

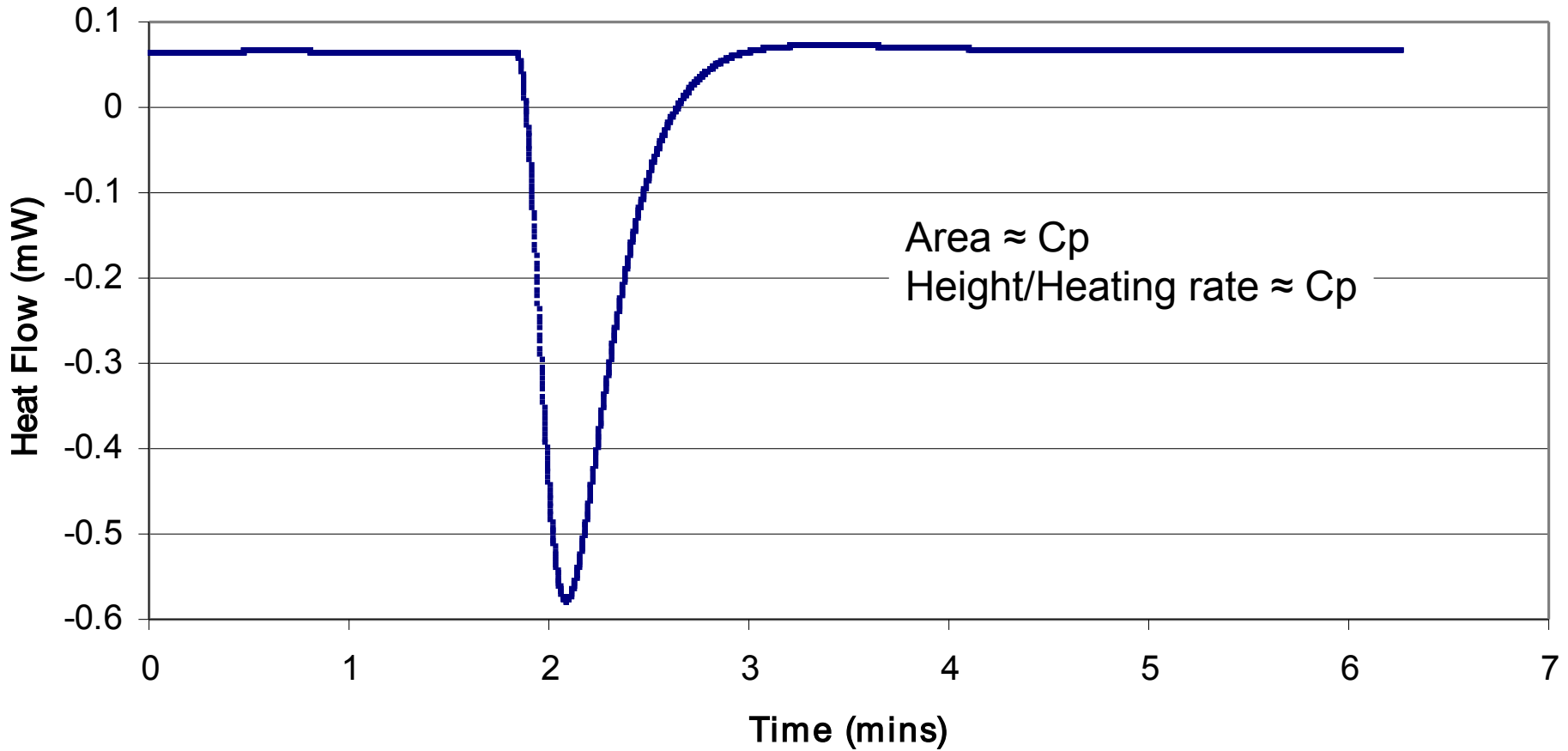
TOPEM[®] - Measuring program and evaluation parameters

Steve Sauerbrunn
Sept 18, 2006

Agenda

- Creating a TOPEM Method
- Correlation of Heat Flow to Impulses
- TOPEM Evaluation
- Cp Frequency Evaluation

