

# Differential Scanning Calorimetry (DSC) **Practical** Training Course

2020

*Part 1*

***Calibration & Optimization & Trios***

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# Training Course Agenda (Part 1)

- Introduction
  - Technique
  - Tzero™ Technology vs. Heat flux DSC
- Experimental Method
- Calibration & Verification
  - Tzero or Baseline (Heat Flux DSC)
  - Heat Flow
  - Temperature
- Optimisation of Experimental Conditions
  - Sample Preparation & Sample Cups
  - Heating/Cooling Rate
- Trios Software: Tips & Tricks

# Introduction

## Technique & Instrumentation



# DSC: The Technique

- Differential Scanning Calorimetry (DSC) **measures** the **temperatures** and **heat flows** associated with transitions in materials as a function of time and temperature in a controlled atmosphere.
- These measurements provide **quantitative and qualitative** information about physical and chemical changes that involve ***endothermic*** or ***exothermic processes***, or ***changes in heat capacity***.



# Definitions

- **Heat Capacity:** amount of heat required to heat up a sample by  $1^{\circ}\text{C}$
- **Endothermic:** heat flow into the sample as a result of either heat capacity (while heating) and/or some endothermic process (melting, evaporation, etc.)
- **Exothermic:** heat flow out of the sample as a result of either heat capacity (during cooling) or some exothermic process (crystallization, thermoset cure, oxidation, etc.)

# Heat Flow in DSC

$$dQ/dt = C_p \cdot dT/dt + f(t, T)$$

where:

$dQ/dt$  = heat flow (W/g or mW)

$W = J/sec$

$C_p$  = specific heat capacity (J/g.°C)

$$C_p(T) = a + b.T + c.T^2$$

$dT/dt$  = heating rate (°C/min)

