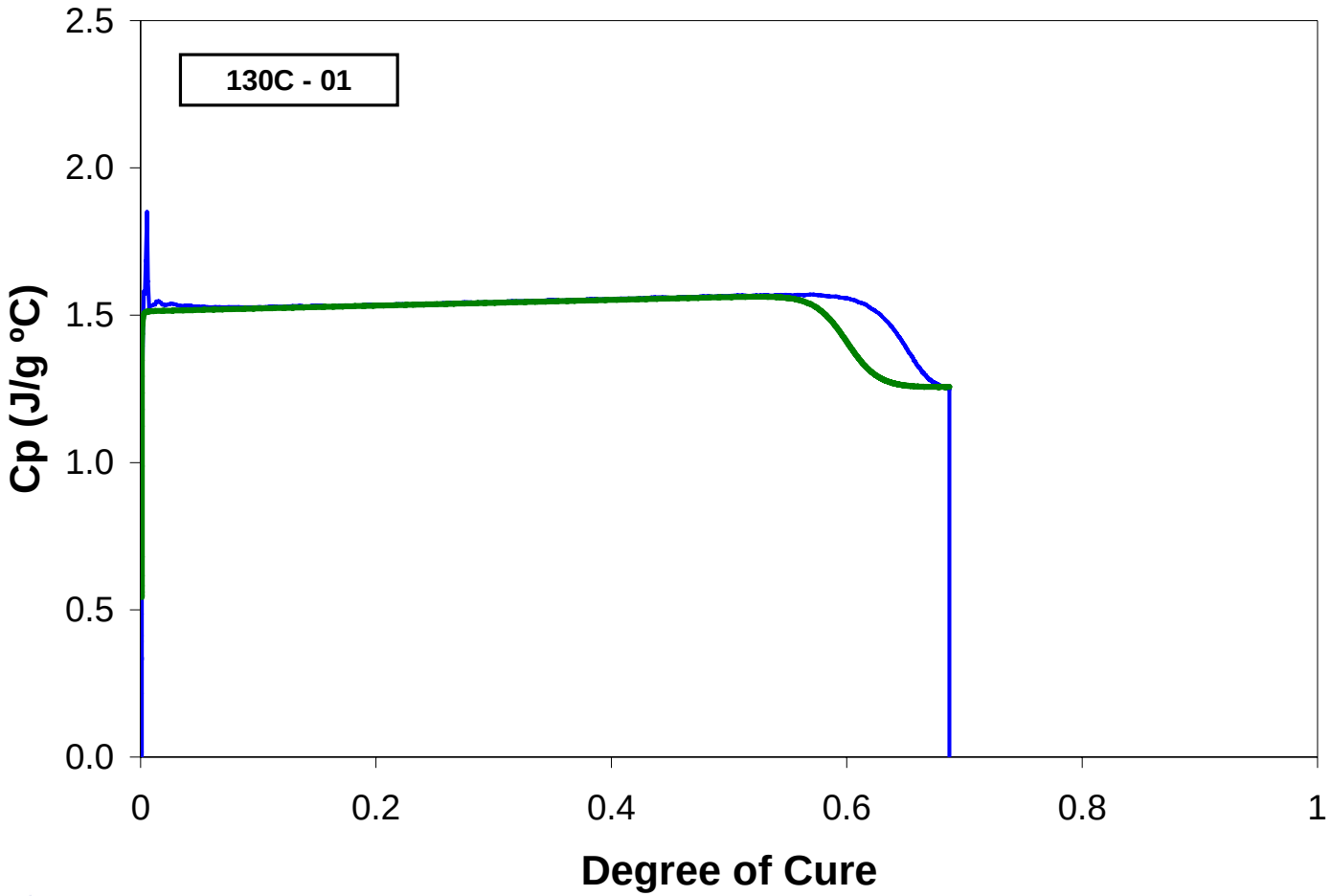
Isothermal Tests – 120C



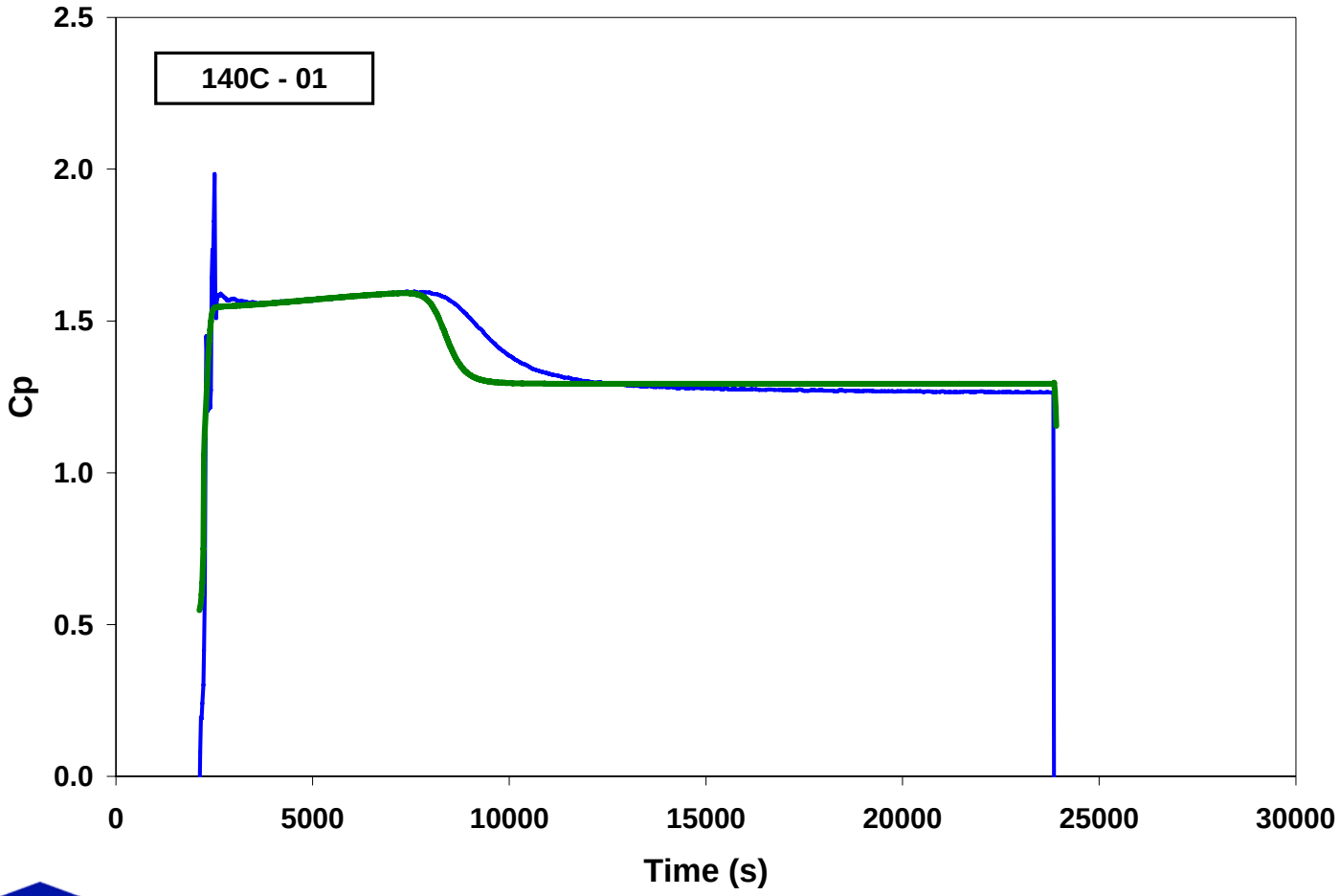
Isothermal Tests – 130C



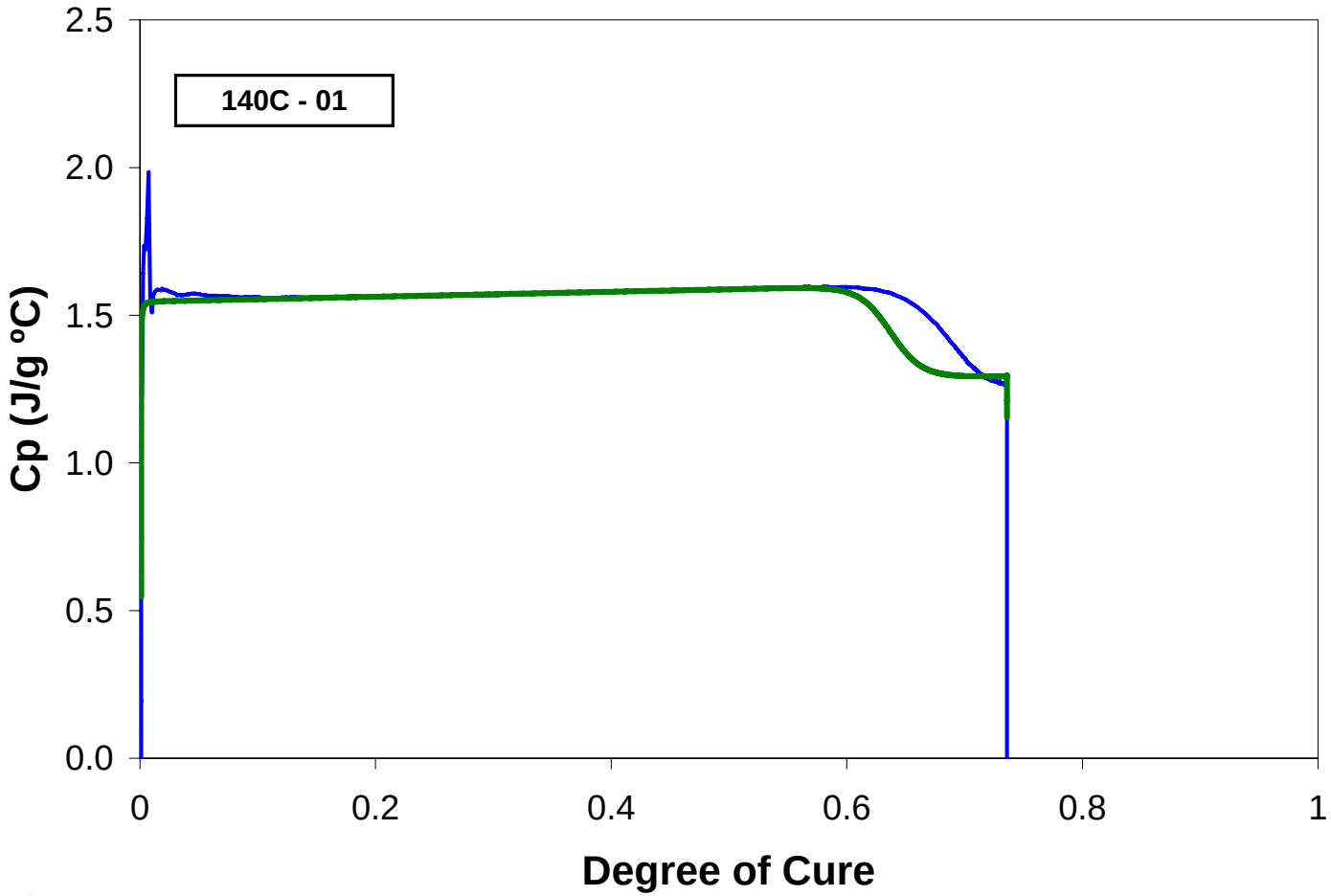