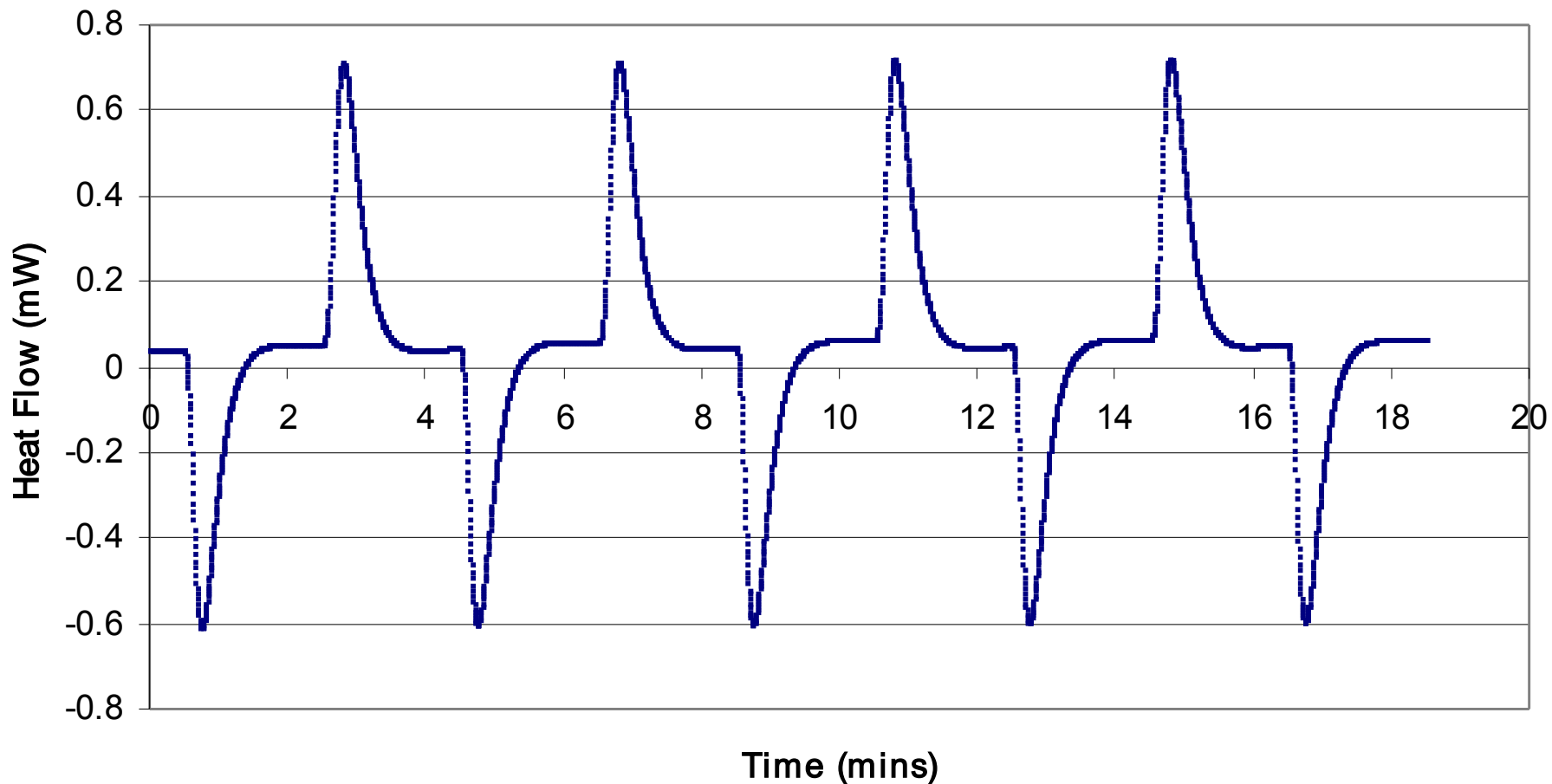
Sapphire with a 1 C Step



Cp is Correlated over Several Impulses

Reversing Heat Flow

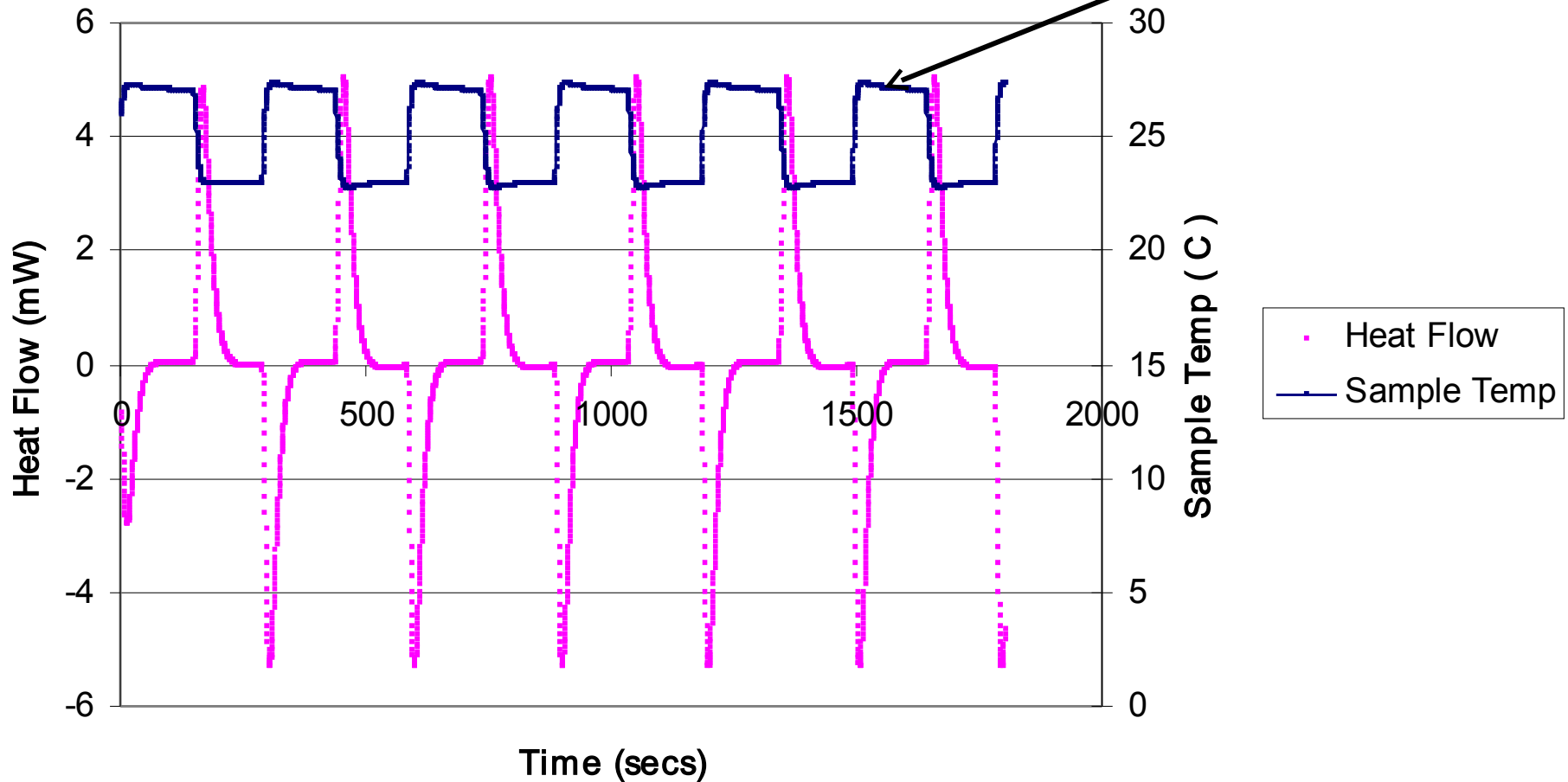
Sapphire 9 Impulses



Sample Temperature

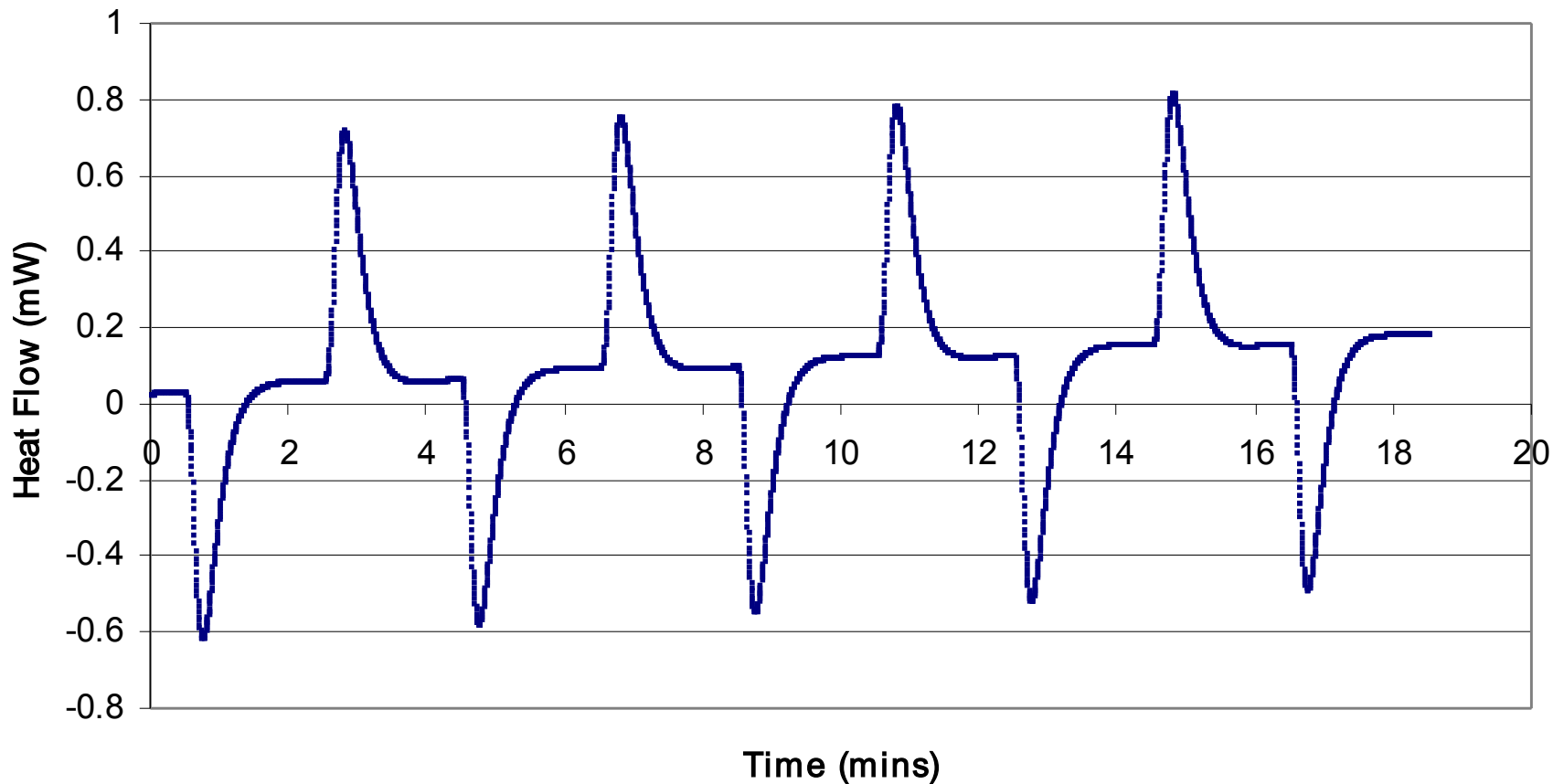
TOPEM of Sapphire at 25 C

True Sample Temp
not Square Wave



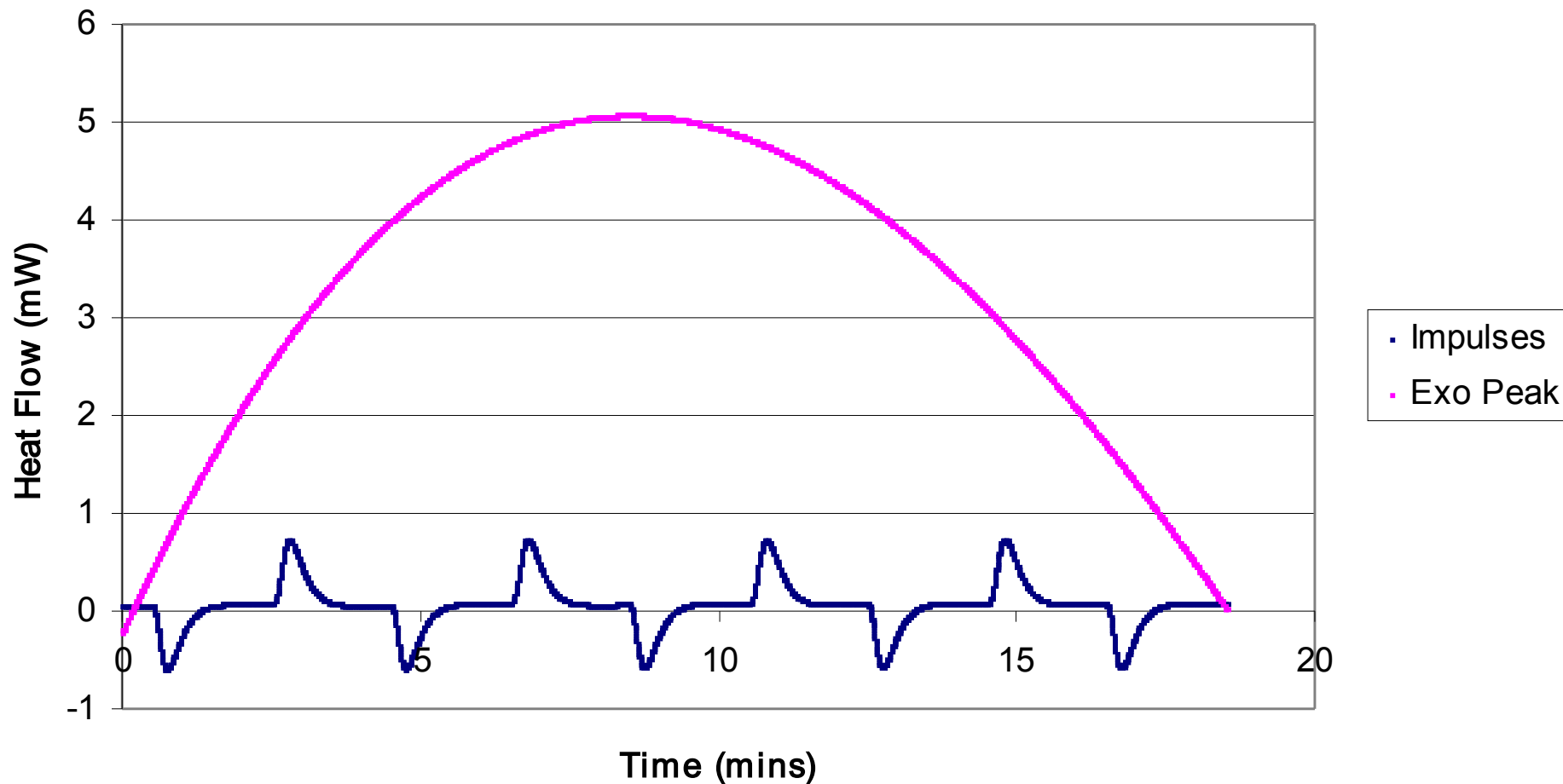
Slope Correlated over Several Impulses

Cp with an Exothermic Slope



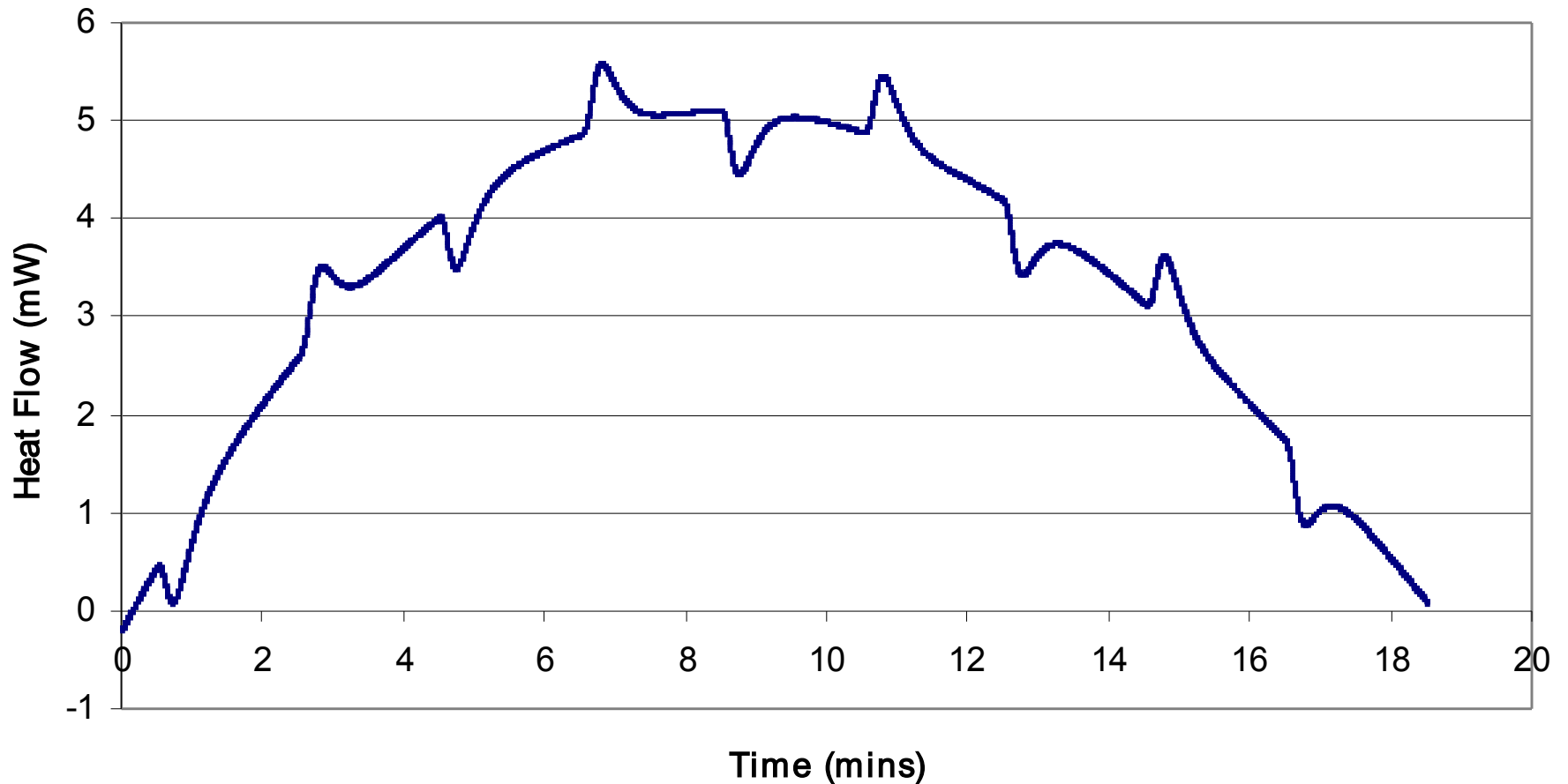
Simulated Exothermic Reaction

TOPEM w Exothermic Peak



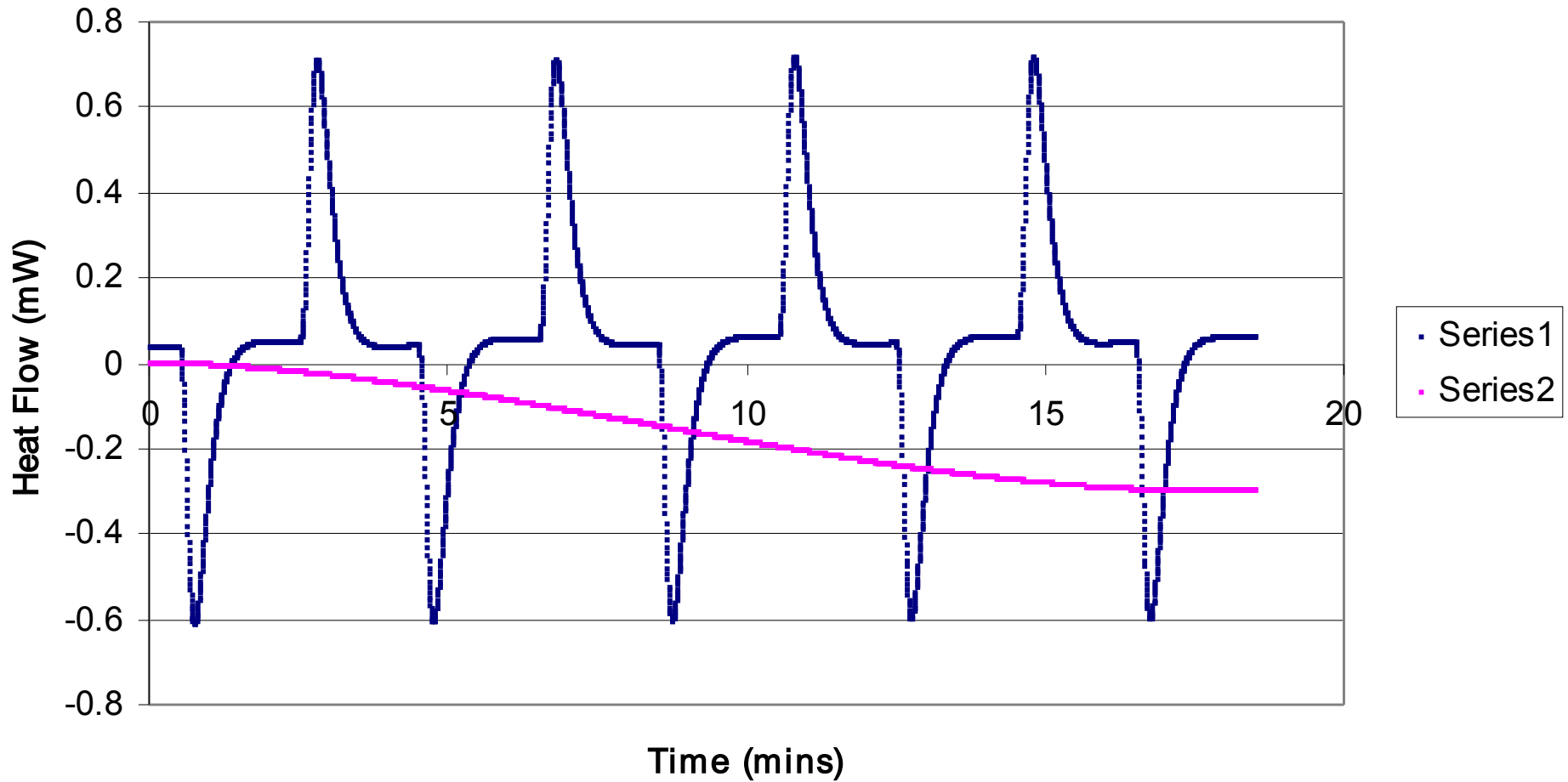
Peak is Correlated Non Reversing Heat Flow