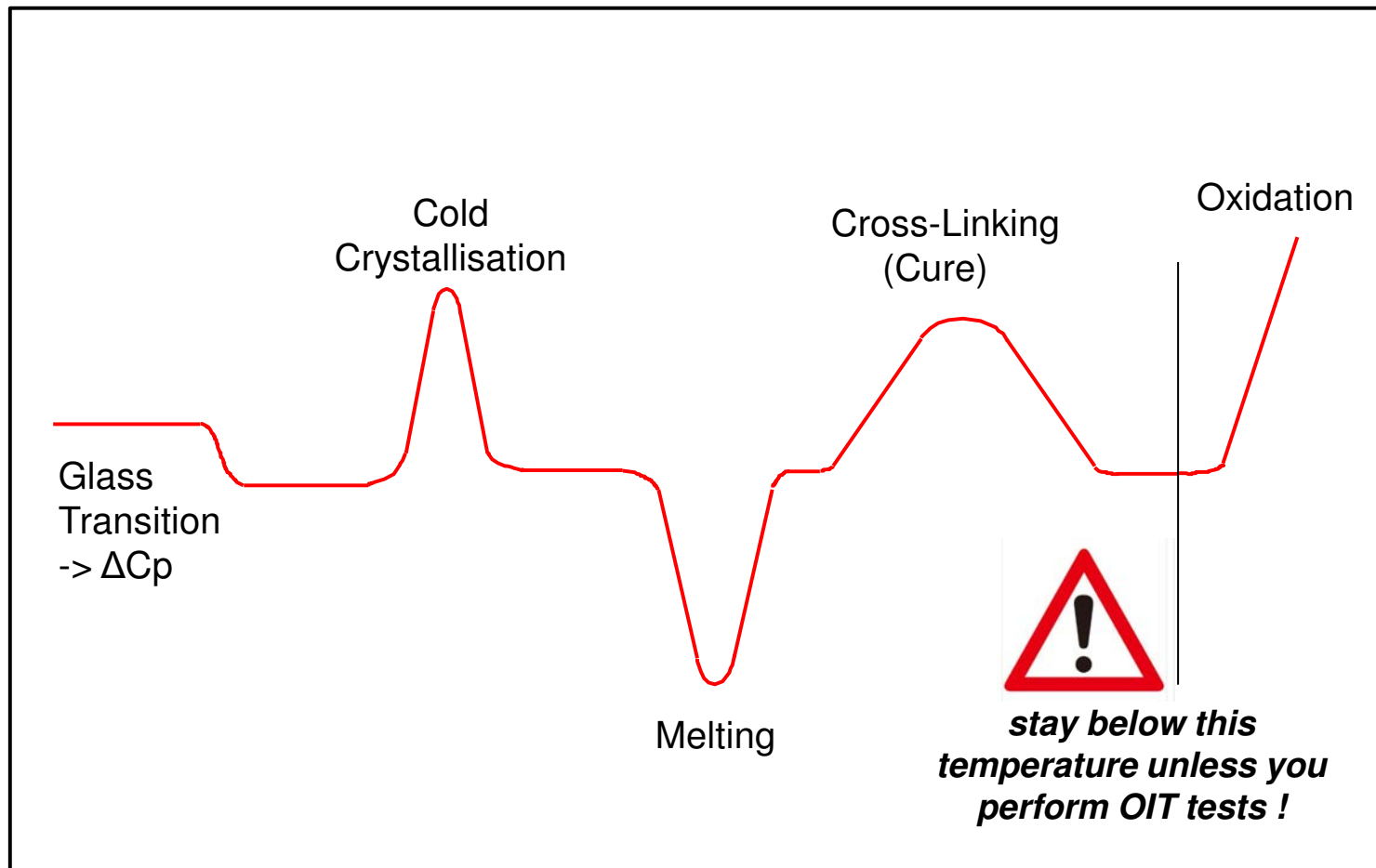
$f(t, T)$  = time dependent

(kinetic) components

e.g. melting, crystallisation, curing,...