Isothermal Tests – 130C



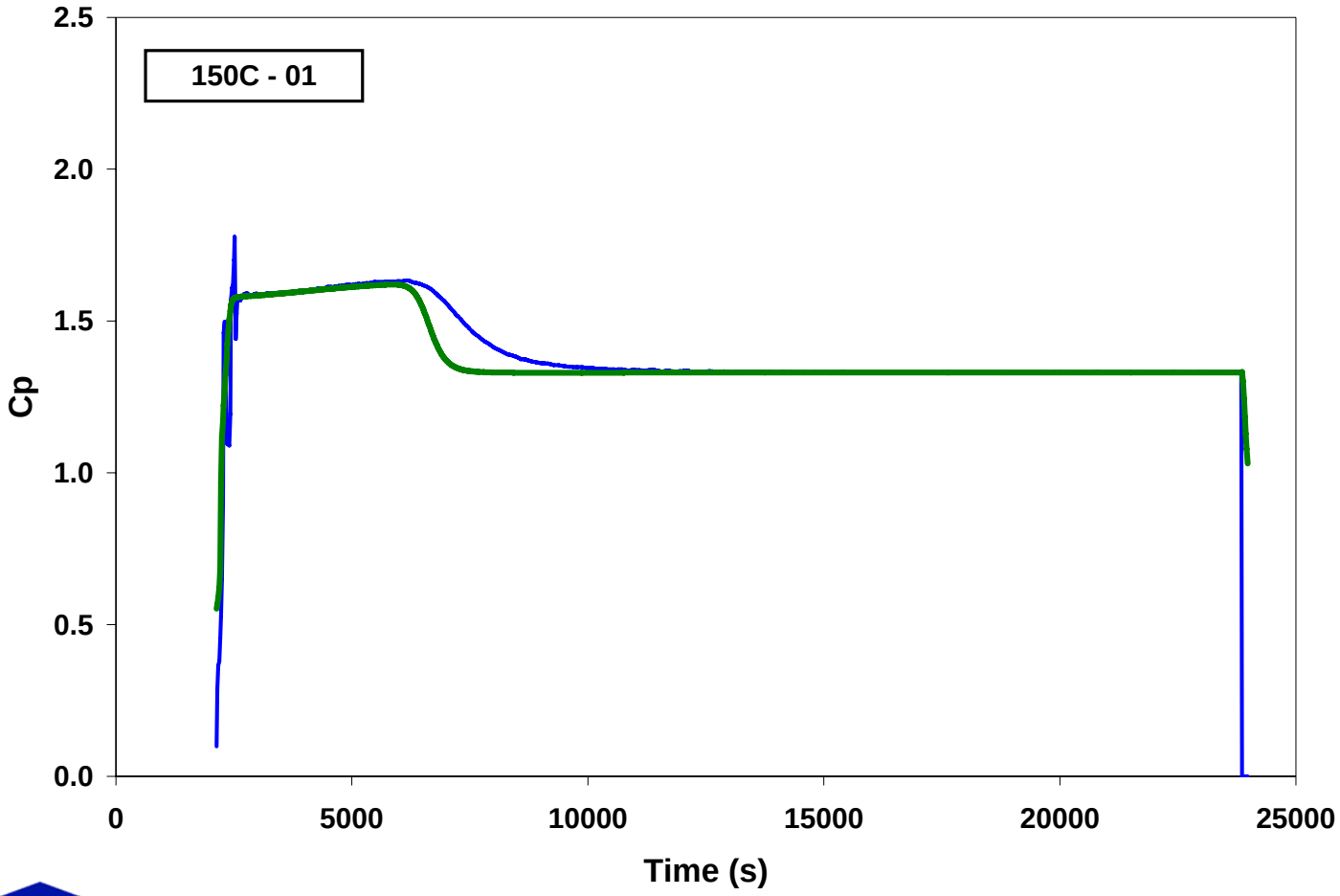
Isothermal Tests – 140C



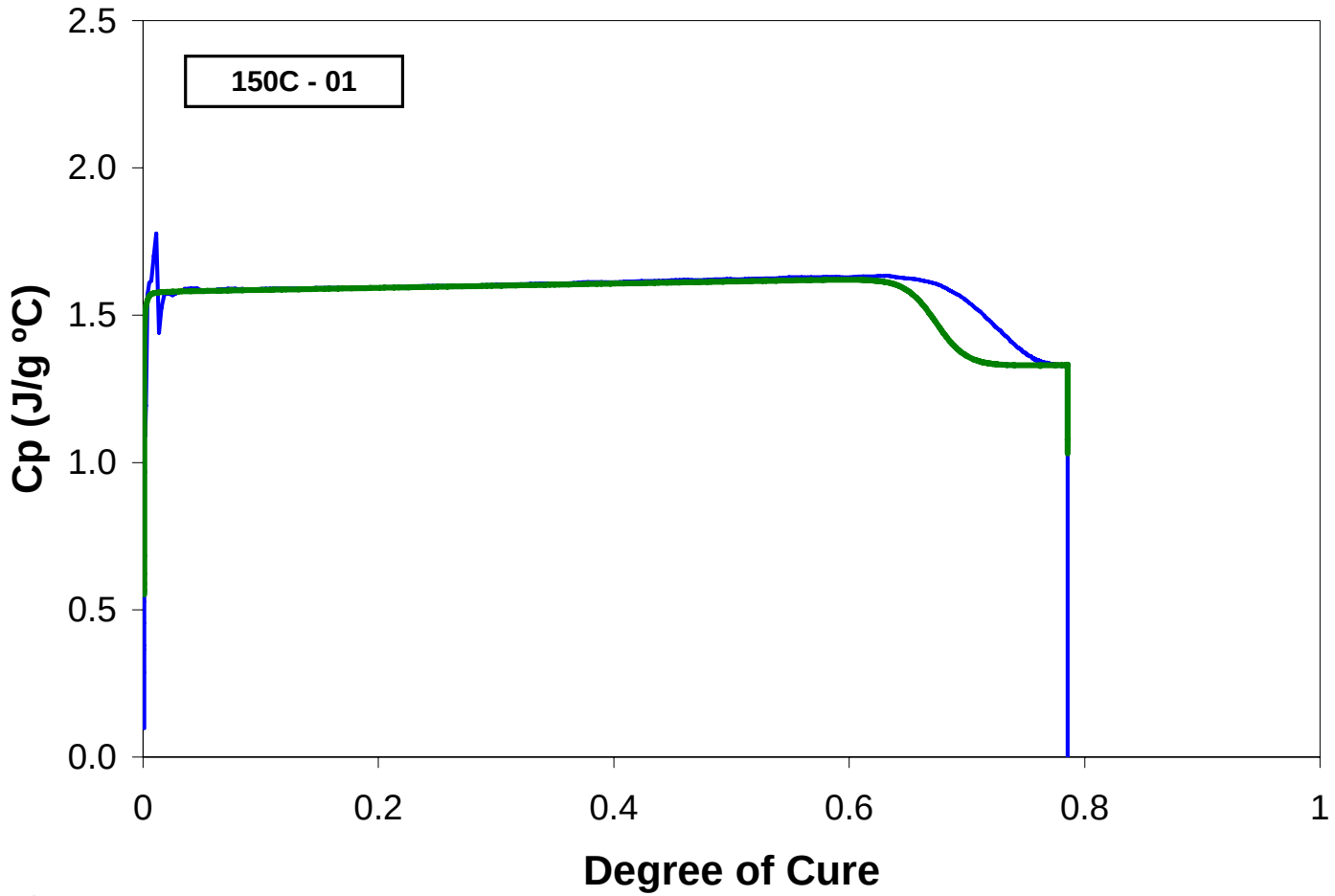
Isothermal Tests – 140C