TOPEM w Exothermic Peak



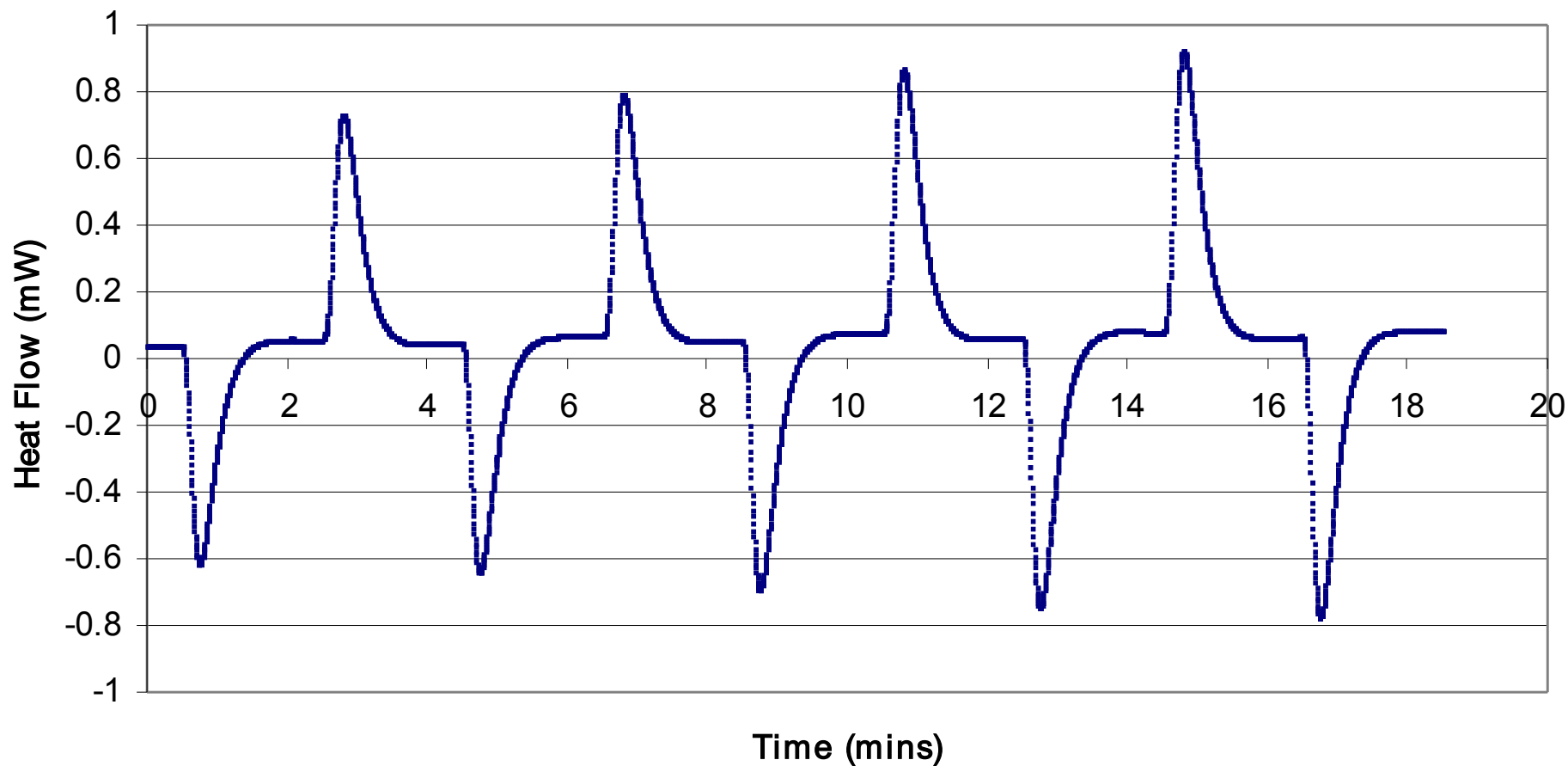
Glass Transition

TOPEM w Glass Transition



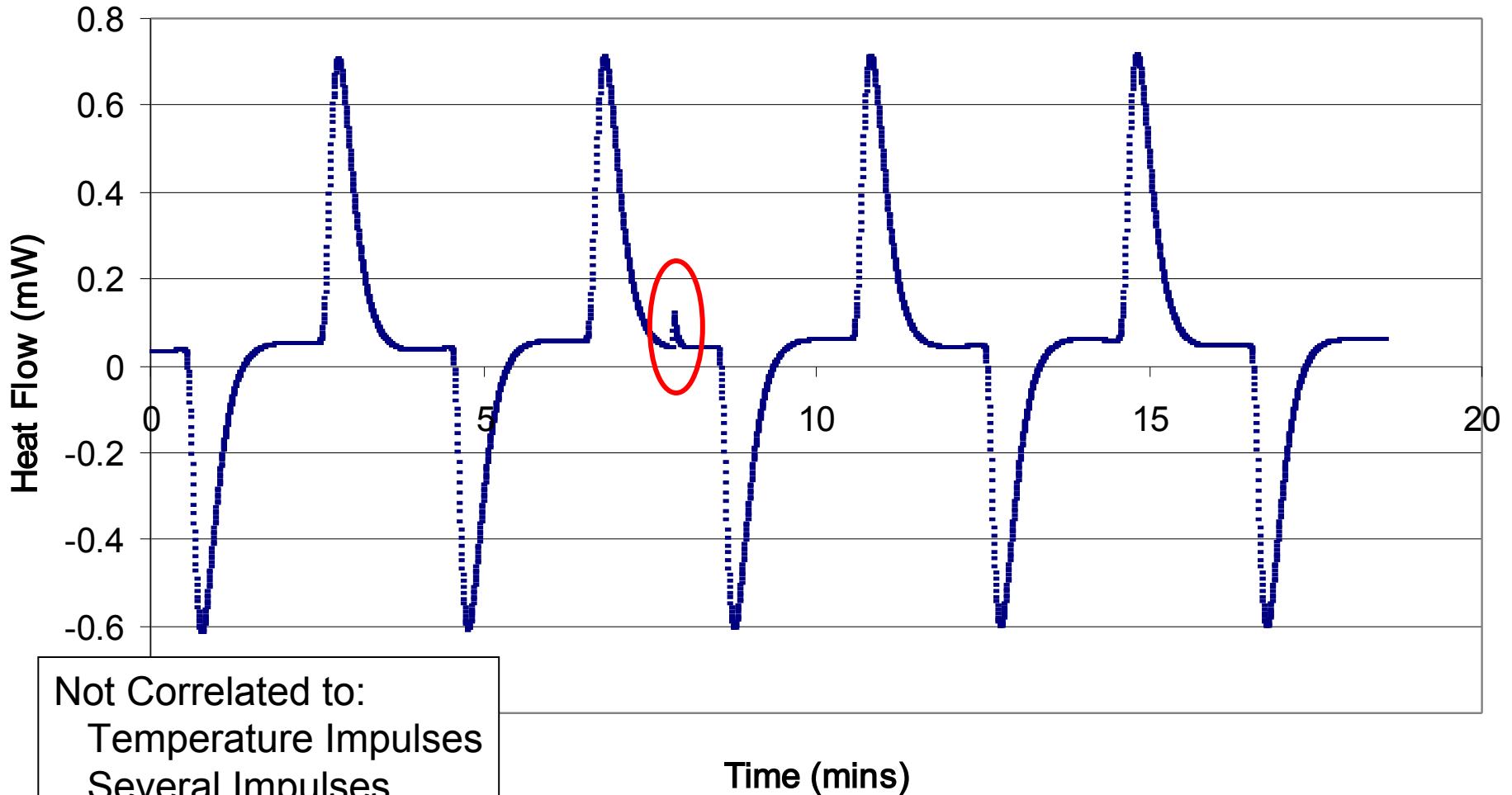
Correlated with Temperature Peaks

TOPEM w Glass Transition



Noise is Not Correlated removed as Instrument Effect

Sapphire plus Noise Spike

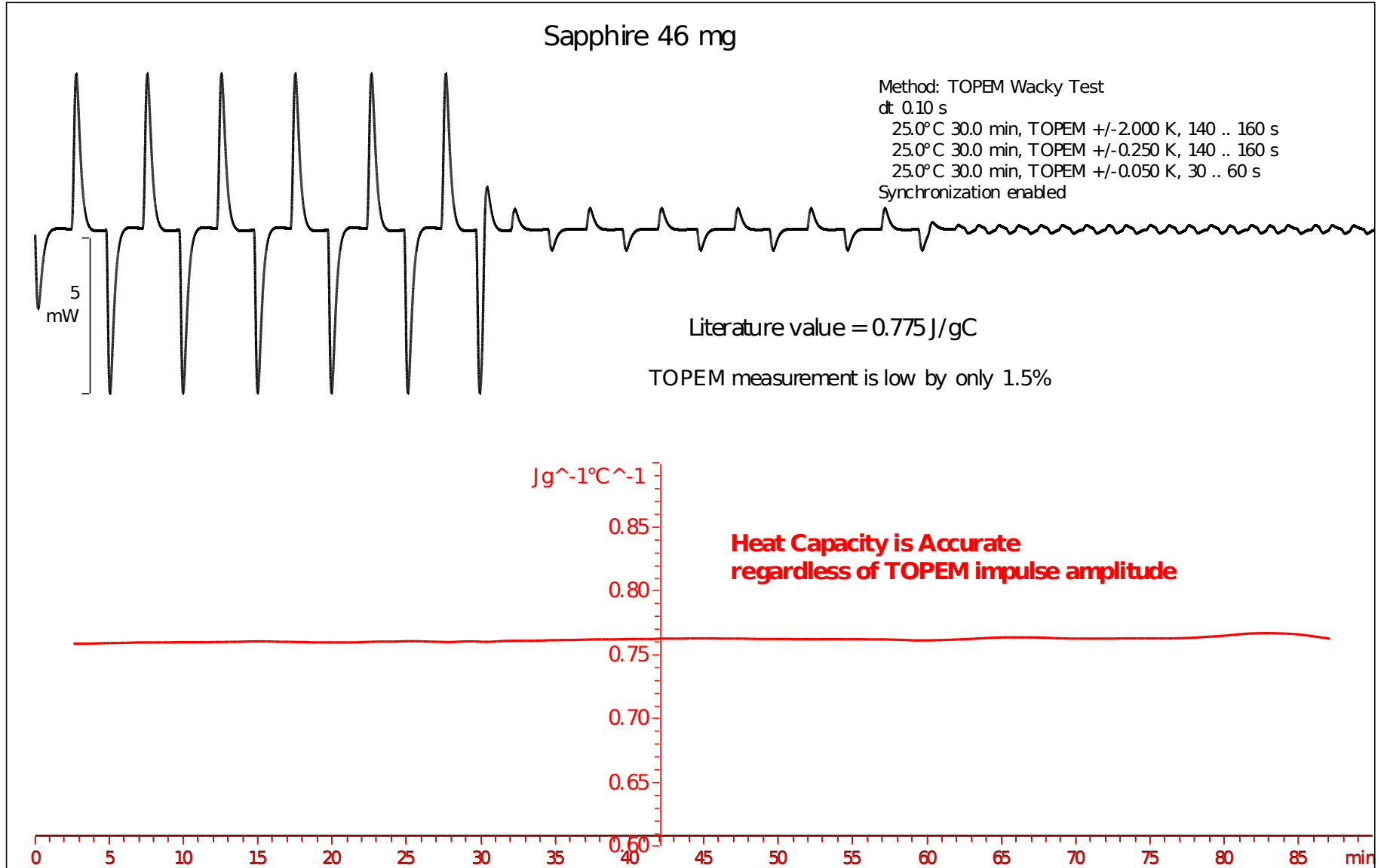


Changing Parameters in a Method

^exo

Changing TOPEM Impulse Amplitude

21.08.2006 10:07:02



DEMO Version

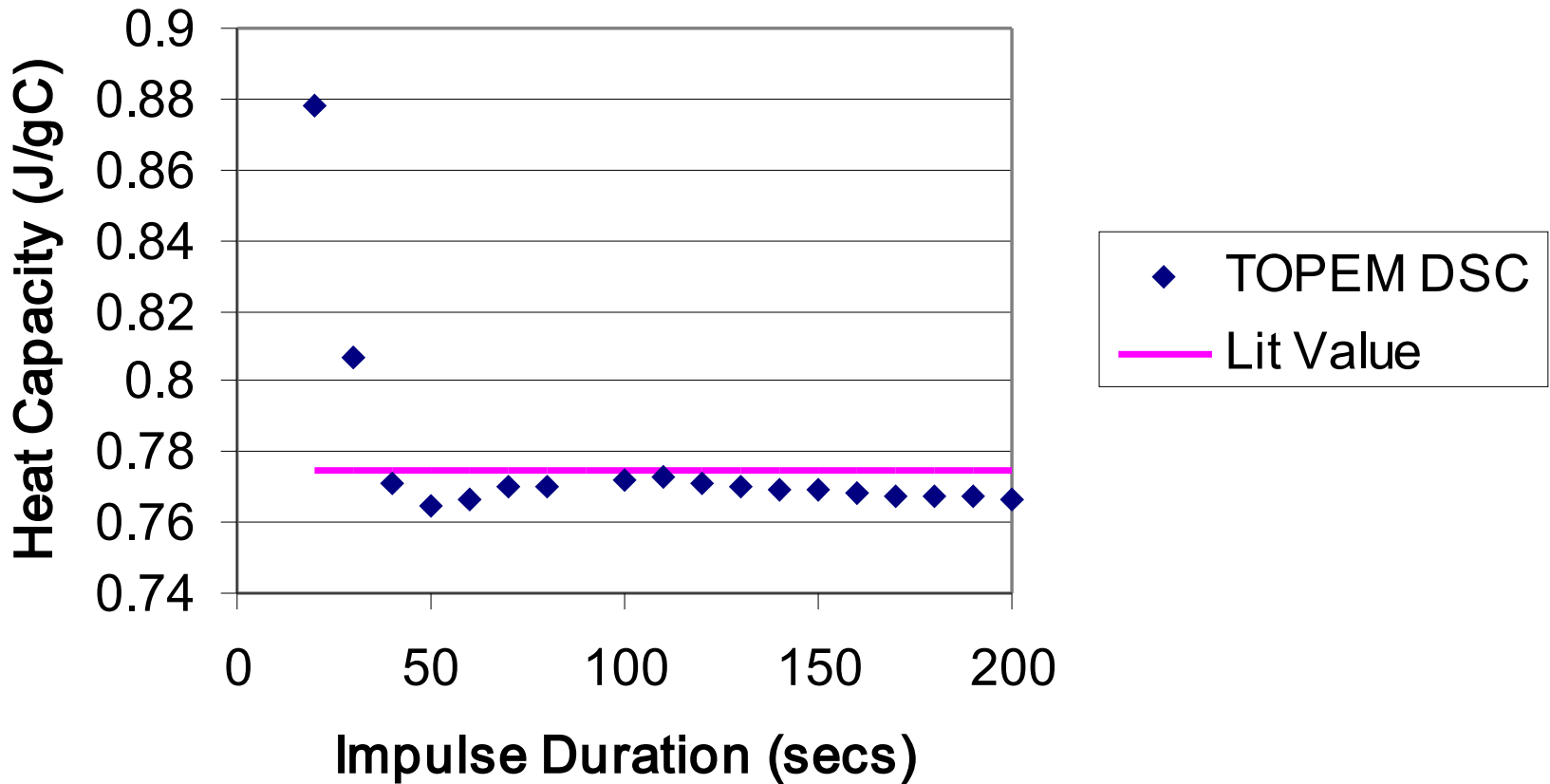
STAR[®] SW 9.0'

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Actual

Affect of Impulse Duration on Cp Value

Sapphire Heat Capacity



Method Guidelines

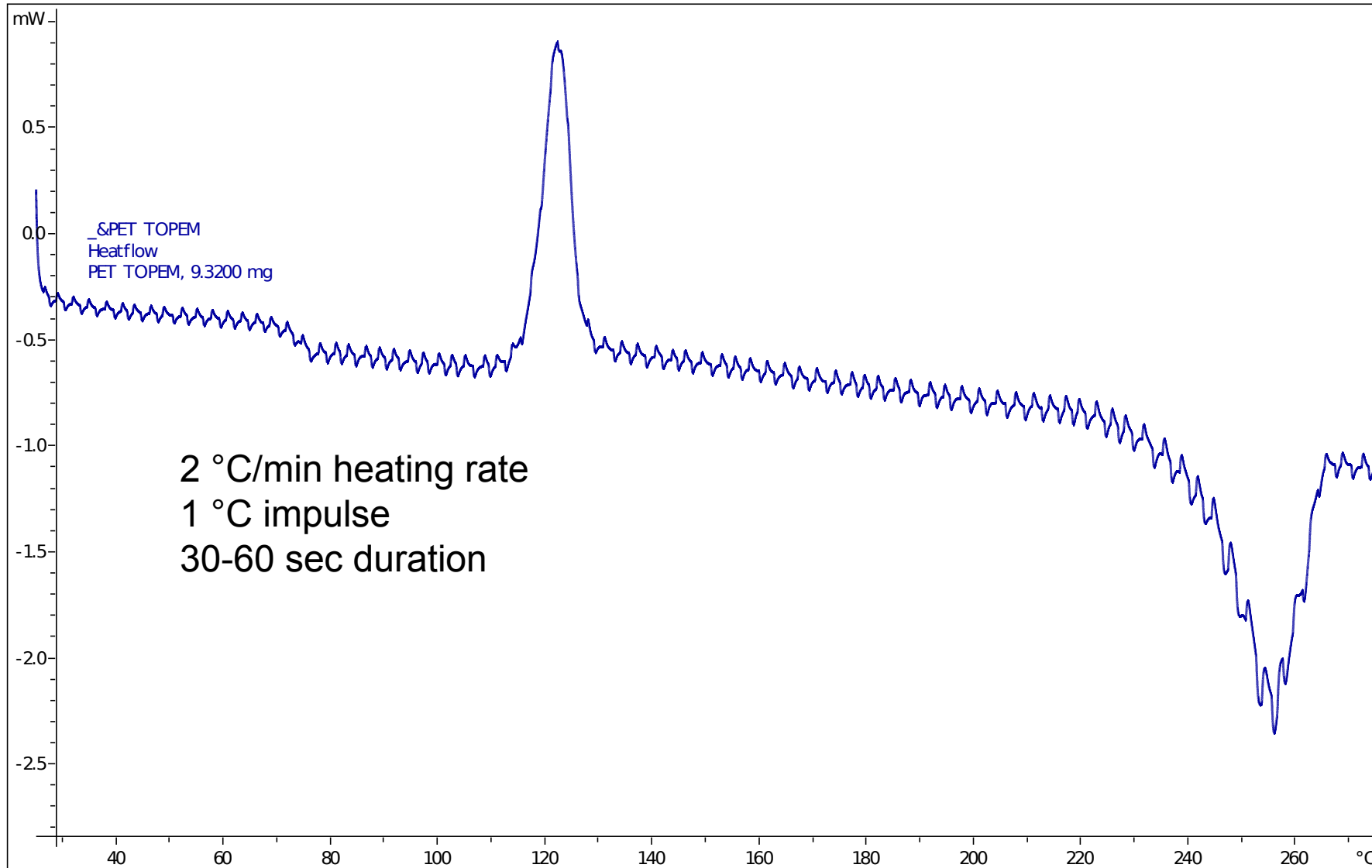
- 5 to 7 impulses per transition
- Duration = 10X time to peak
 - 15 to 30 secs for FRS5
 - 30 to 60 secs for HSS7
- Heating Rate
 - Calculated from duration and narrowest peak
- Amplitude
 - 1 C for most scans
 - 2 to 5 C for small T_g
 - 0.05 to 0.1 C for melting