# DSC Thermogram

endothemic <- Heat Flow -> exothermic



Temperature

# How is Heat Flow Measured ?

DSC25xx with or without AS

Heat Flux DSC  
DSC 25

Basic **Tzero™** DSC  
DSC 250

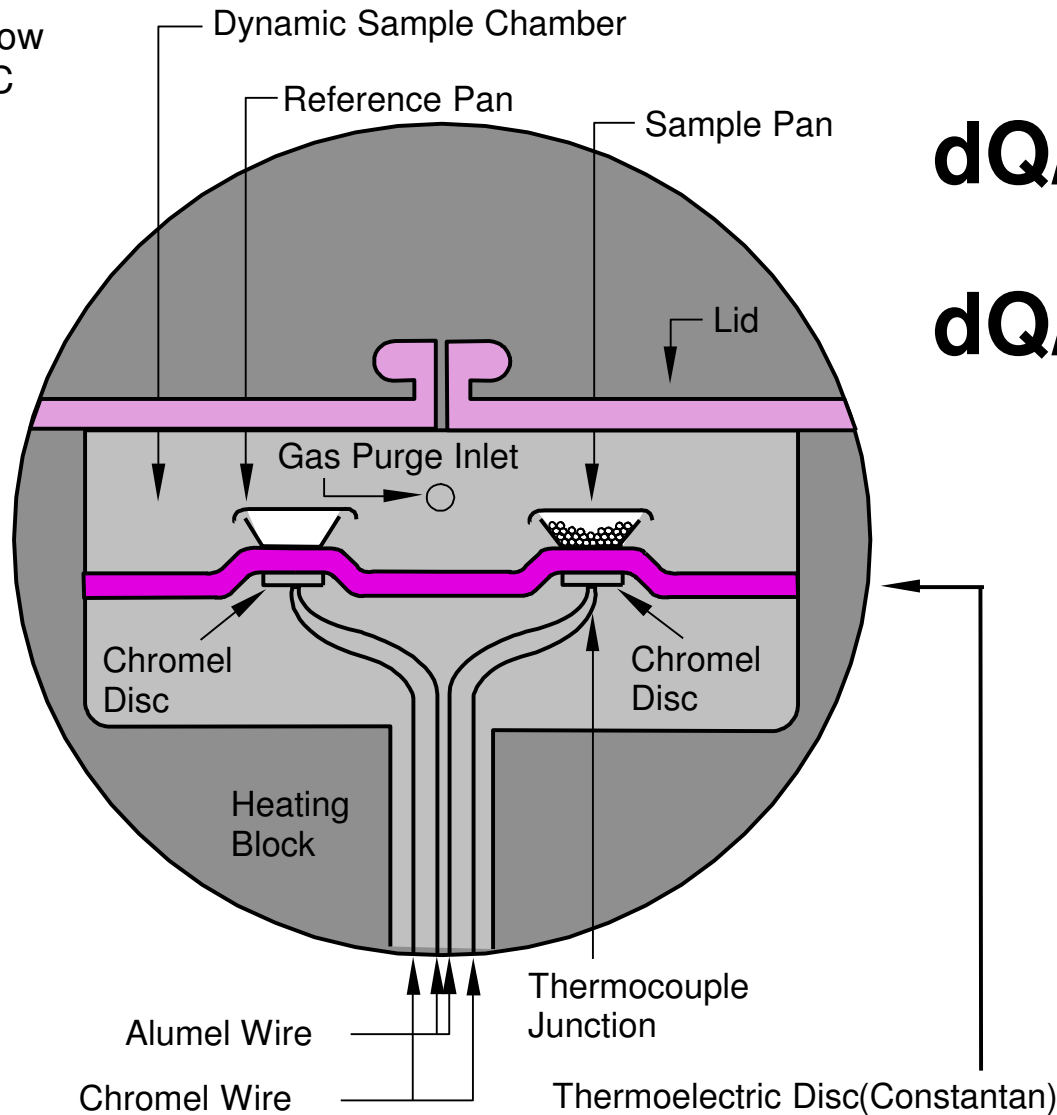
Advanced **Tzero™**  
DSC 2500



First generation  
Discovery DSC  
Advanced **Tzero™**

# Heat Flux DSC Instrument DSC 25

Note: schematics show a 2920 heat flux DSC



$$dQ/dt = Q_s - Q_r$$

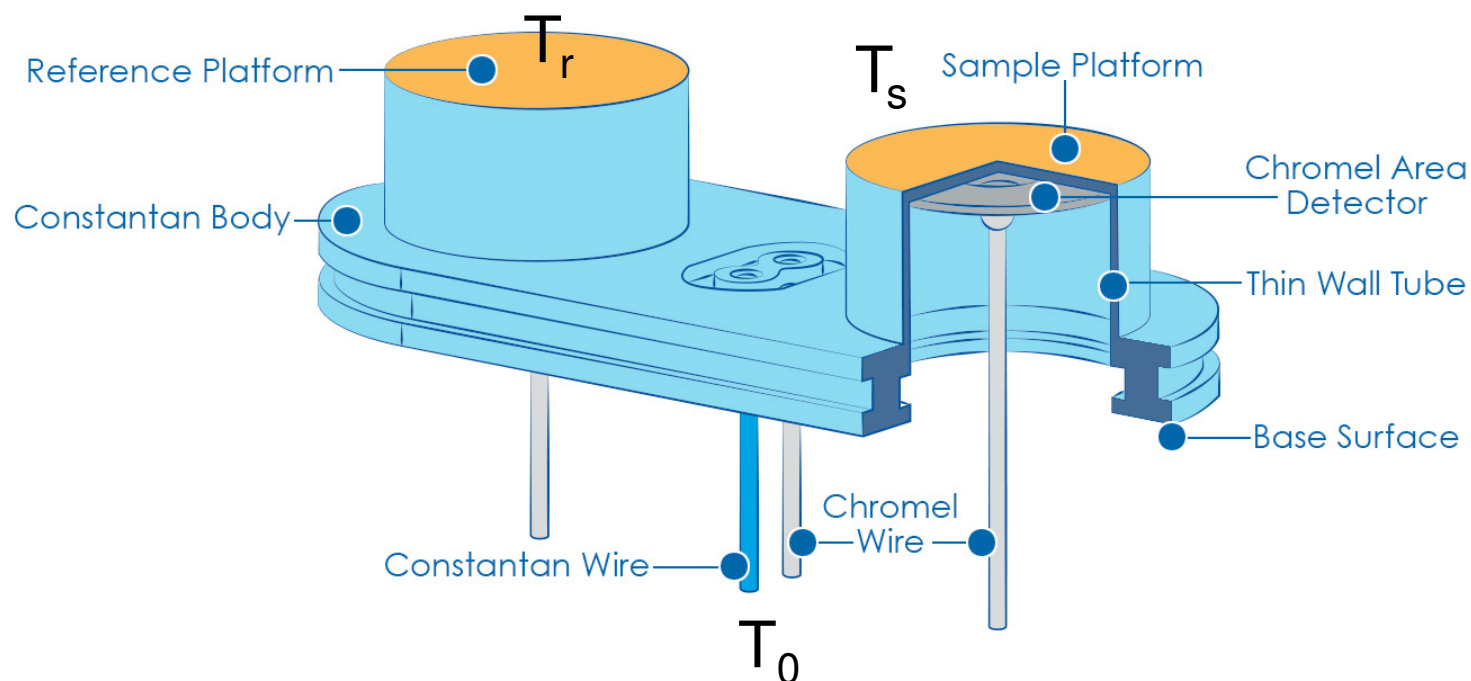
$$dQ/dt = \Delta T / R$$