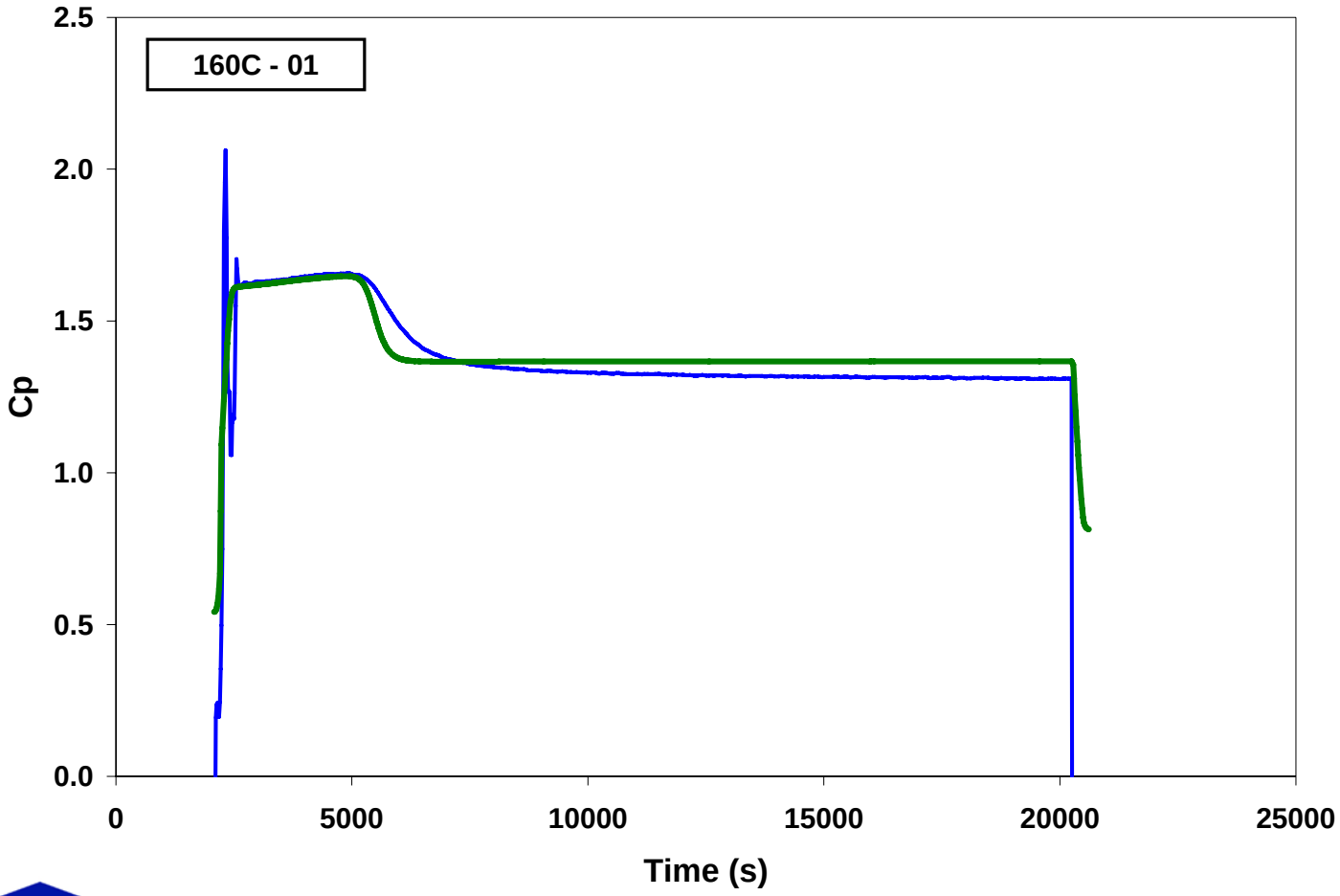
Isothermal Tests – 150C



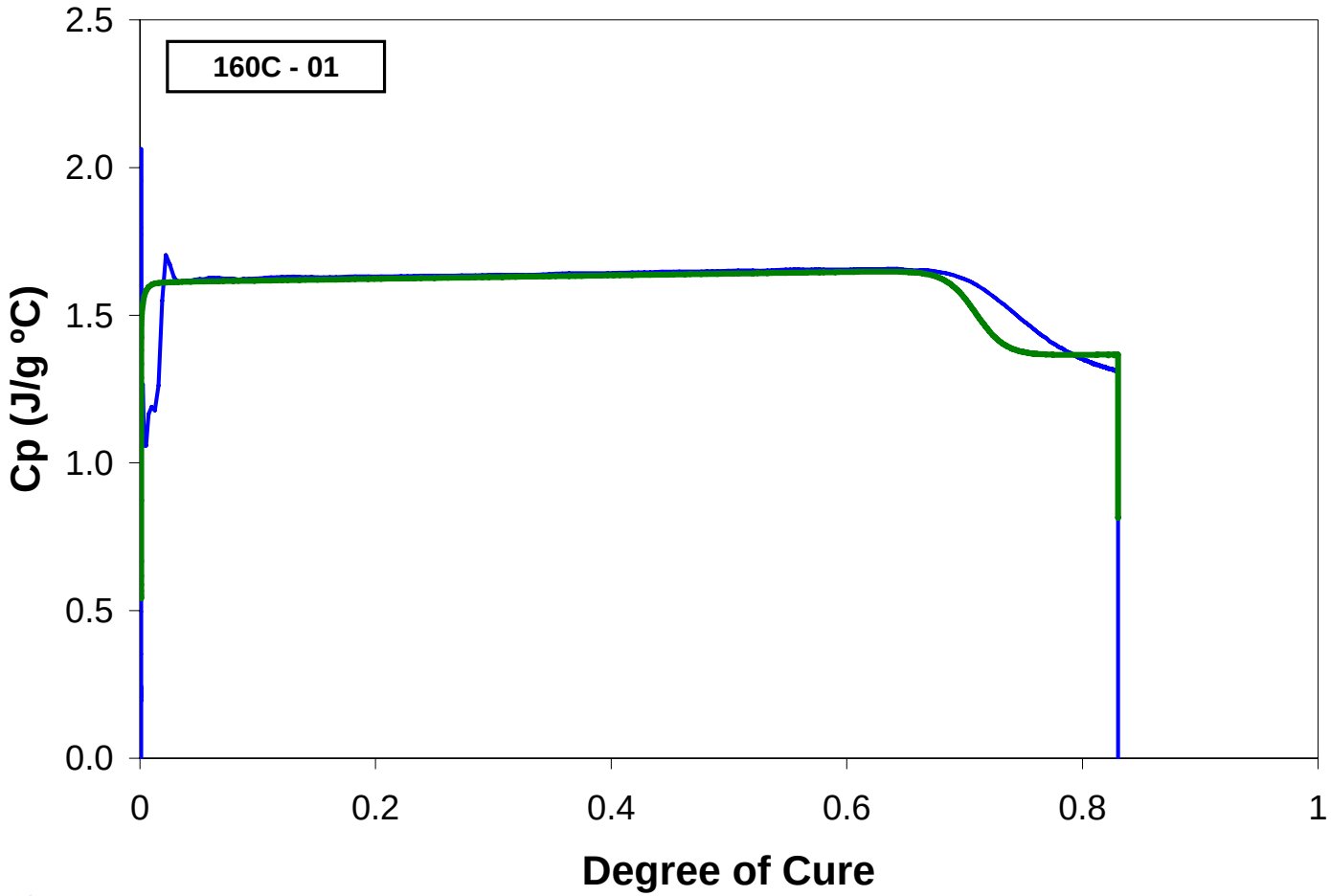
Isothermal Tests – 150C



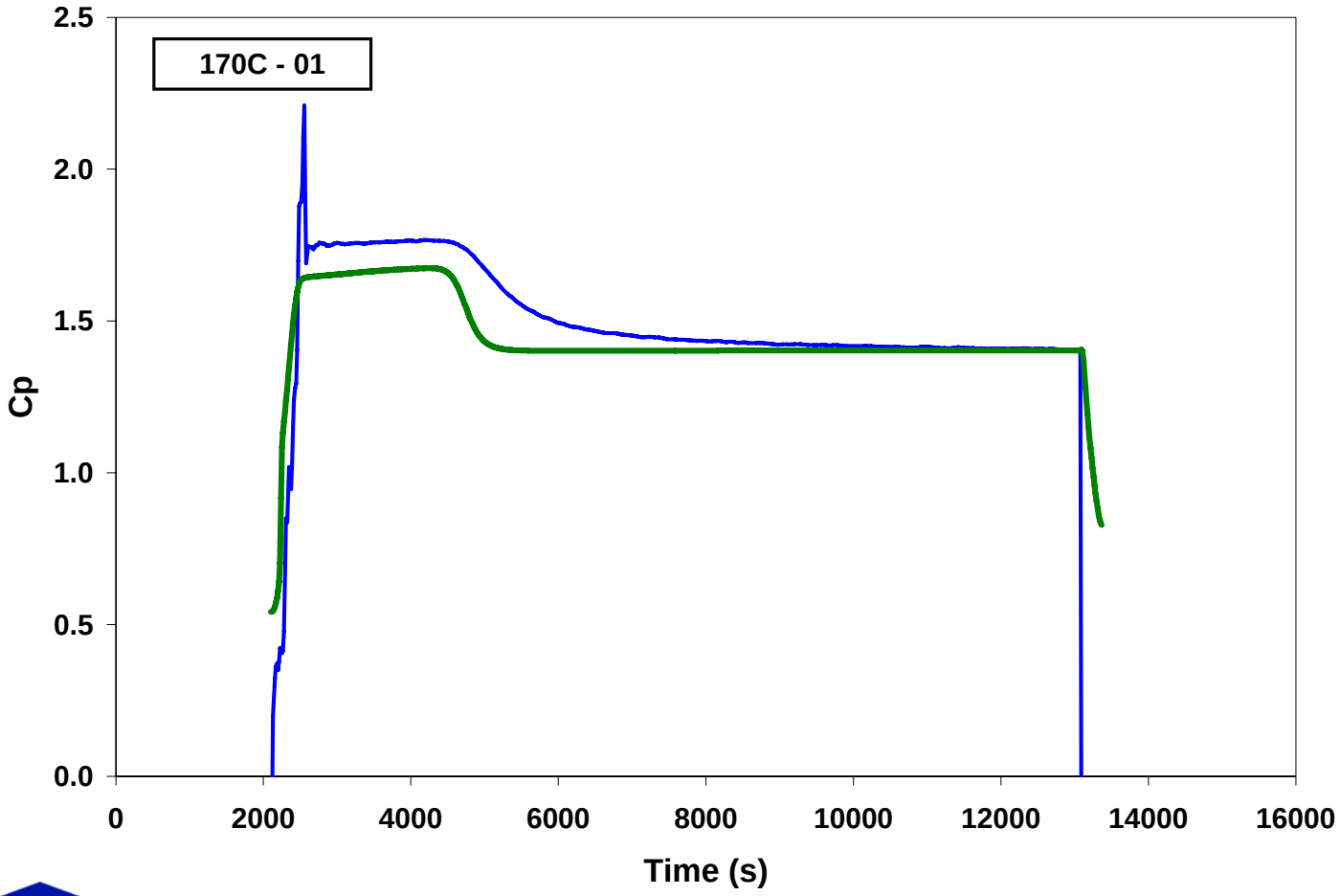