TOPEM of PET at 2 °C/min

TOPEM PET After BL Subtraction

28.08.2006 07:58:18

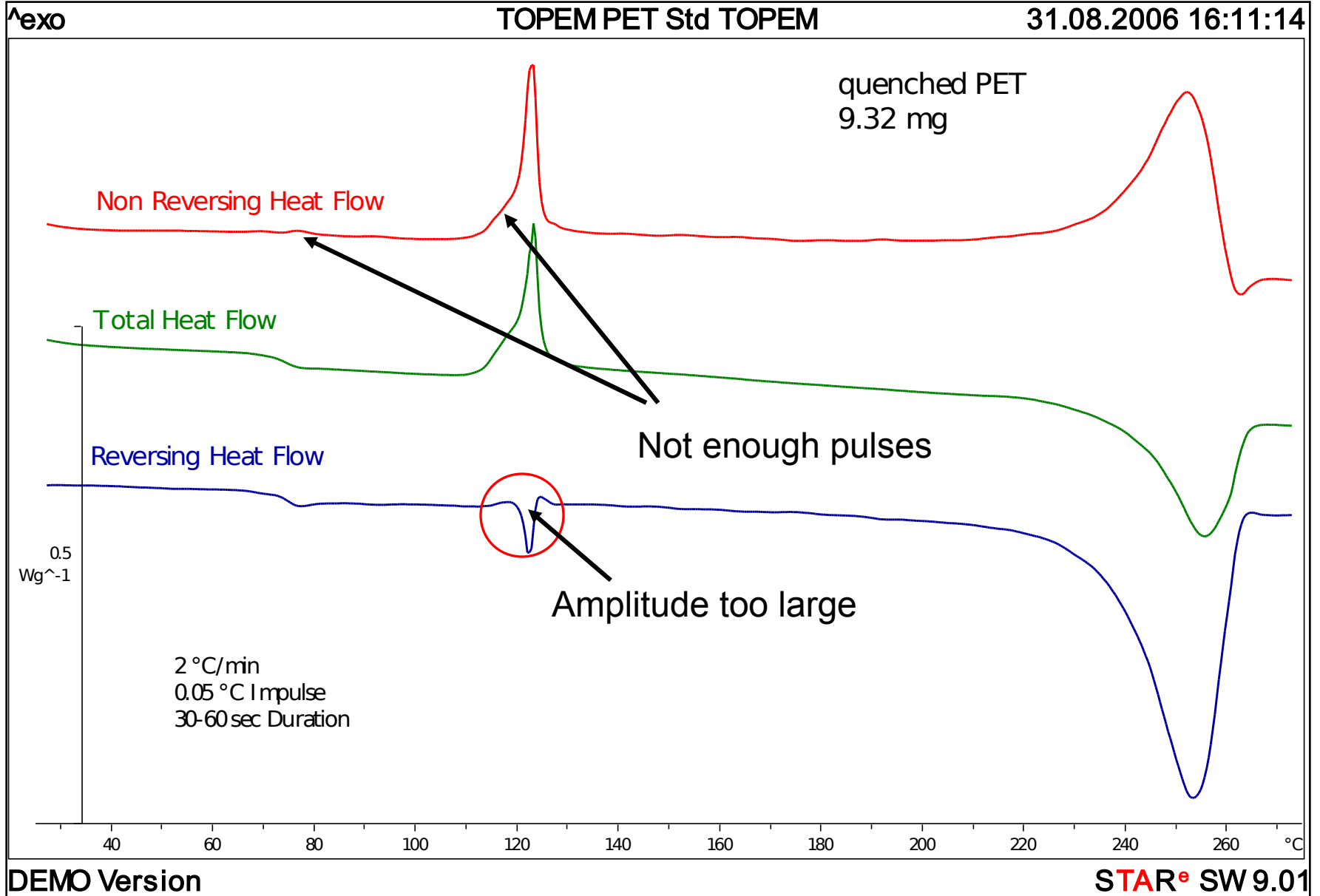
^exo



DEMO Version

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PET Basic TOPEM Results

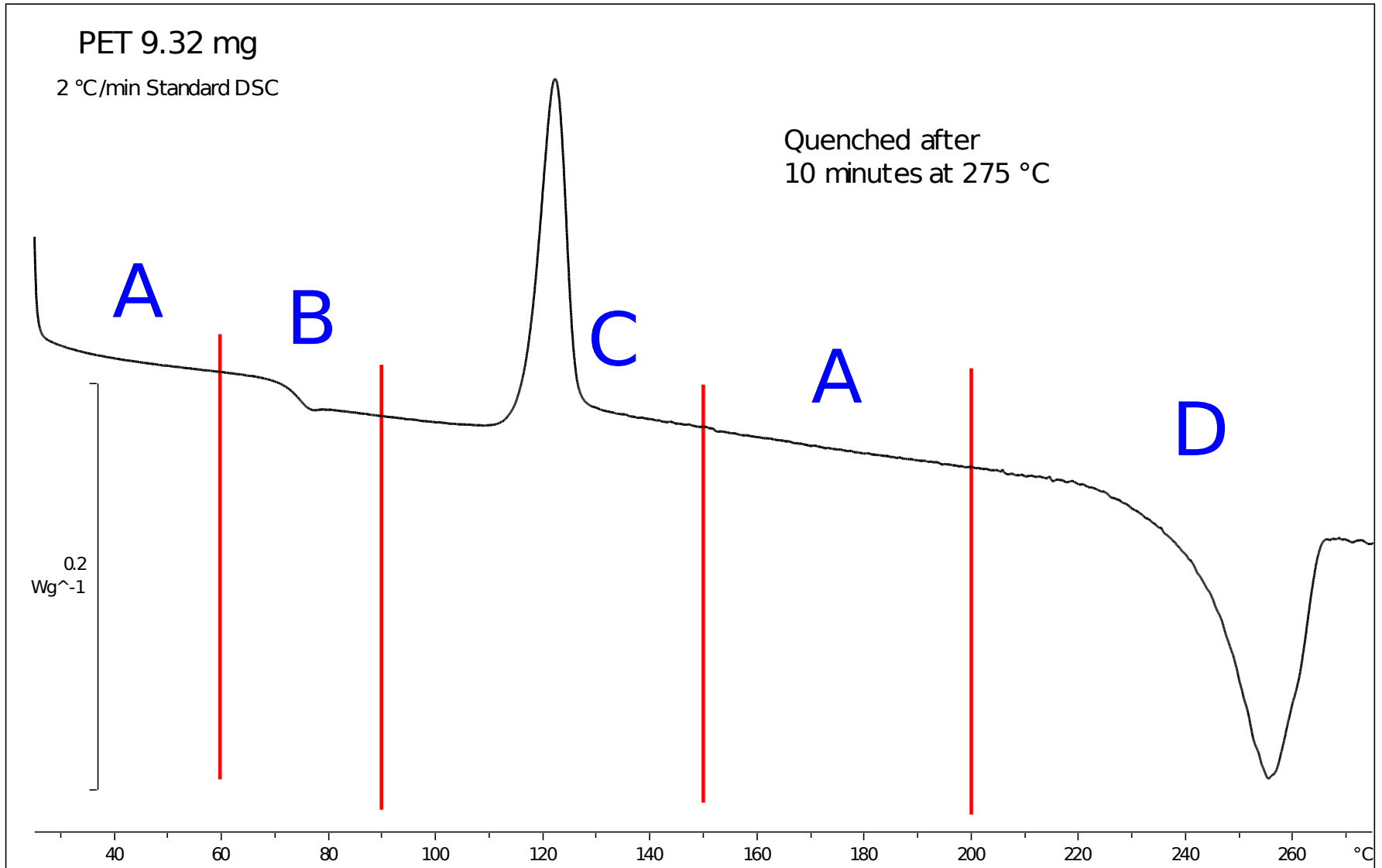


TOPEM of Amorphous PET

^exo

PET Standard DSC

25.08.2006 15:22:26

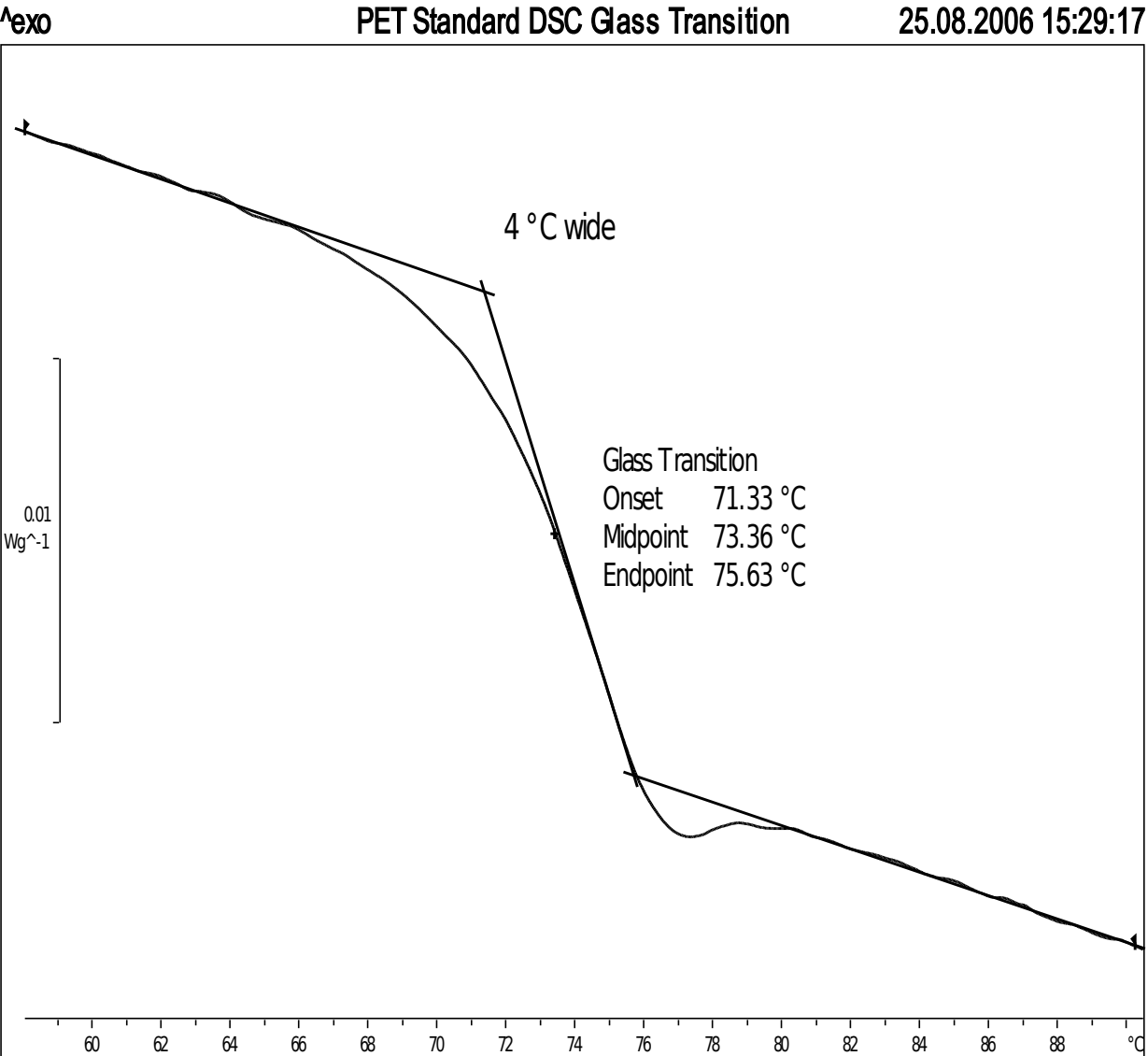


DEMO Version

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PET Glass Transition



2 °C Impulse Amplitude
Maximum Cp sensitivity
20-120 secs duration
Wide frequency range
2 mins/impulse
10 impulses/transition
Best resolution
20 mins/transition
4 °C / 20 mins = 0.2 °C/min