$$\Delta T = T_s - T_r$$

Assumes symmetry between sample and reference calorimeter

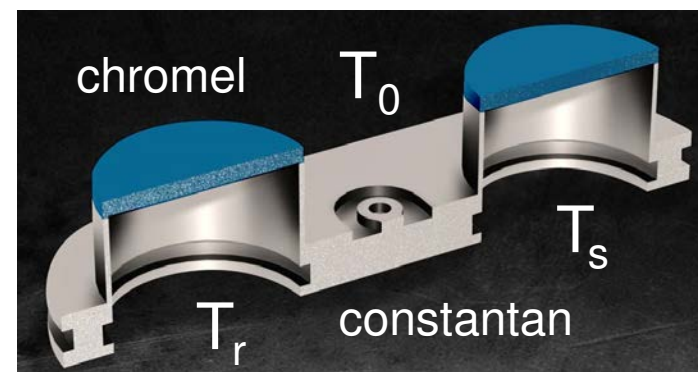
**T1**

# Tzero™ Sensor



**DSC 2500 – DSC 250 – (DSC 25)**

**First generation Discovery:  
diffusion bonded sensors**



# Tzero™ Heat Flow Equation T4

$$q = q_s - q_r \quad \Delta T = T_s - T_r \quad \Delta T_0 = T_0 - T_s$$

$$q = -\frac{\Delta T}{R_r} + \Delta T_0 \left( \frac{1}{R_s} - \frac{1}{R_r} \right) + (C_r - C_s) \frac{dT_s}{dt} - C_r \frac{d\Delta T}{dt}$$