Isothermal Tests – 160C



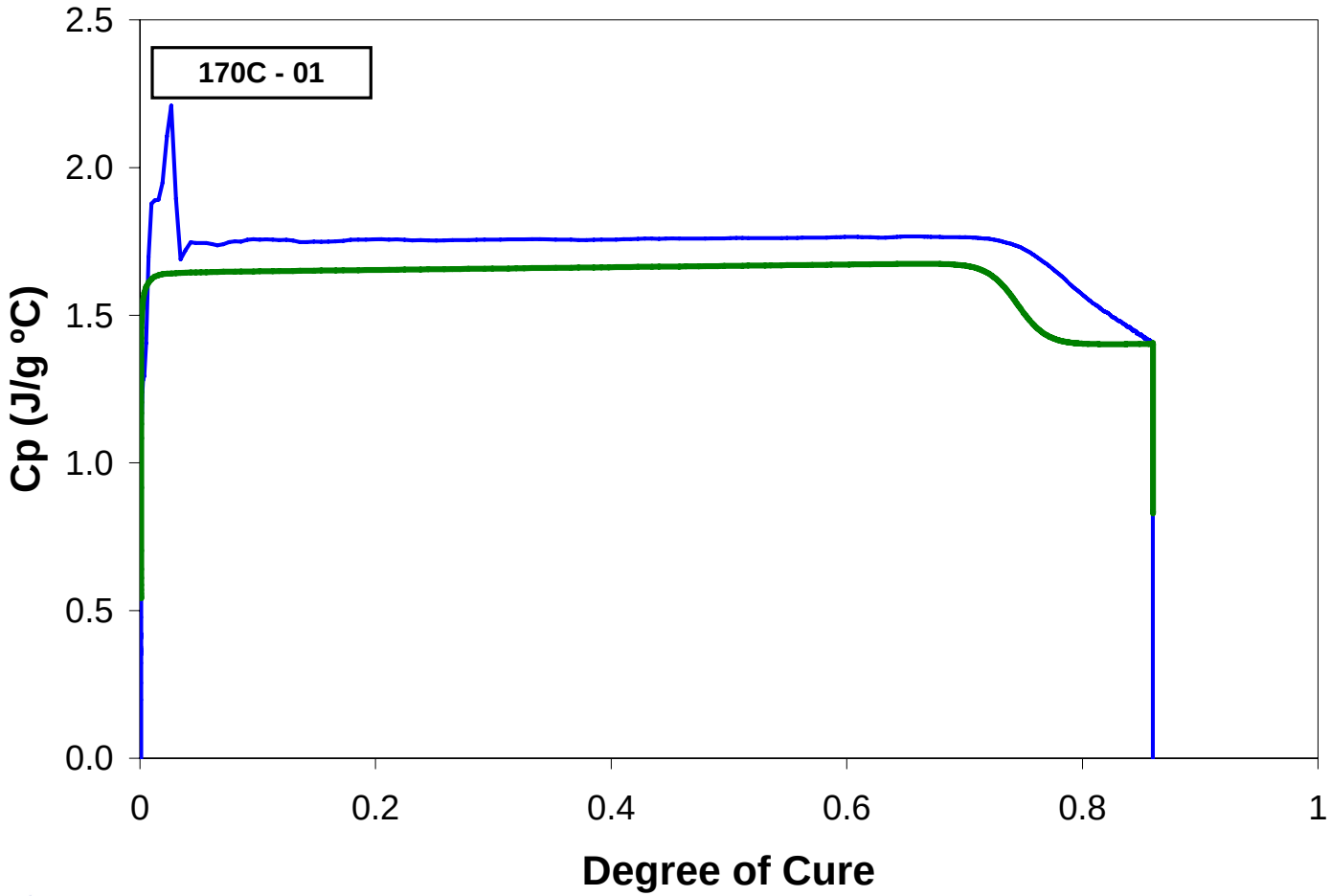
Isothermal Tests – 160C



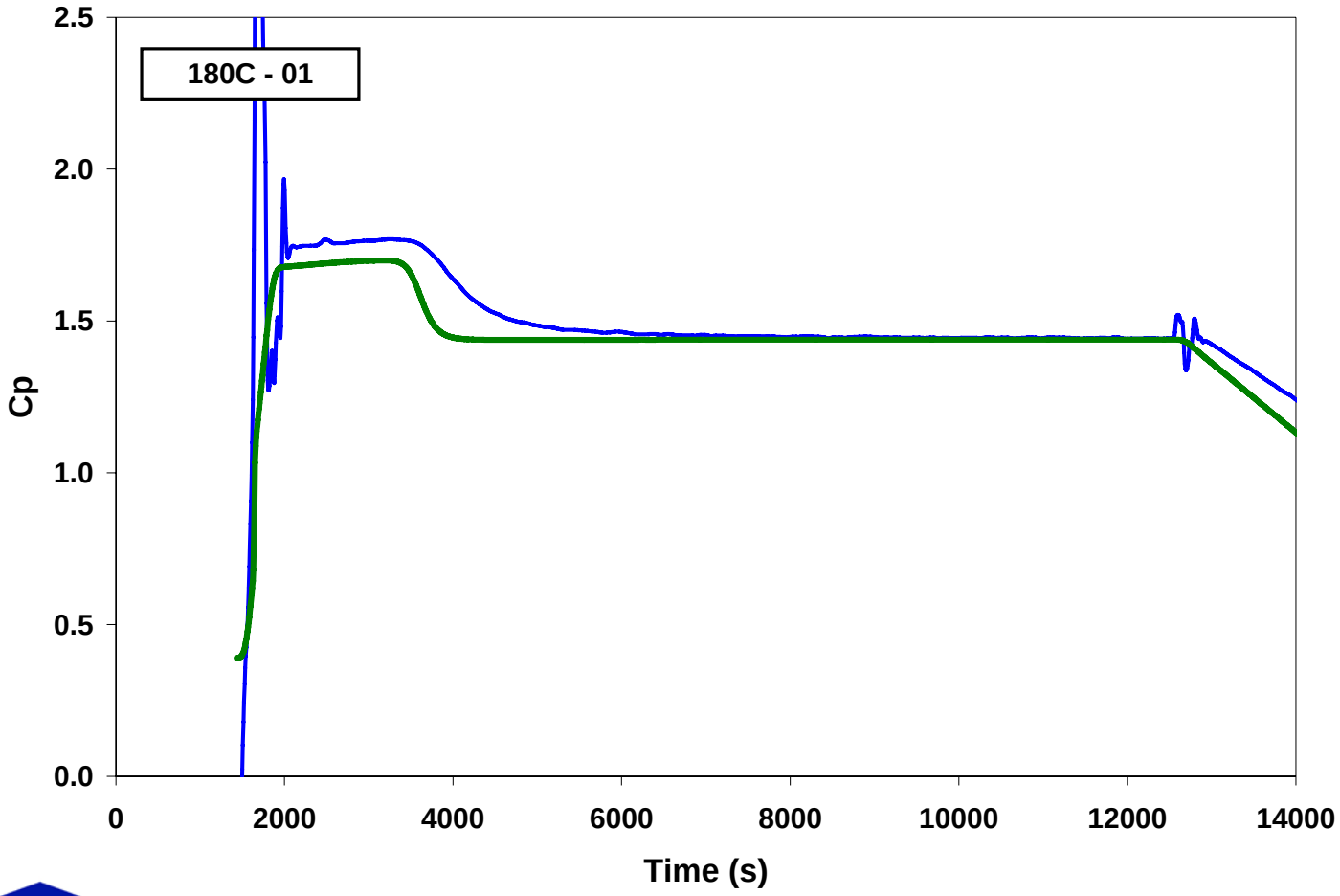
Isothermal Tests – 170C