Optimal Settings

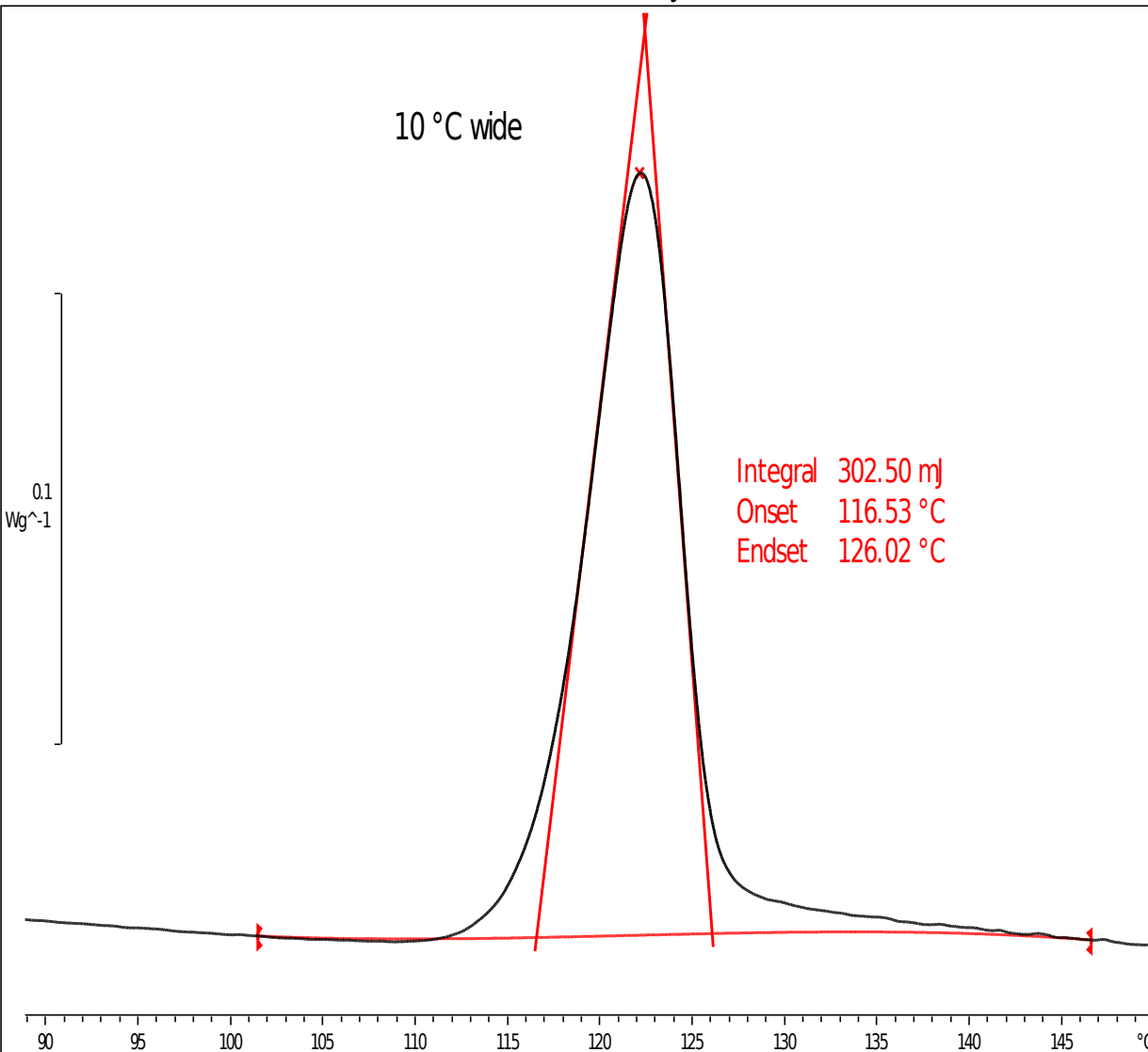
0.2 °C/min
2 °C impulse
20-120 secs duration

PET Cold Crystallization

^exo

PET Standard DSC Cold Crystallization

25.08.2006 15:35:07



0.05 °C Impulse Amplitude
Minimize Super Heating/Cooling

30-60 secs duration

Default setting

1 min/impulse

10 impulses/transition

Best resolution

10 mins/transition

10 °C / 10 mins = 1 °C/min

Optimal Settings

1 °C/min

0.05 °C impulse

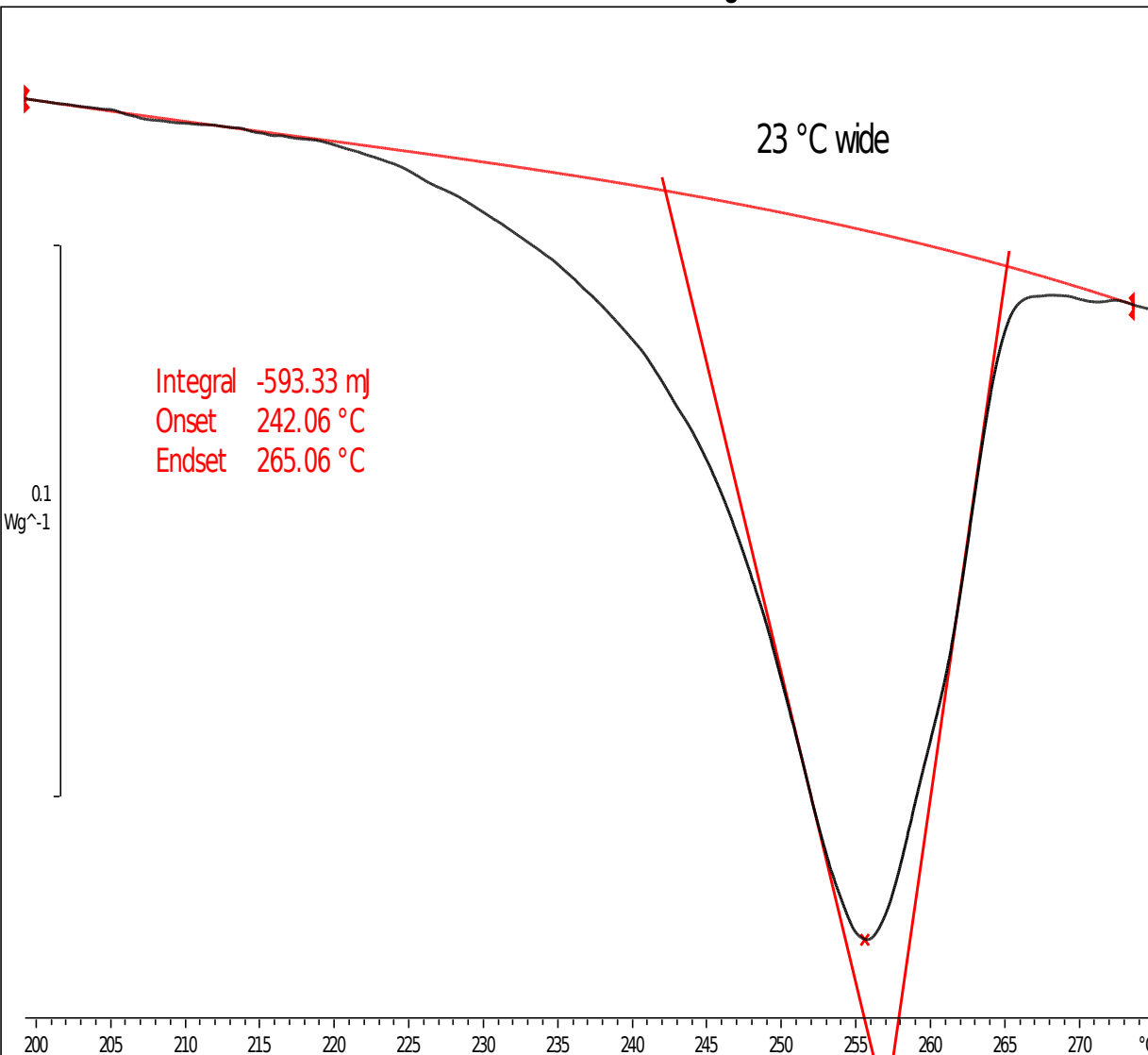
30-60 secs duration

PET Melting Transition

^exo

PET Standard DSC Melting

25.08.2006 15:40:23



0.05 °C Impulse Amplitude
Minimize Super Heating/Cooling
30-60 secs duration
Default setting
1 min/impulse
10 impulses/transition
Best resolution
10 mins/transition
23 °C / 10 mins = 2.3 °C/min

Optimal Settings

2 °C/min
0.05 °C impulse
30-60 secs duration

Creating a TOPEM Method

- Default method window
- Select TOPEM segment
- Select Dynamic segment
- Select impulse amplitude
- Select impulse duration
- Select crucible
- Save method

Adding 1st Segment

The screenshot displays the METTLER STARe Method software interface. The main window title is "(METTLER) - STARe Method". The menu bar includes File, Edit, View, Segment, Miscellaneous, and Settings. The toolbar contains various icons for file operations and analysis. The main area features a graph with temperature (°C) on the y-axis (0 to 200) and time (min) on the x-axis (0 to 90). A dialog box titled "Dynamic TOPEM Segment" is overlaid on the graph, containing the following fields and buttons:

Parameter	Value	Unit
Start	25	°C
End Temperature	35	°C
Heating Rate	1	°Cmin ⁻¹
Pulse Height	1	K

Buttons: OK, Cancel, Next, Help, More >>

At the bottom of the window, there is a status bar with the text "For Help, press F1", "Lab - 2004 to Date: METTLER", and "Samples: 0".

Adding 1st Segment

(METTLER) - STARe Method

File Edit View Segment Miscellaneous Settings ?