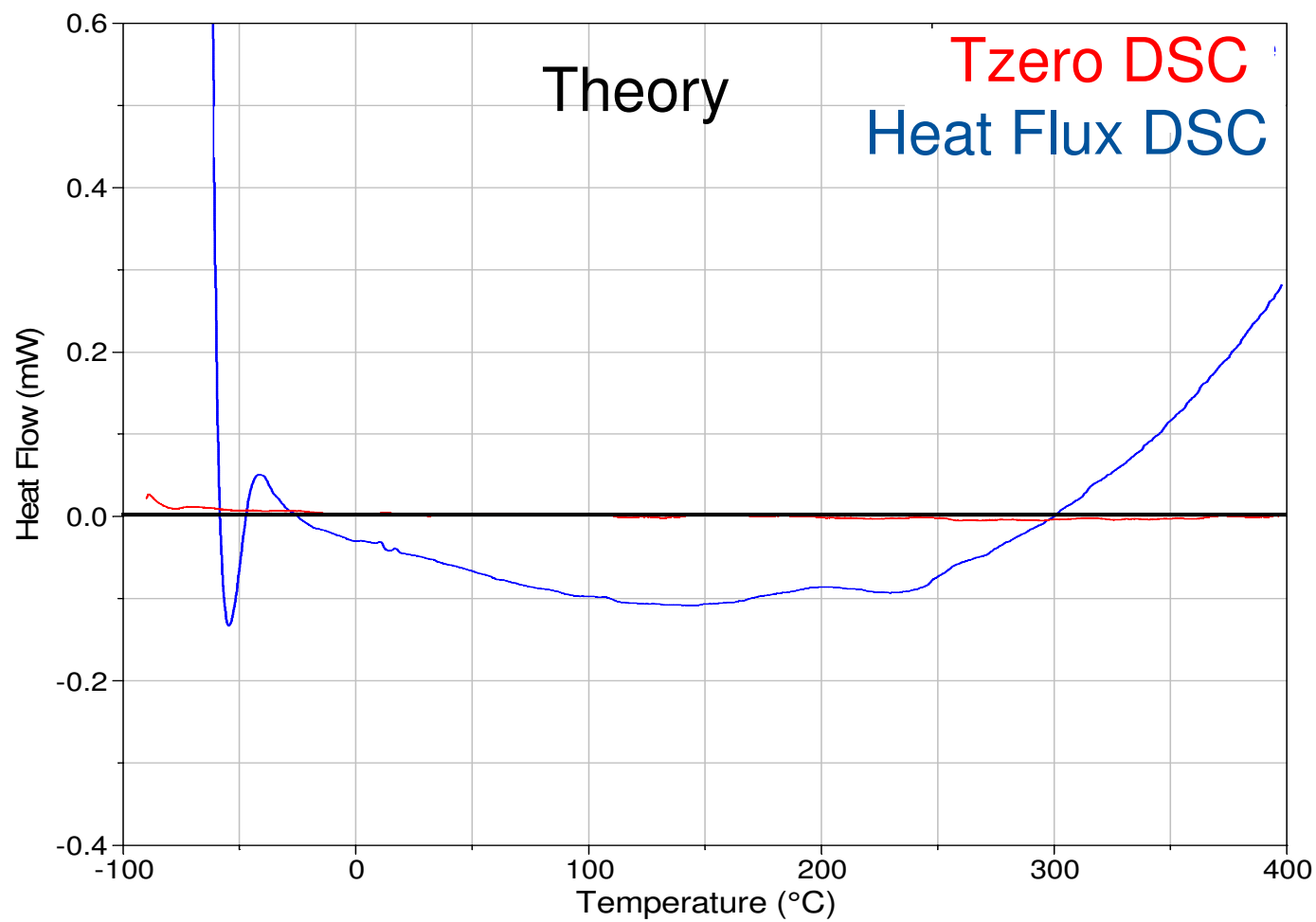
Principal DSC Heat Flow

Thermal Resistance Imbalance

Heat Capacity Imbalance

Heating Rate Difference

# Baseline (= Empty Cell Run)

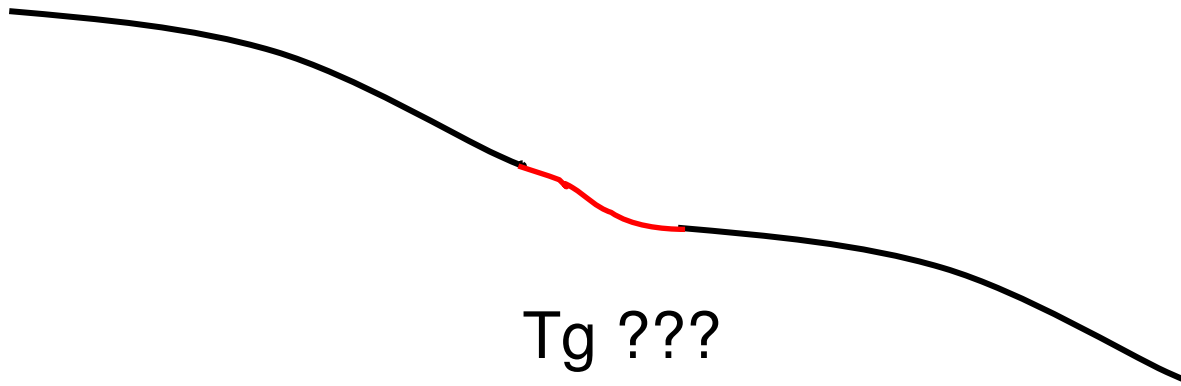




# Baseline Curvature Hinders Detection of Weak Transitions



Weak, broad Tg



Tg ???