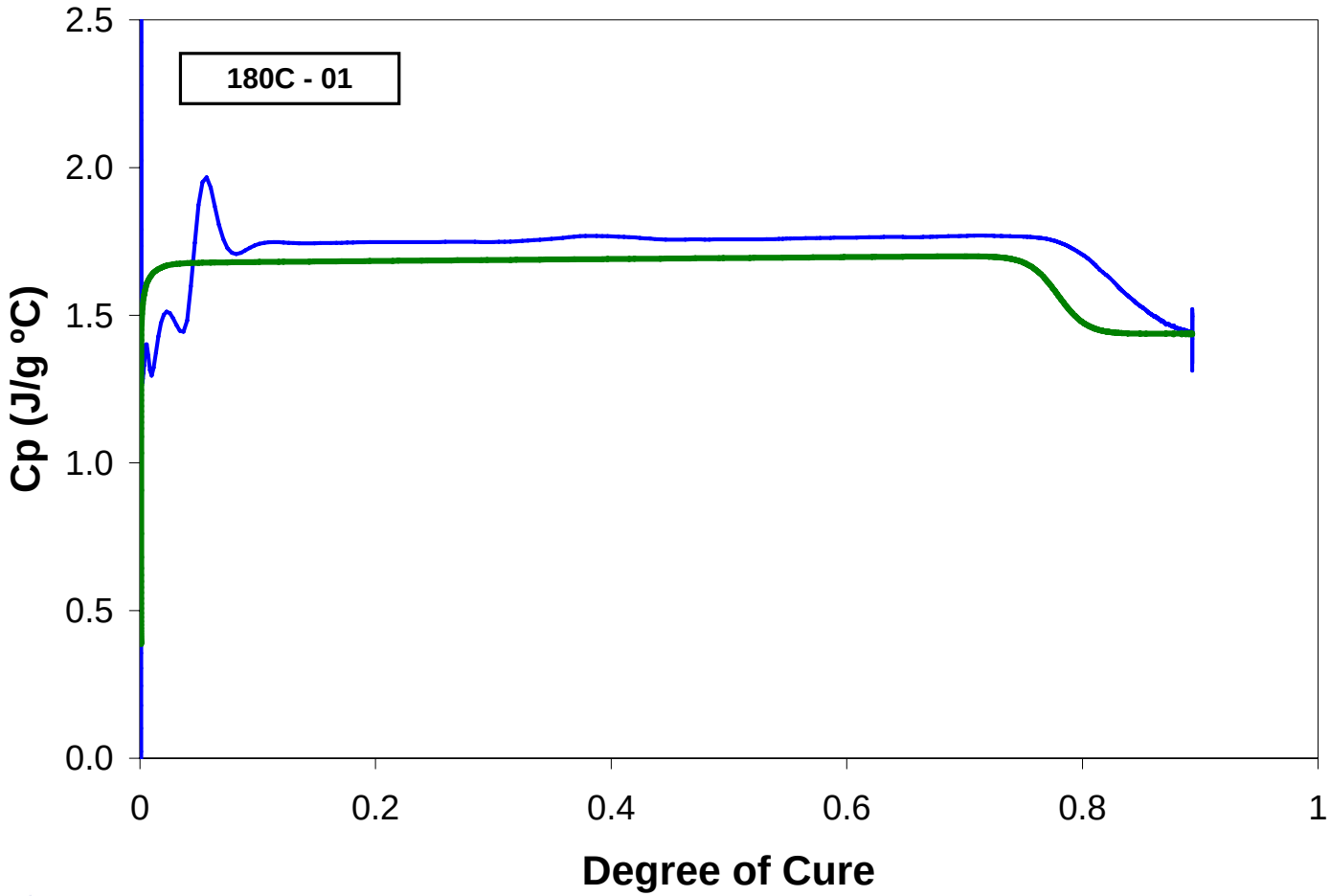
Isothermal Tests – 170C



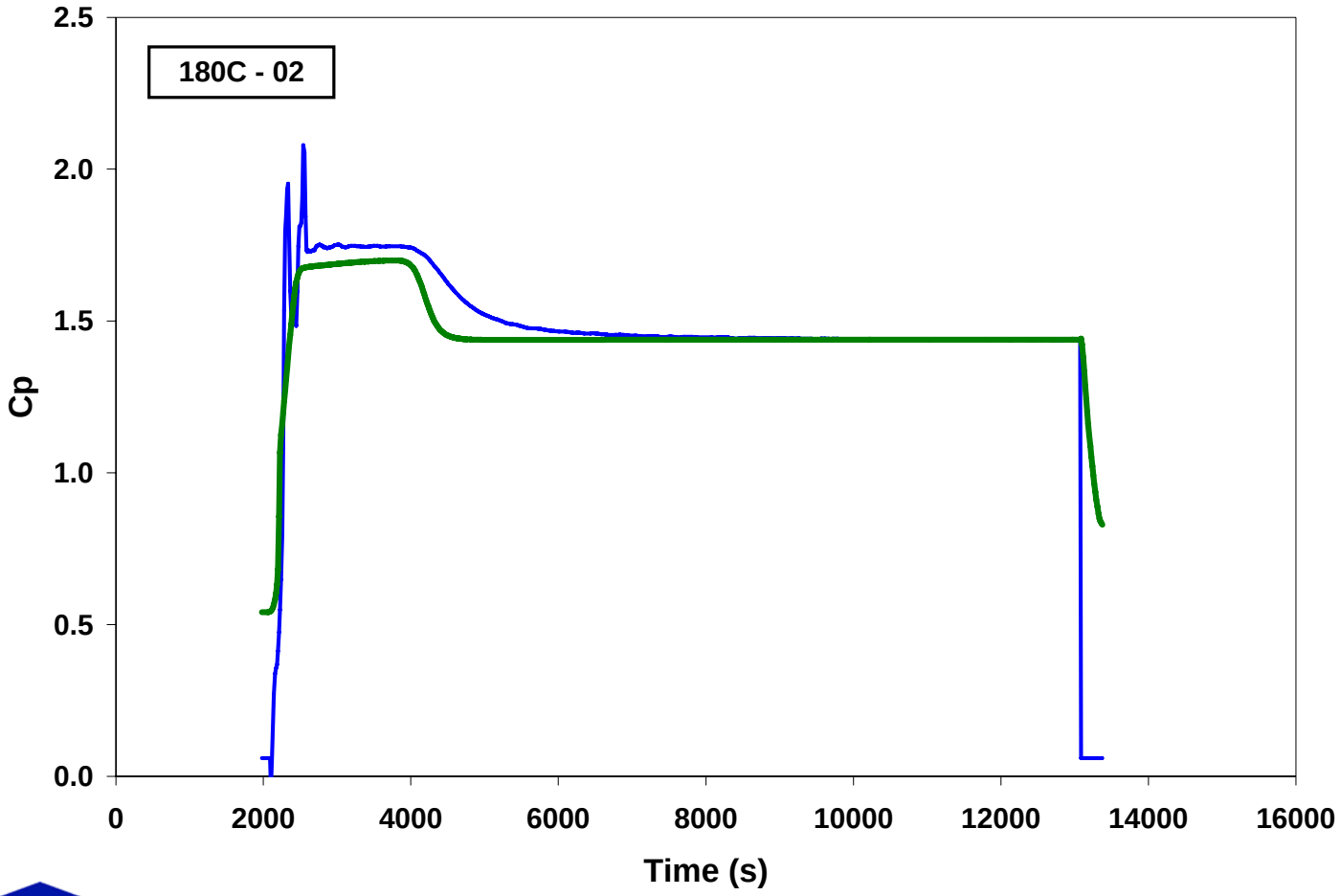
Isothermal Tests – 180C-1



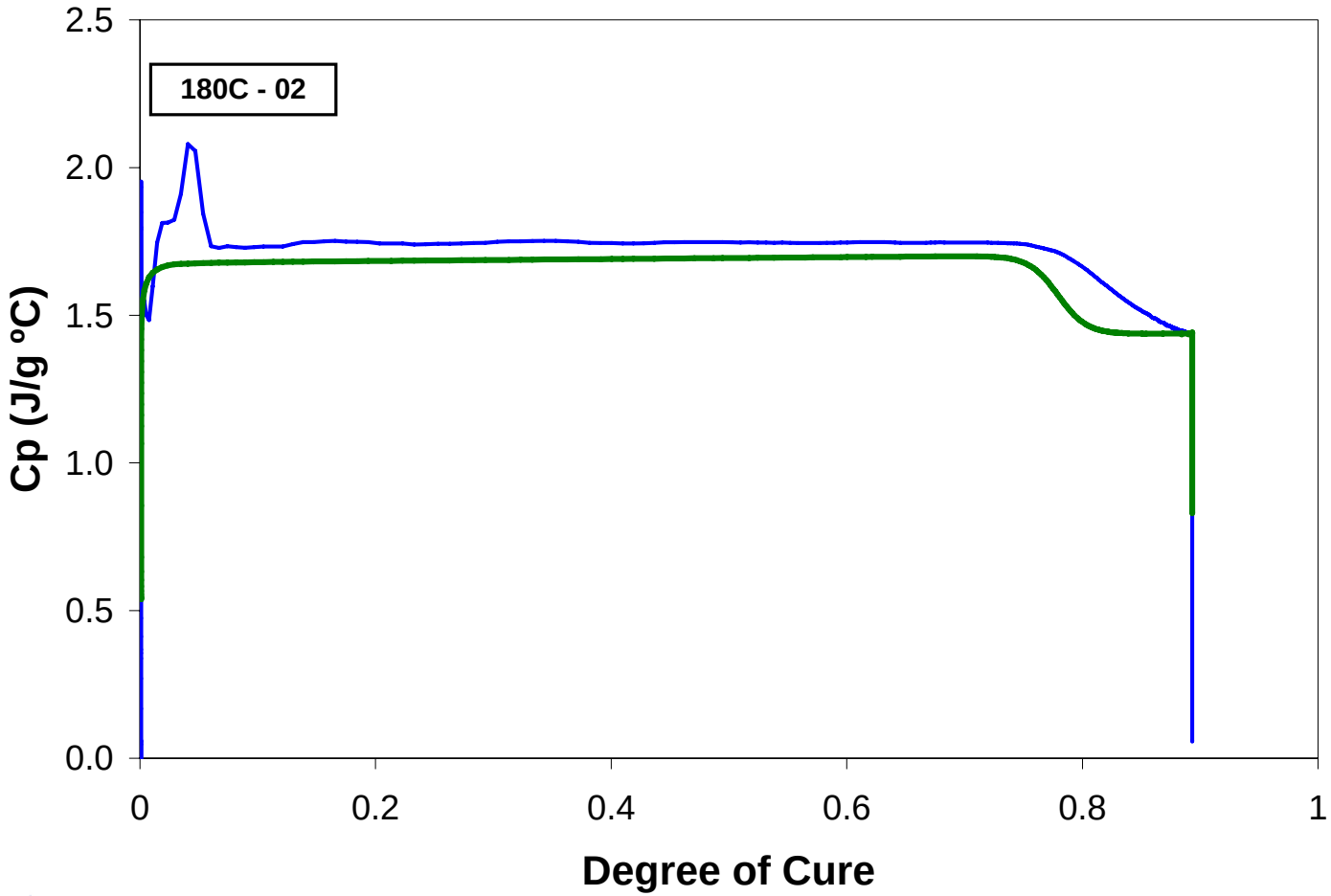