TA Technique DSC Sample Holder Aluminum Standard 40ul Segment Type TOPEM

Dynamic TOPEM Segment

Start 25 °C

End Temperature 60 °C

Heating Rate 2 °Cmin⁻¹

Pulse Height 1 K

Pulse Width

Min. 30 Max. 60 s

OK

Cancel

Next

Help

<< Less

Impulse Duration

°C

200

150

100

50

0

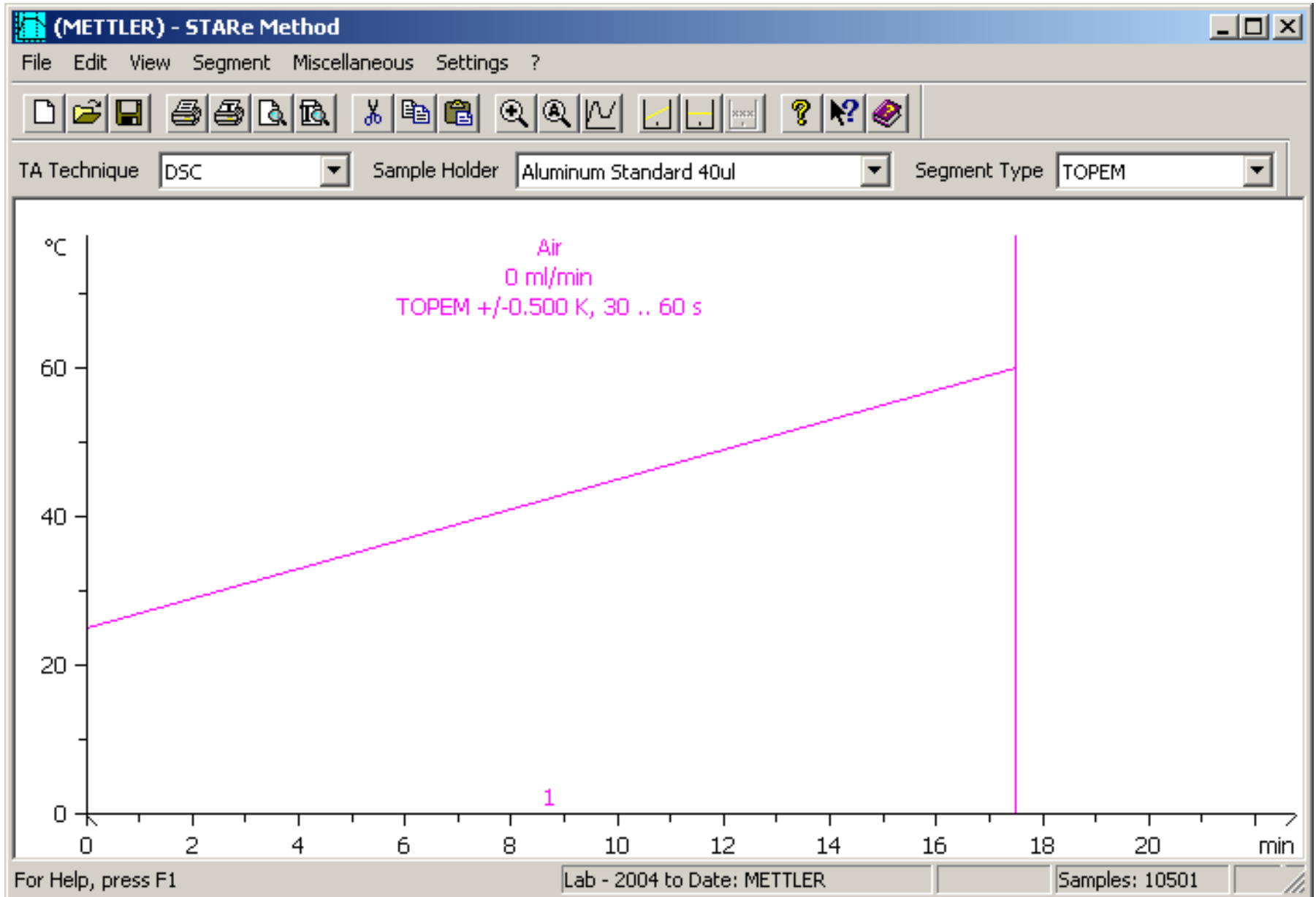
0 10 20 30 40 50 60 70 80 90 min

For Help, press F1

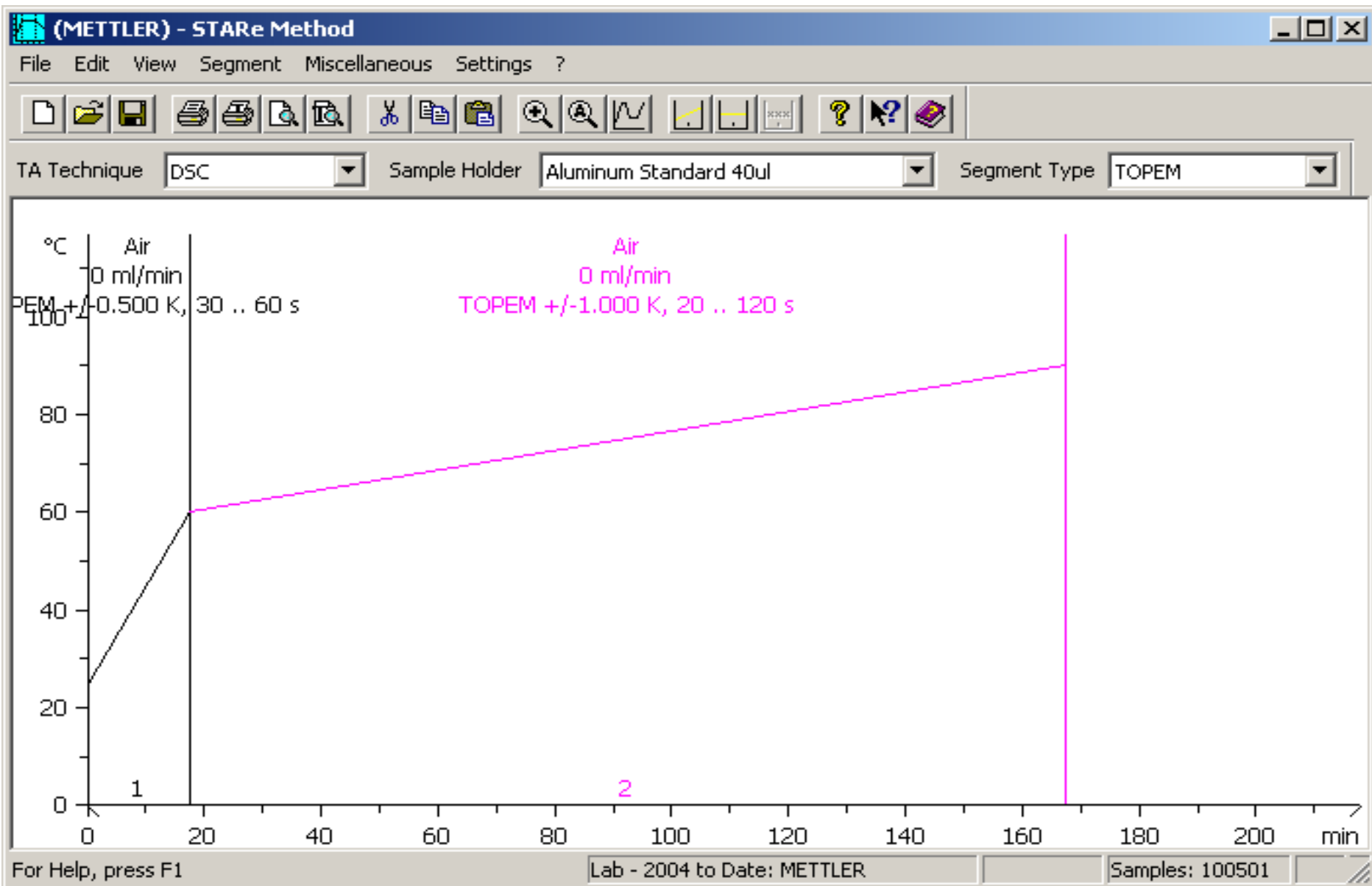
Lab - 2004 to Date: METTLER

Samples: 0

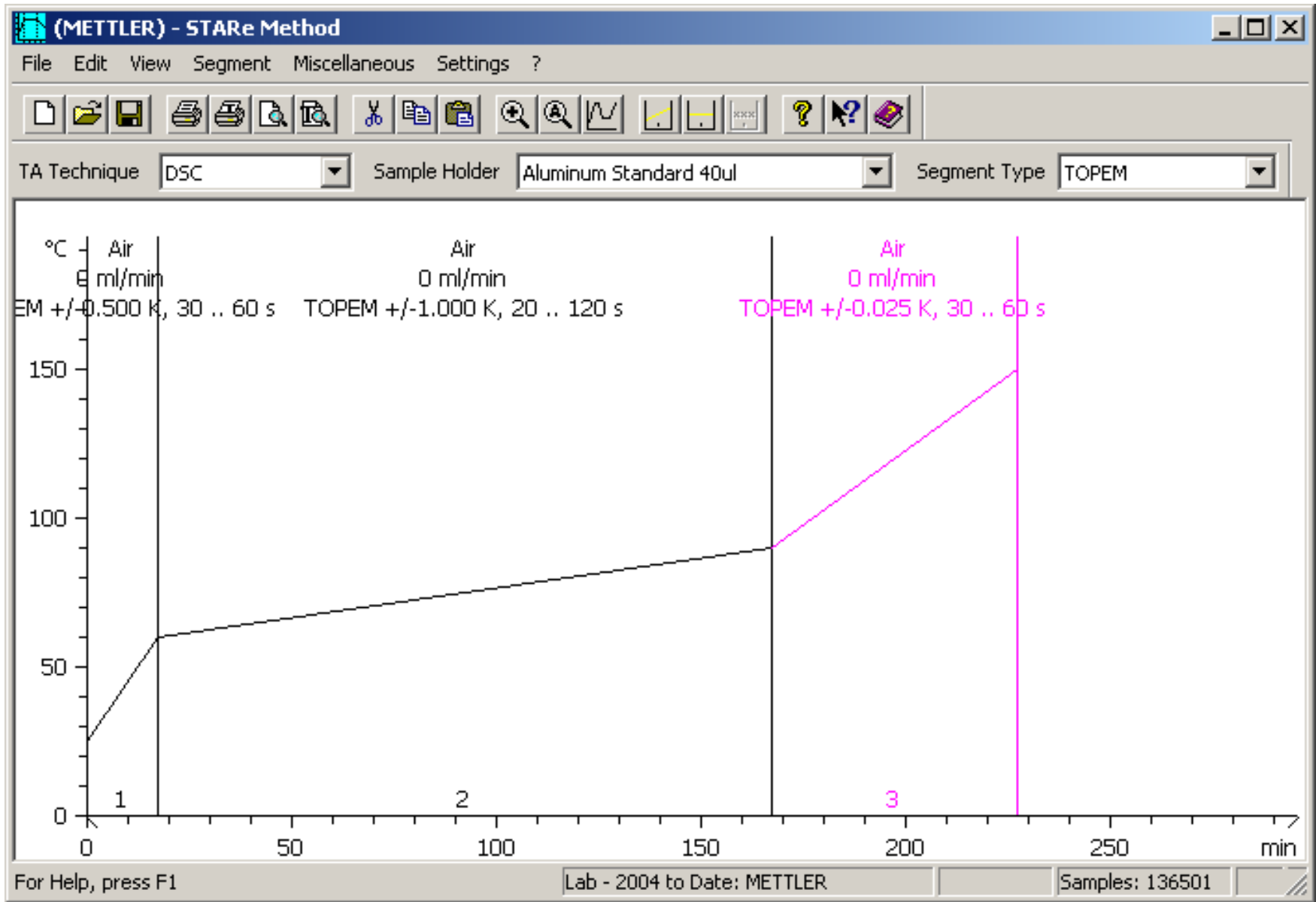
1st Segment Complete



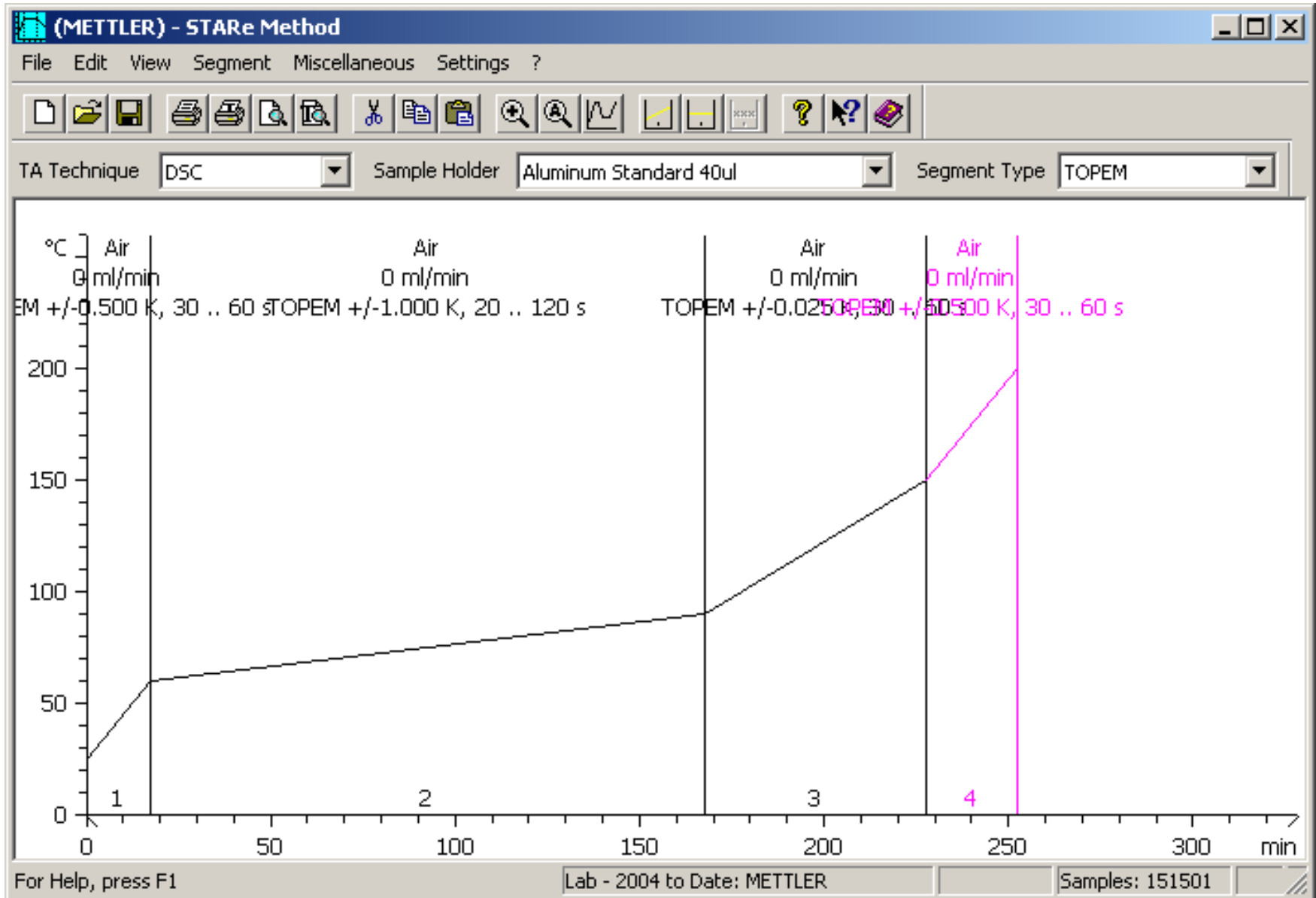
Add Second Segment



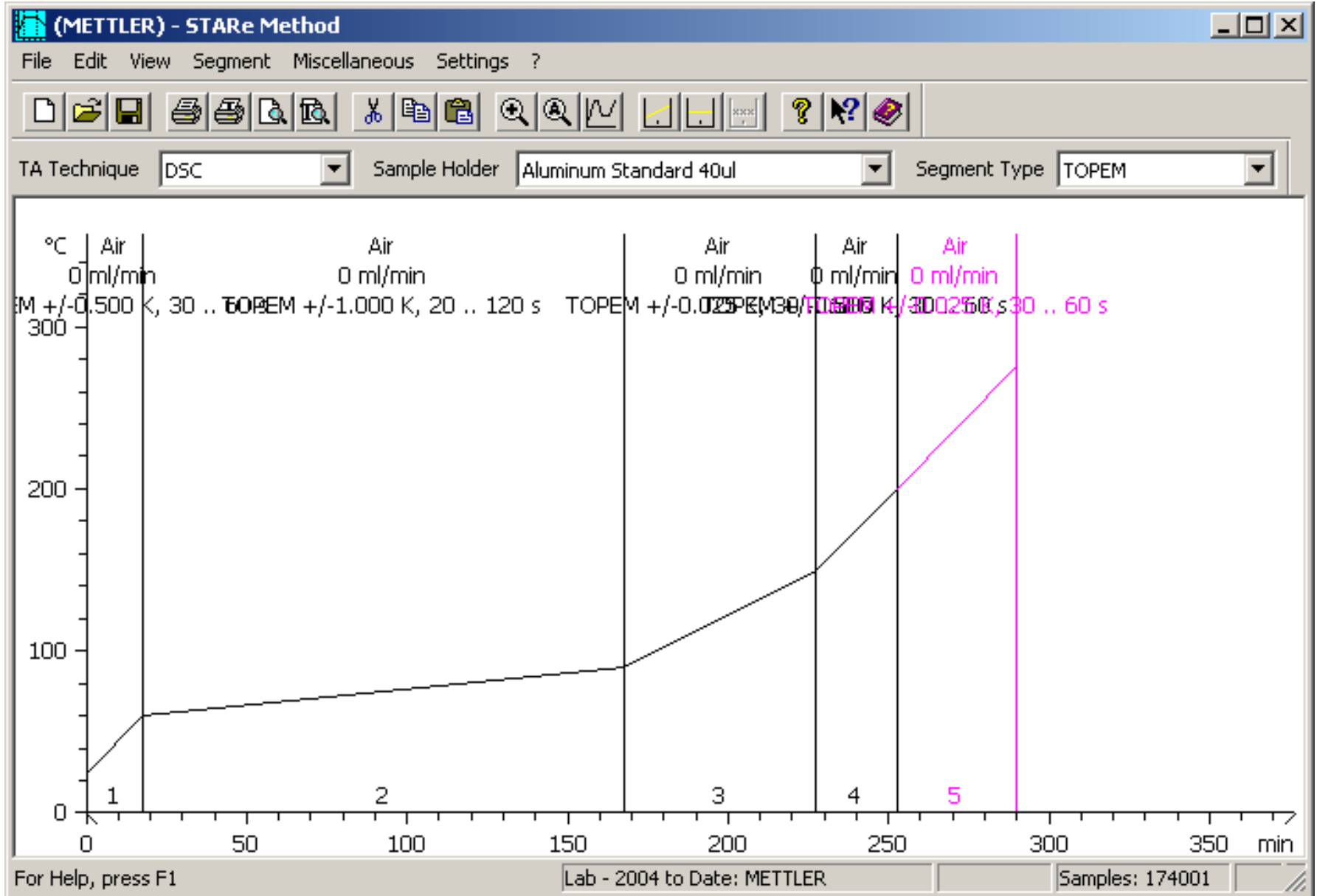
Third Segment



Fourth Segment



Fifth Segment, etc.



Enter the Experiment Info

PET TOPEM (METTLER) - STARE Experiment

File View Miscellaneous ?

Select Method TOPEM 2 Cpm 25-275C 30-60s 50mK 20uL

Run Blank Curve

Sapphire Reference Curve

Start 25 °C

Sample Holder ... Aluminum Light 20uL

Pan Weight 22.07 mg Reference Pan Weight 21.8500 mg

Sample Preparation

Gas List

Sample Name PET TOPEM DSC

Weight 9.32 mg (0.00 - 1000.00)

Position 101

Order Number

Customer ... Mettler

Remarks ... PET was quenched after holding at 275 °C

Module DSC823e/700/126/IC

Send Experiment

For help, press F1 Lab - 2004 to Date: METTLER NUM

Select Method

Sample Pan Weight

Reference Pan Weight

Sample Name

Sample Mass

Location on Carrousel

Select Customer

Add Remarks

Select DSC832s

Send Experiment

Experiments in Queue

DSC (METTLER) - DSC823e/700/126/IC: PET Quench

File Edit View Control Calib/Adjust Settings Service ?