# Tzero™ Heat Flow Terms T4

$$-\frac{\Delta T}{R_r}$$

## Principal DSC Heat Flow

provides main heat flow signal  
this is the only term used in heat flux DSC

$$\Delta T_0 \left( \frac{1}{R_s} - \frac{1}{R_r} \right)$$

## Thermal Resistance Imbalance

improve baseline

$$(C_r - C_s) \frac{dT_s}{dt}$$

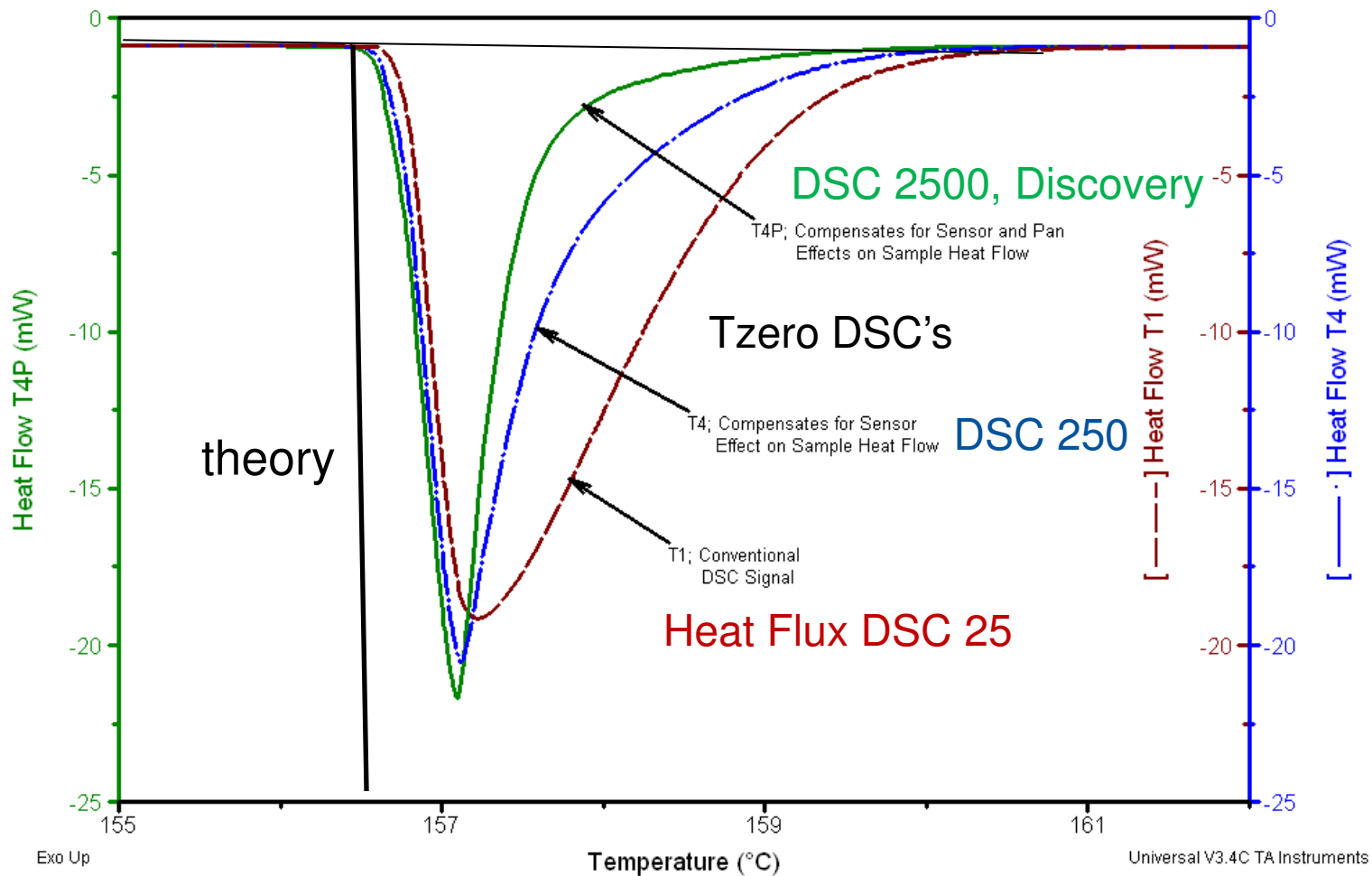
## Heat Capacity Imbalance

$$-C_r \frac{d\Delta T}{dt}$$