Isothermal Tests – 180C-1



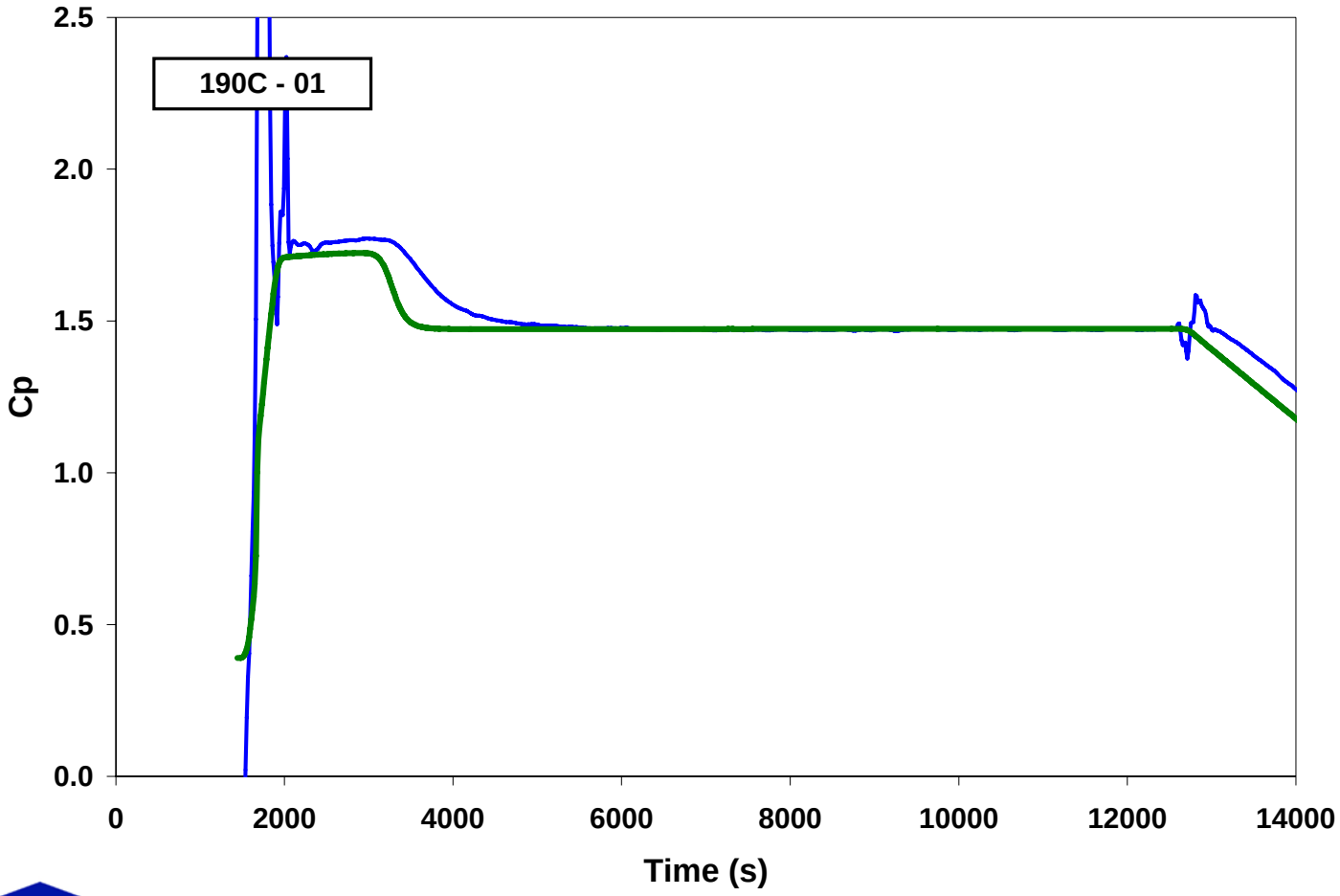
Isothermal Tests – 180C-2



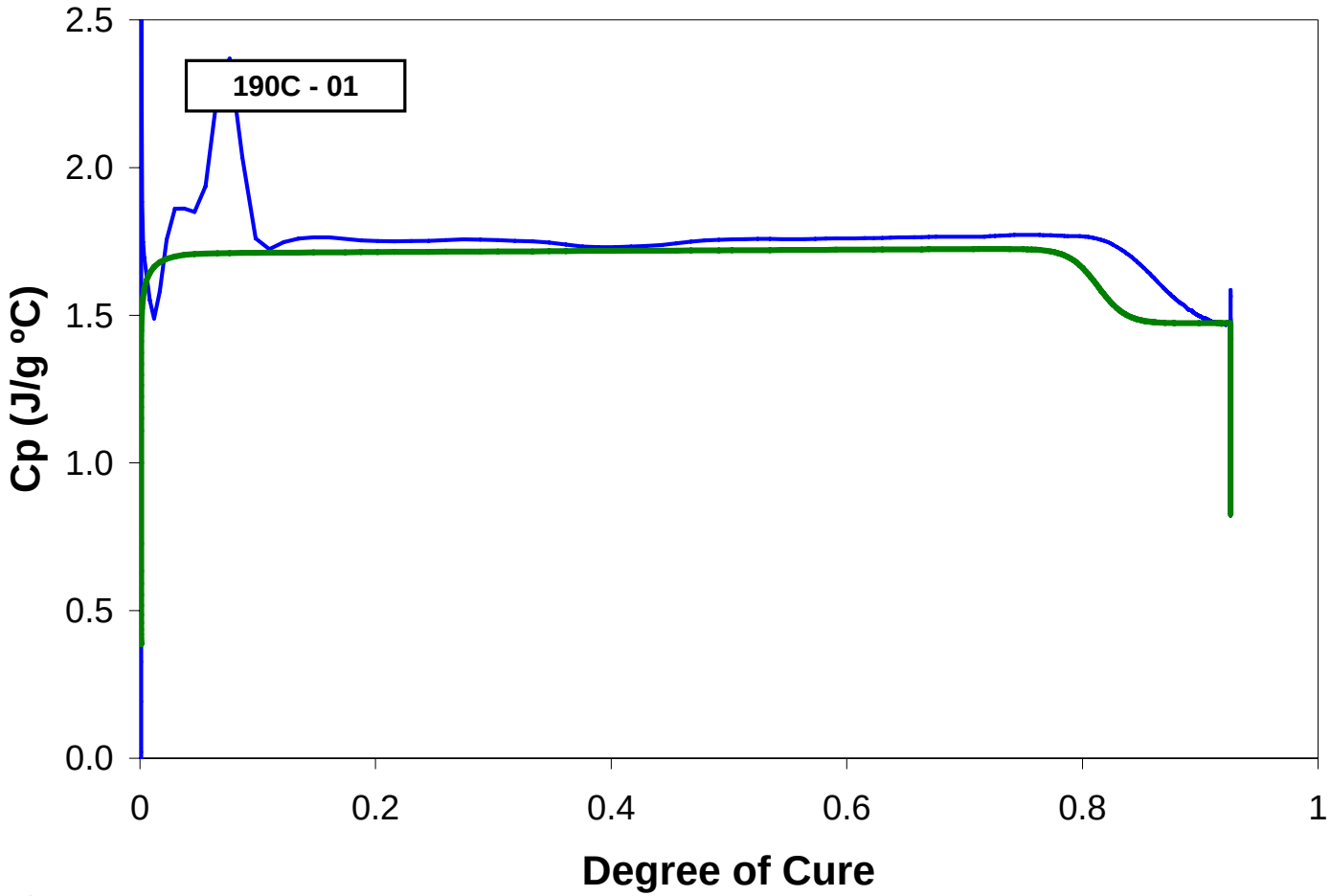
Isothermal Tests – 180C-2