Routine Editor
Experiments - performed
Experiment - on module
EXP PET Quench (METTLER)
Experiments - pending
EXP PET TOPEM Special test (METTLER)
EXP TOPEM Baseline (METTLER)
EXP PET Quench (METTLER)
EXP PET TOPEM (METTLER)
EXP TOPEM Baseline (METTLER)

Pos	User	Experiment	Method	Sample
101 (in progress)	METTLER	PET Quench	Iso 275 C for 10min	9.3200
101 (pending - n...)	METTLER	PET TOPEM Special test	TOPEM PET analysis	9.3200
102 (pending)	METTLER	TOPEM Baseline	TOPEM PET analysis	0.0000
101 (pending)	METTLER	PET Quench	Iso 275 C for 10min	9.3200
101 (pending)	METTLER	PET TOPEM	TOPEM 2 Cpm 25-275C 30-60s 50mK 20uL	9.3200
102 (pending)	METTLER	TOPEM Baseline	TOPEM 2 Cpm 25-275C 30-60s 50mK 20uL	0.0000

Cell Temperature (Tc): 275.0 °C
Measurement

Reset OK

For help, press F1

Lab - 2004 to Date: METTLER

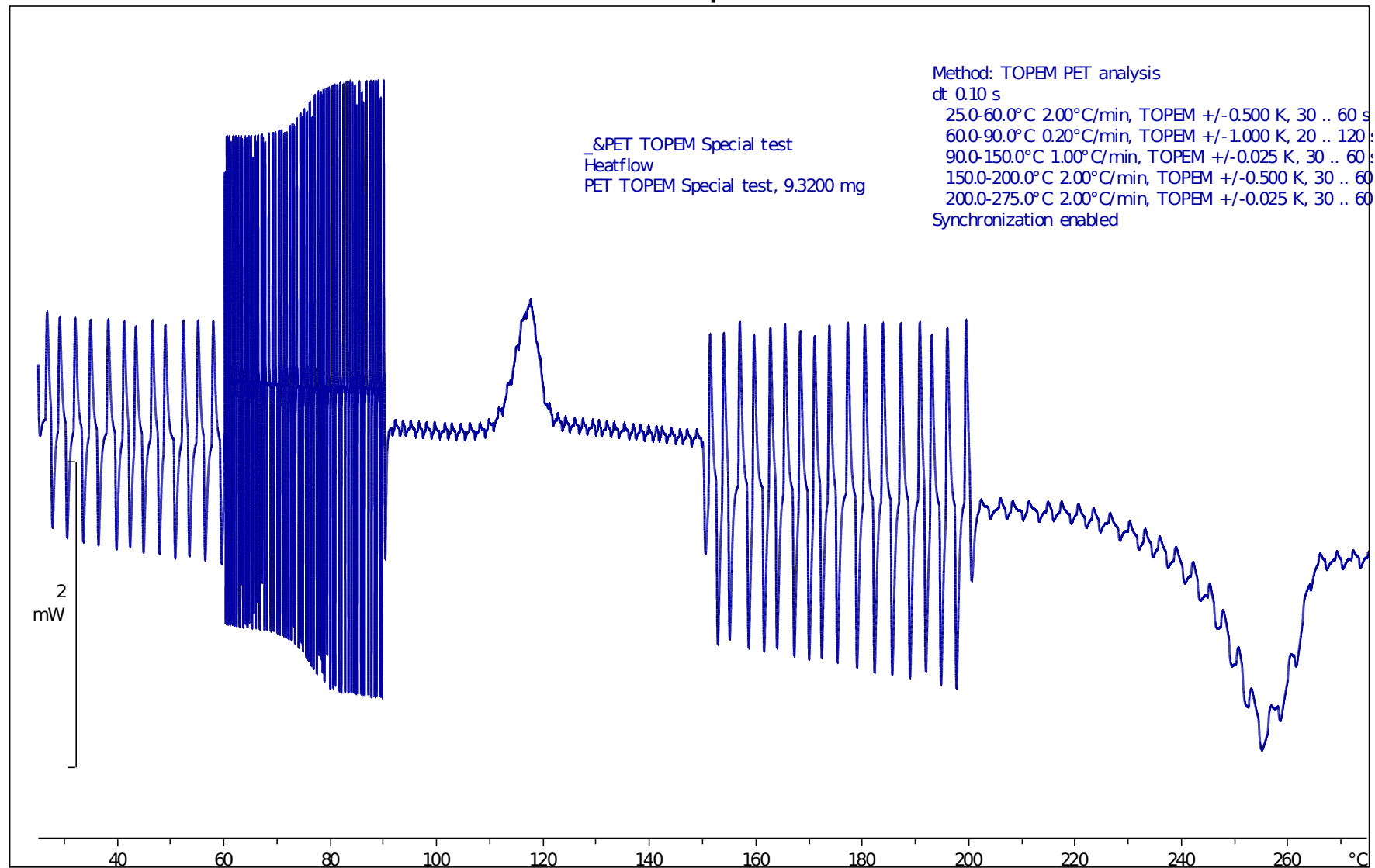
NUM

TOPEM PET after Raw Data

^exo

TOPEM PET Special Test

28.08.2006 08:20:15



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TOPEM Evaluation

10% of Calc Window

Time of two impulses

The image shows a software dialog box titled "TOPEM Evaluation". It is divided into two main sections: "Evaluation Parameters" and "Smoothing".

- Evaluation Parameters:**
 - "Width of Calculation Window:" with a text input field containing "120" and a unit "s". A red arrow points from the text "10% of Calc Window" to this field.
 - "Shift of Calculation Window:" with a text input field containing "12" and a unit "s". A red arrow points from the text "Time of two impulses" to this field.
 - An "Advanced..." button is located below these fields.
- Smoothing:**
 - "Width of Smoothing Window:" with a text input field containing "120" and a unit "s". A red arrow points from the text "Time of two impulses" to this field.

On the right side of the dialog box, there are four buttons: "OK", "Cancel", "Settings...", and "Help".

TOPEM Evaluation Settings

TOPEM Evaluation - Settings

Curve Selection

- cp0
- HFtot
- HFrev
- HFnon-rev

Existing Evaluation

- Overwrite

OK

Cancel

Help

Select the curves to plot

Advanced TOPEM Evaluation Parameters

Advanced TOPEM Evaluation Parameters

Sample Pan Weight: mg

Reference Pan Weight: mg

Sample Response Parameter:

Instrument Response Parameter:

HFnon rev

Linear

Quadratic

OK

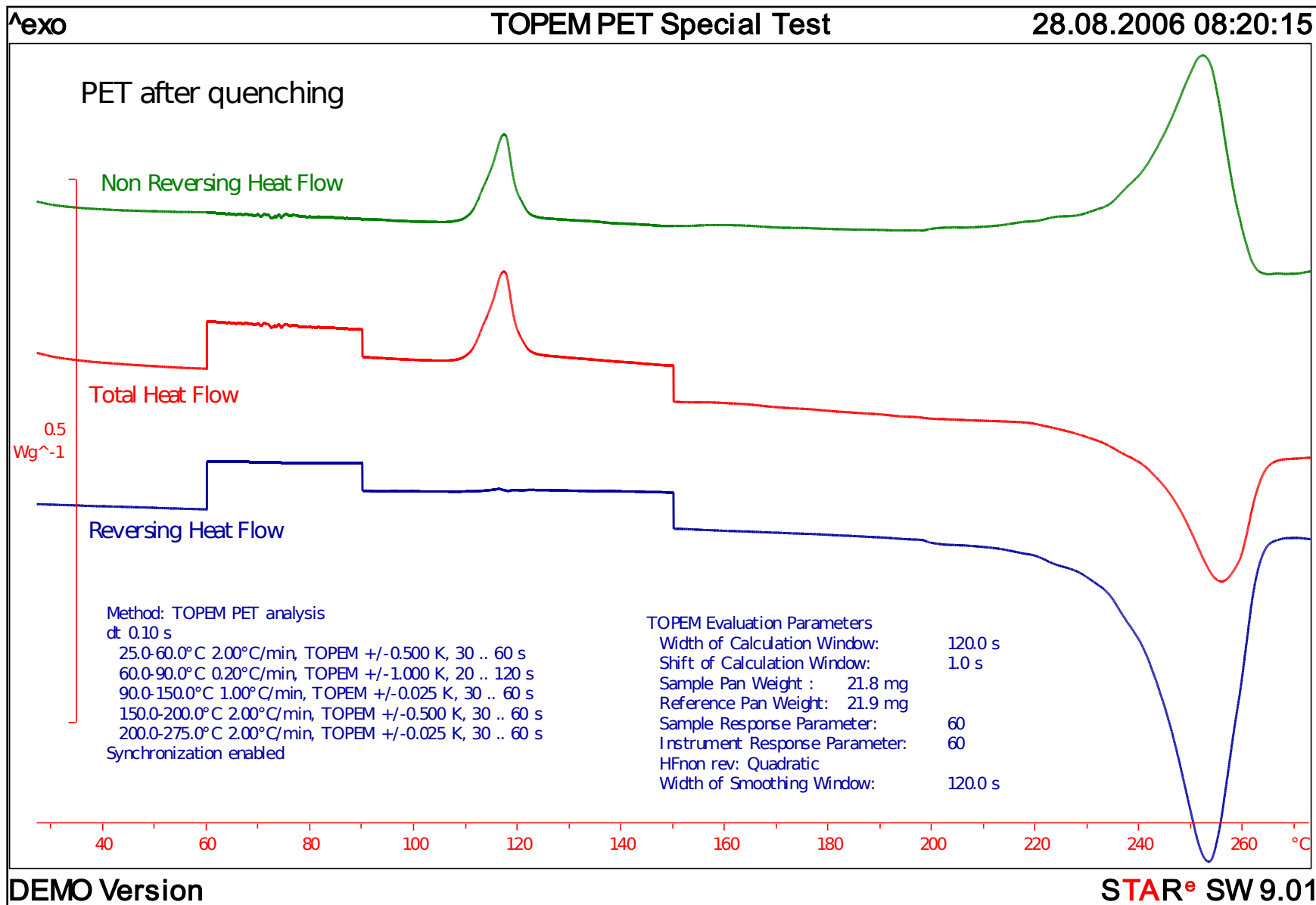
Cancel

Help

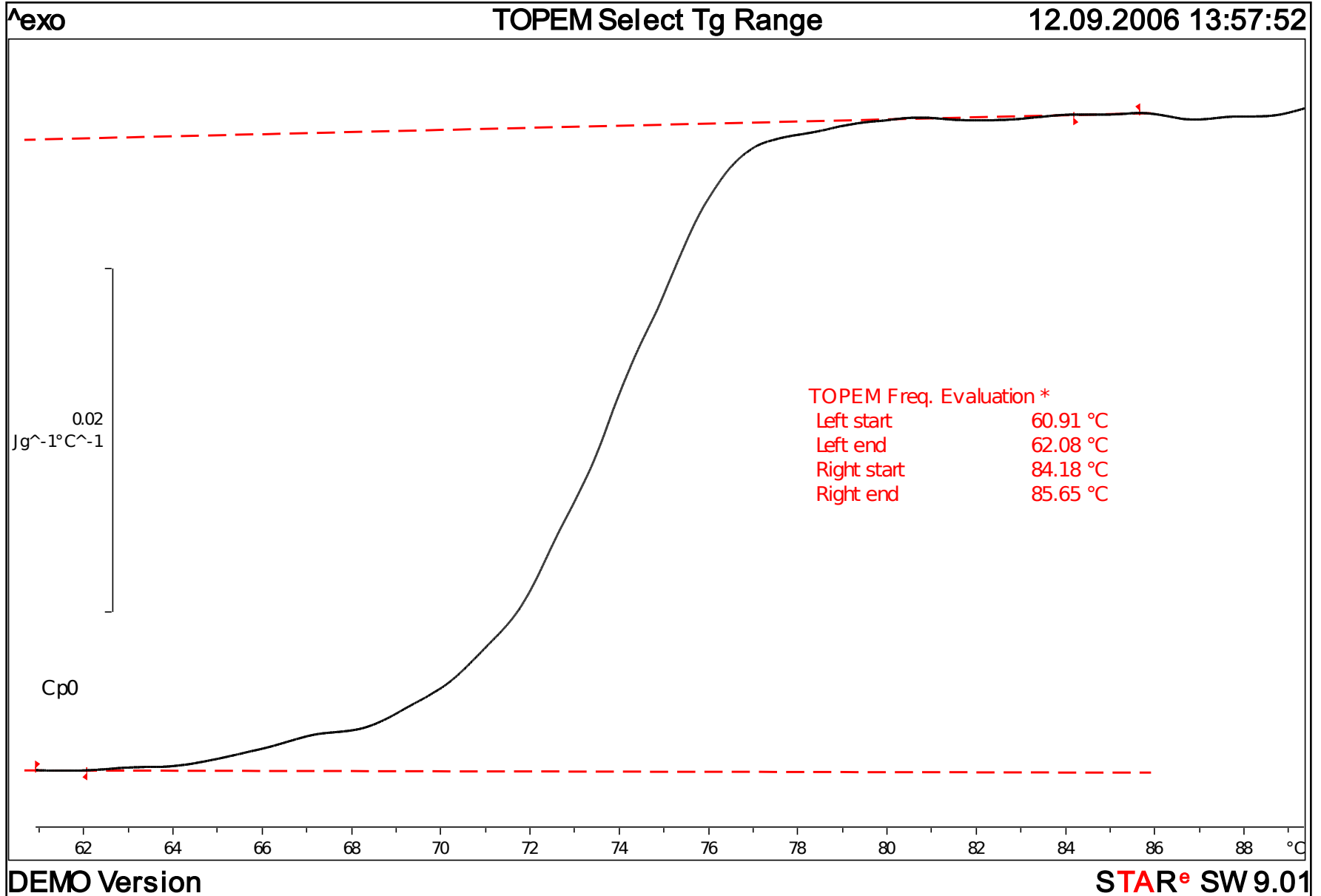
Corrects for Cp difference

Number of variables in equations

First plot of PET Special



Select the Glass Transition Region



Select Frequencies

TOPEM Frequency Evaluation

Frequencies: mHz

Type of Correction

None

Left

Right

Left and right

Smoothing

Width of Smoothing Window: s

OK

Cancel

Settings...

Help

Select Curves

TOPEM Frequency Evaluation - Settings

Curve Selection

cp'

cp''