## Heating Rate Difference

improves resolution

# Resolution : Indium Melting Run

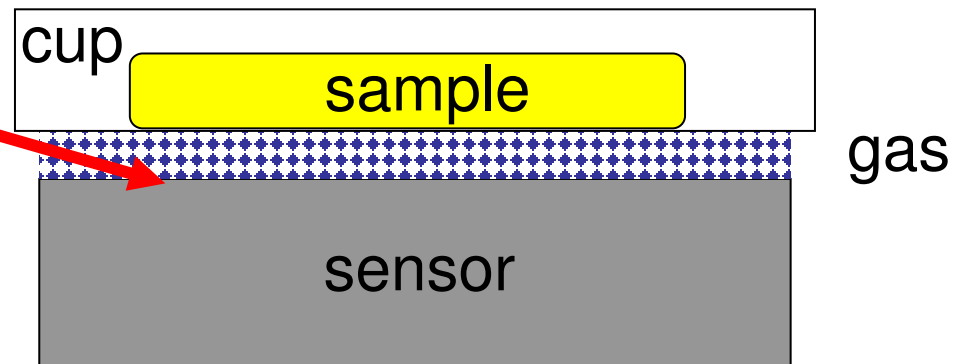


# Temperature / Heating Rate

What temperature is being measured and displayed by the DSC ?

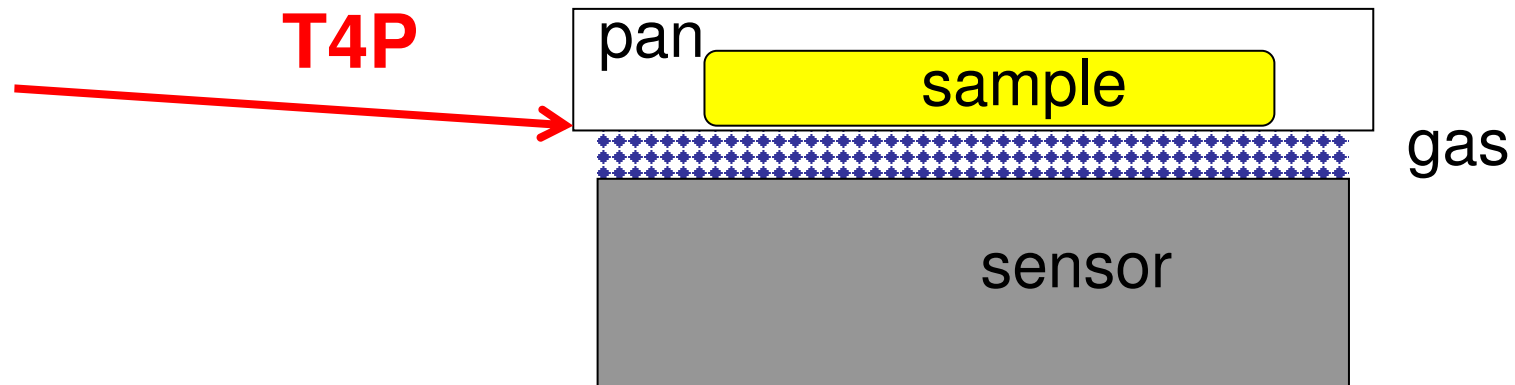
- The actual **temperature** of the **sample** is never measured by DSC
- **Sensor Temp**: used by most DSC's. It is measured at the sample platform with an area thermocouple