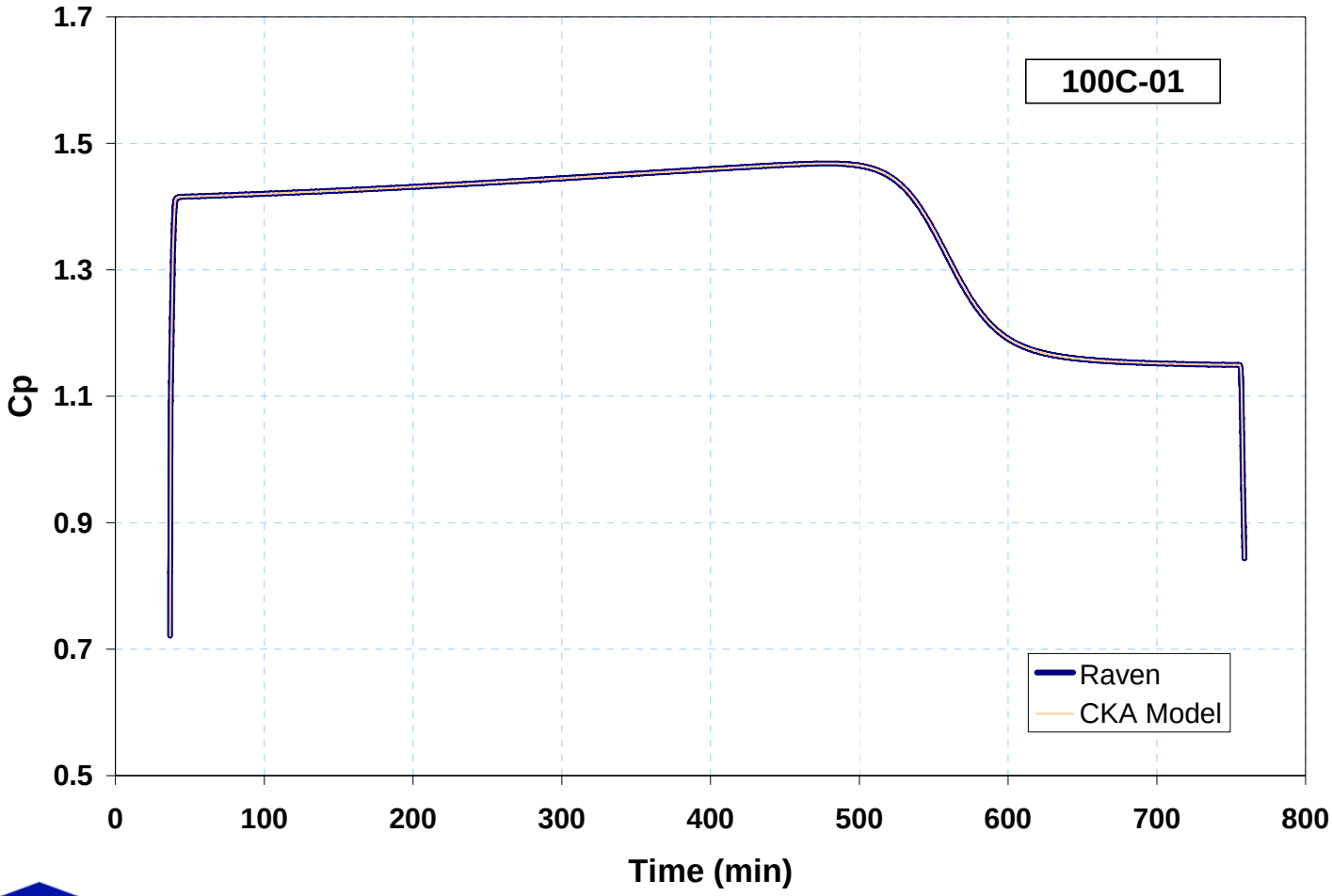
Isothermal Tests – 190C



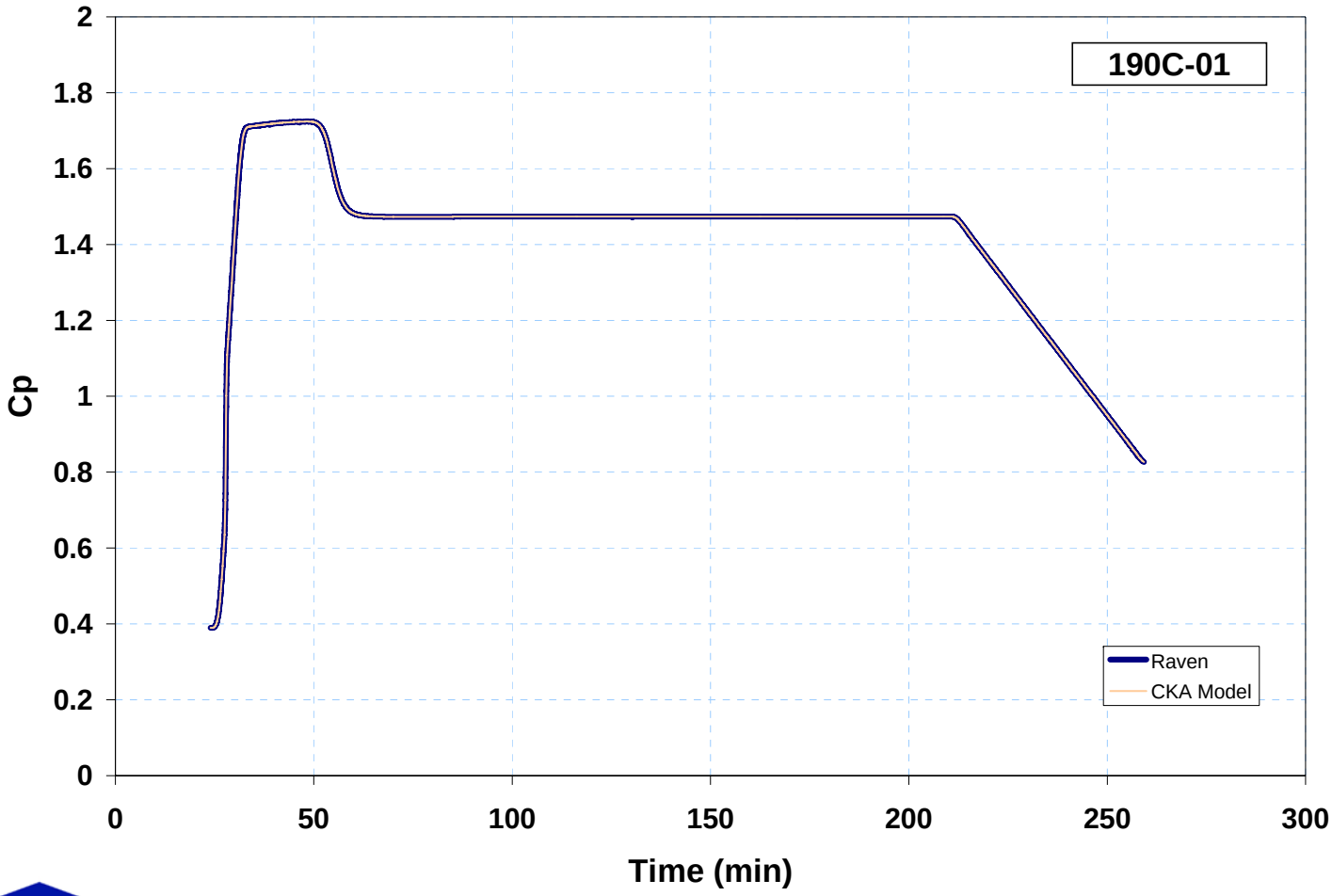
Isothermal Tests – 190C



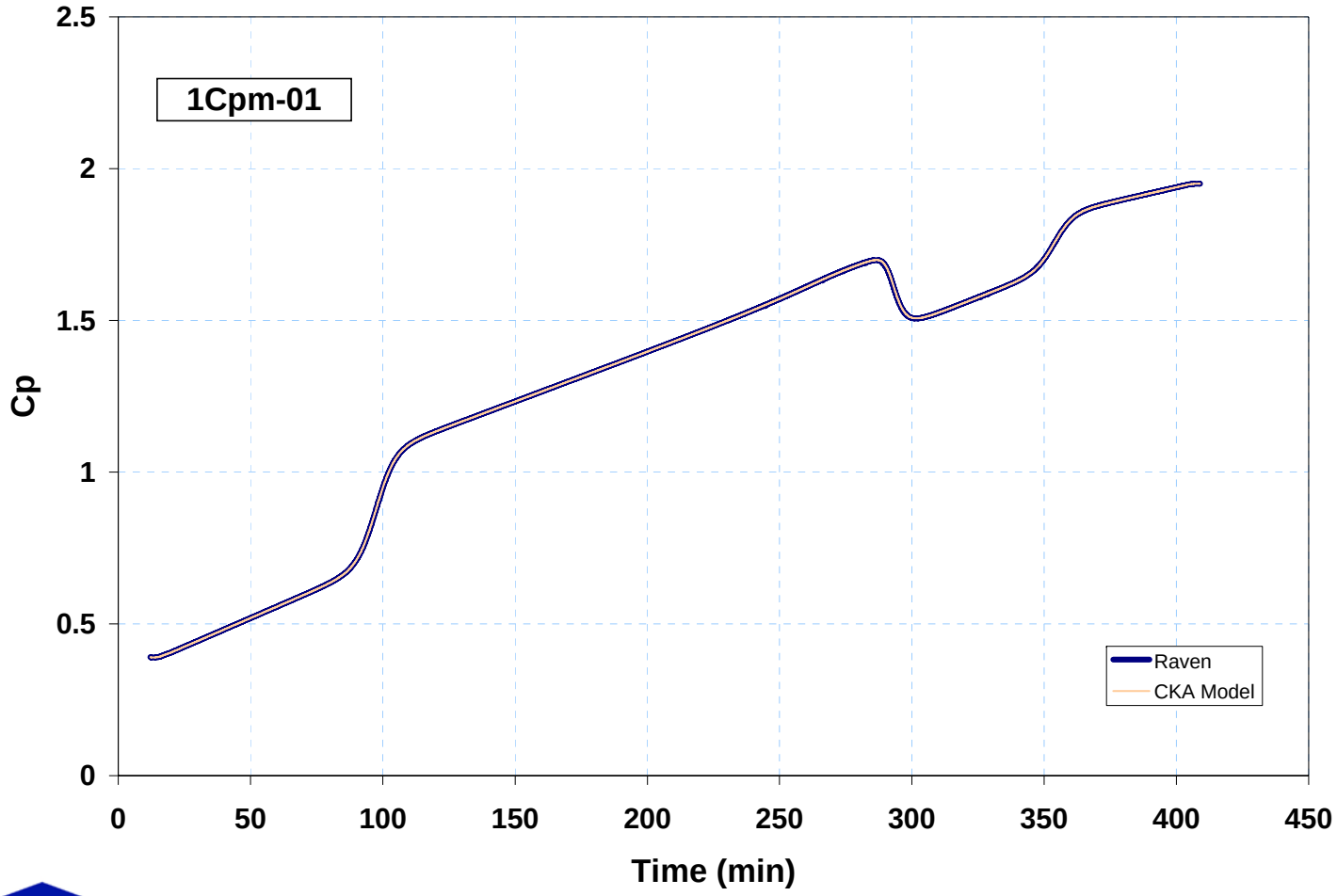
Material Model Verification – 100C



Material Model Verification – 190C



Material Model Verification – 1Cpm



Material Model Verification – 10Cpm