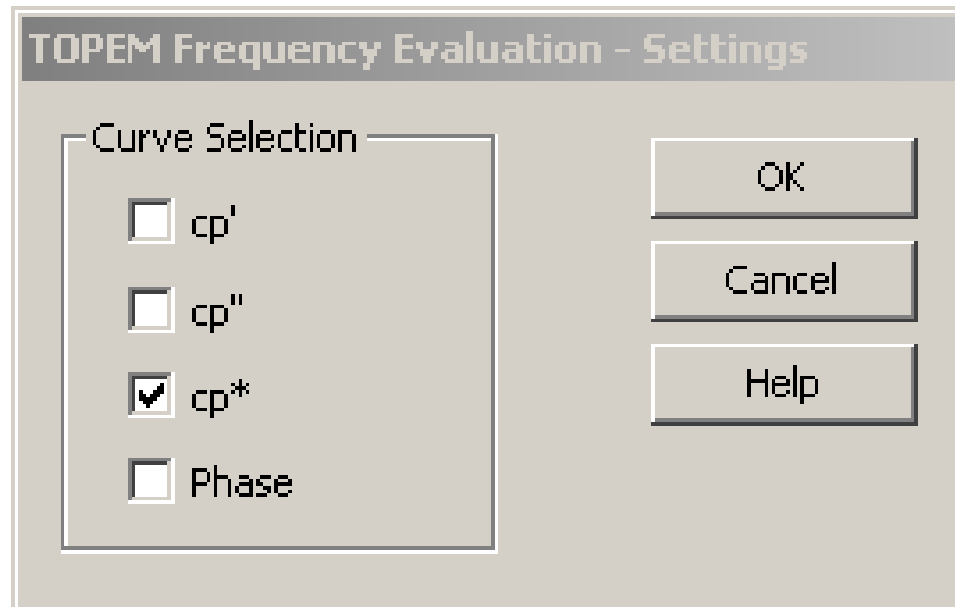
cp*

Phase

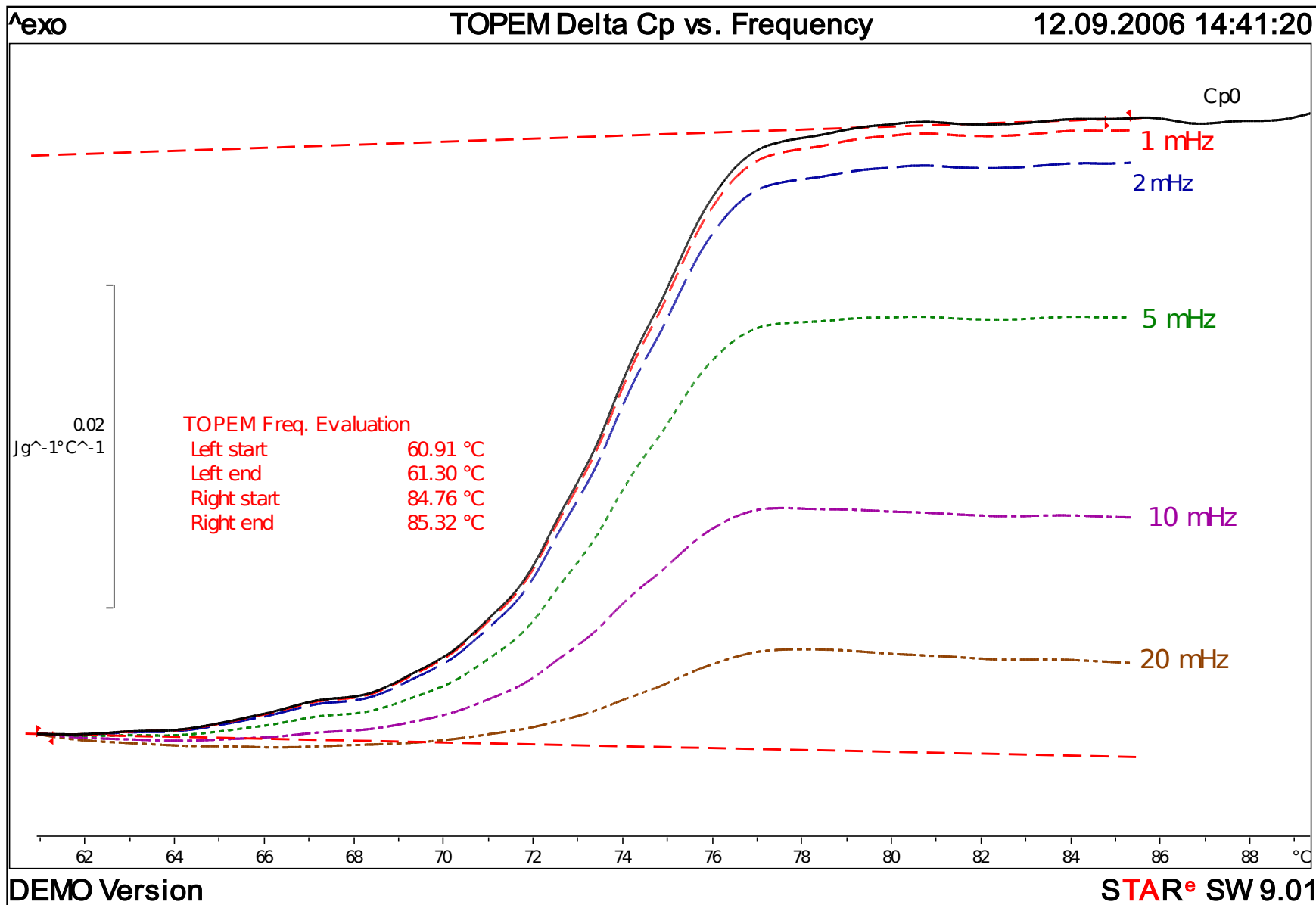
OK

Cancel

Help



Heat Capacity with Left Correction

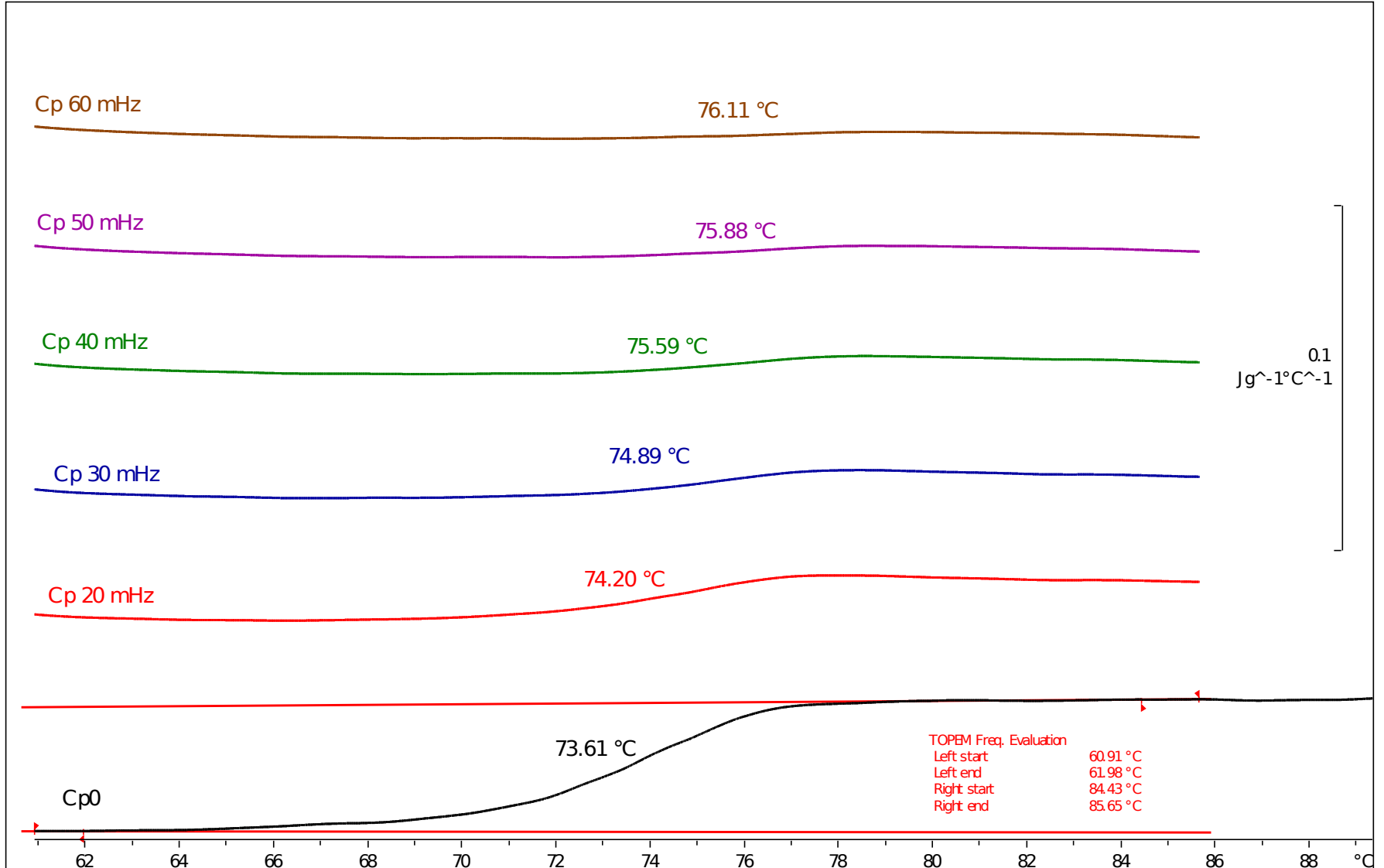


Glass Transition Shift with Frequency

^exo

TOPEM PET Tg Frequency Evaluation

06.09.2006 13:21:30

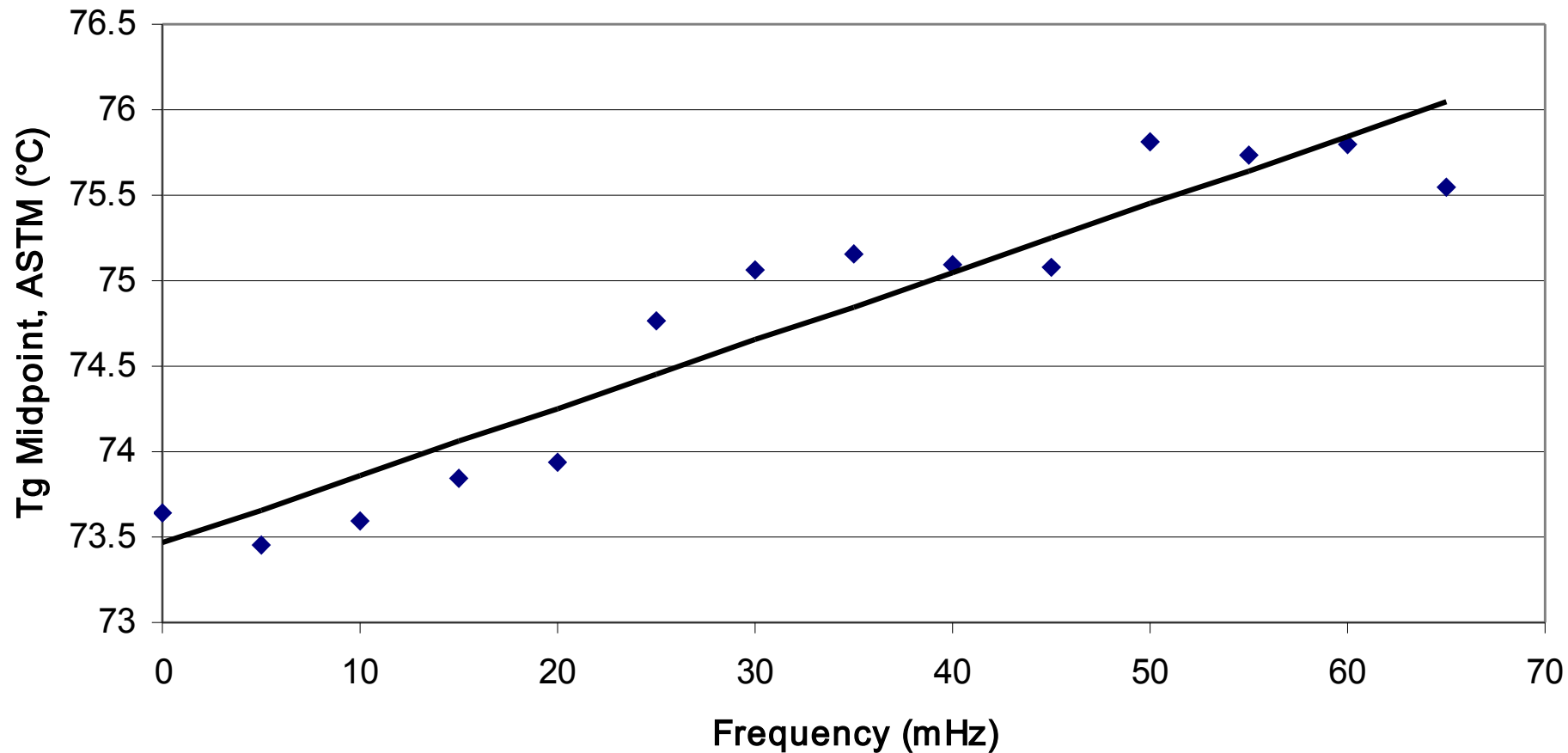


DEMO Version

STAR^e SW 9.01

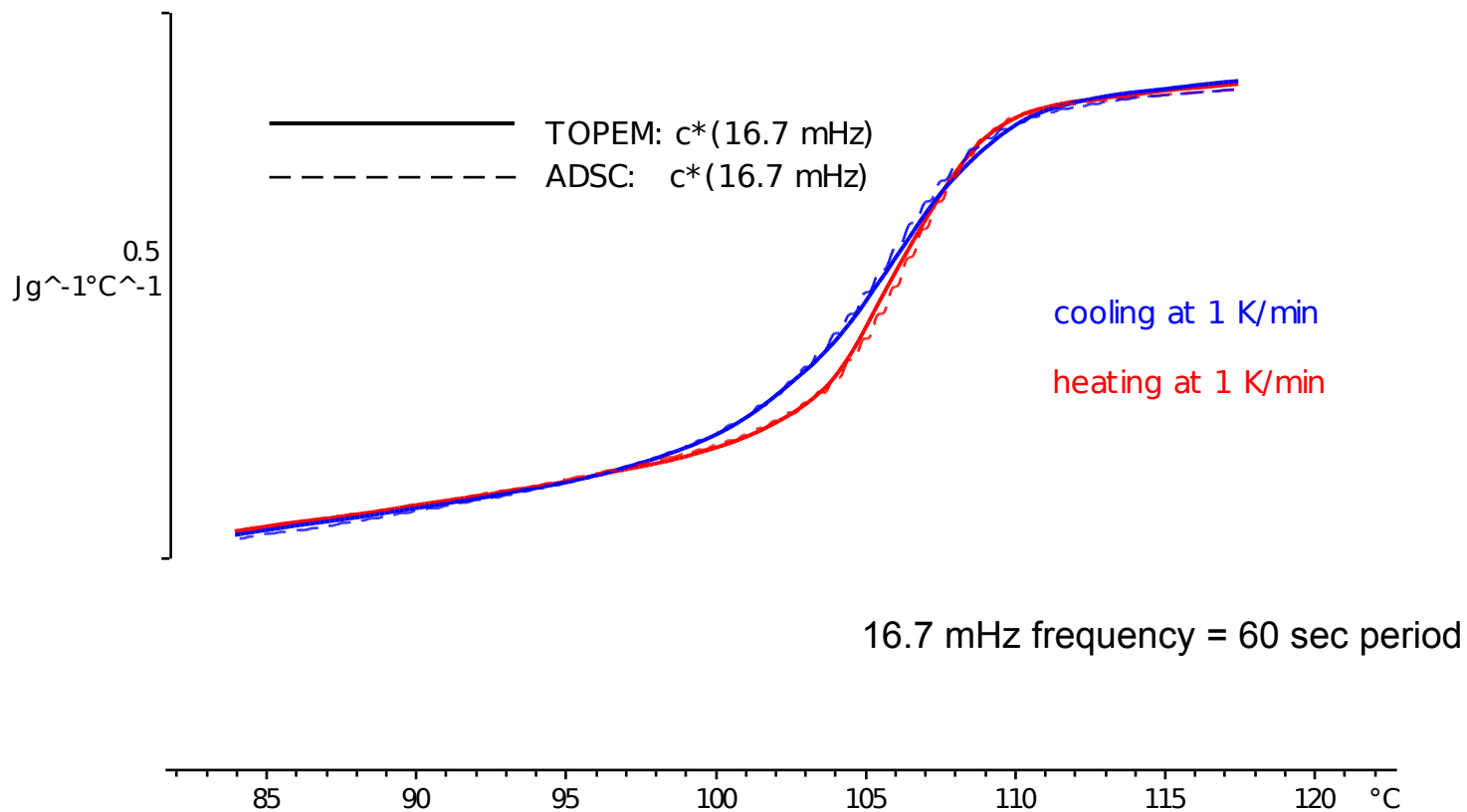
Glass Transition vs. Frequency

PET Tg versus Frequency



Comparison of TOPEM and ADSC

glass transition of PS



Summary

- TOPEM[®] DSC measures the reversing and non reversing heat flow from a sample at all frequencies by using a stochastic temperature impulse in combination with an isotherm or temperature ramp.
 - TOPEM DSC solves the heat flow equation for the samples response to temperature impulses.
 - The mathematical solution is applied to a temperature sine wave of a fixed frequency.
 - TOPEM calculates the heat capacity, reversing and non reversing heat flow for the sample.
- TOPEM DSC produces the same results as ADSC (Mettler) or MDSC[®] (TAI).
- It does not matter if the temperature sine wave is applied to the sample directly (ADSC & MDSC[®]) or to the mathematical model after the data is collected (TOPEM), the results are the same.
- TOPEM DSC and MDSC[®] (or ADSC) should all fall under the definition of Modulated Temperature DSC since they all give the same result.