DSC 25 / DSC 250



# Temperature / Heating Rate

- **Pan Temp**: calculated by DSC 2500 and Discovery based on pan material and shape
- Uses **weight** of pan, resistance of pan (**type** of pan), and thermoconductivity of **purge gas**
- **Resolution** even more improved



# Experimental Method



# Heating/Cooling Method

- Heating Method

(NOTE: No equilibrate segment necessary if starting at or near ambient temperature)

1) Ramp 10°C/min to 300°C

(NOTE: No equilibrate at RT necessary for cooling down, defined in post test options)

- Cooling Method

1) Equilibrate at 300°C

2) Ramp 10°C/min to 25°C

Advanced

Load Window

Use Standby Temperature

Use 25.00 °C to 26.00 °C

End of Test

Discard pan in waste bin at end of test

Use Standby Temperature

General

Temperature 39.84 °C

Flange Temperature -13.99 °C

Cooler Selection RCS 90

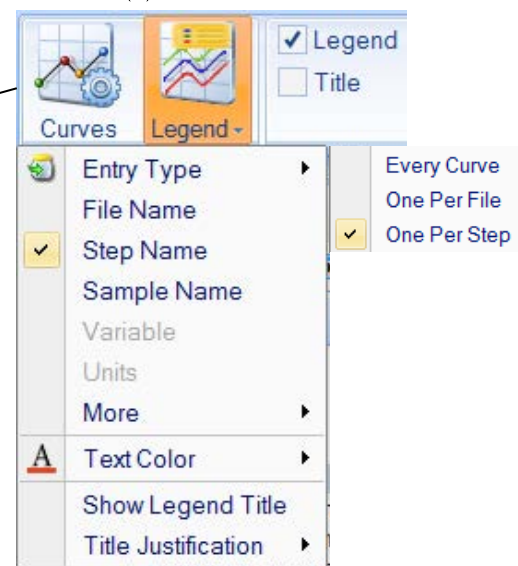
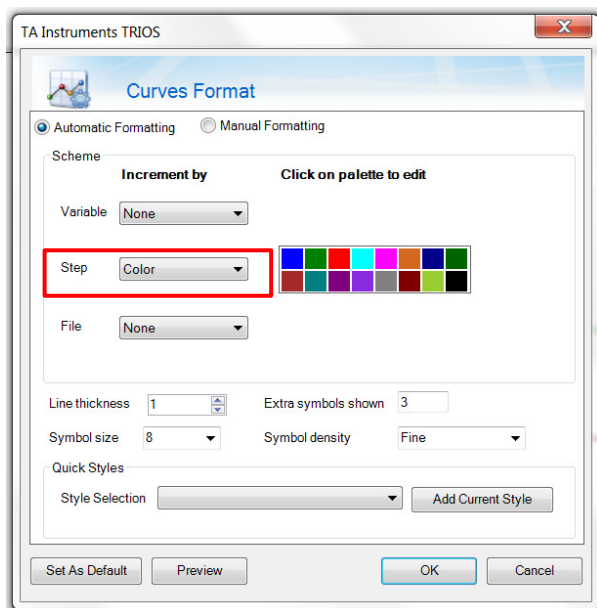
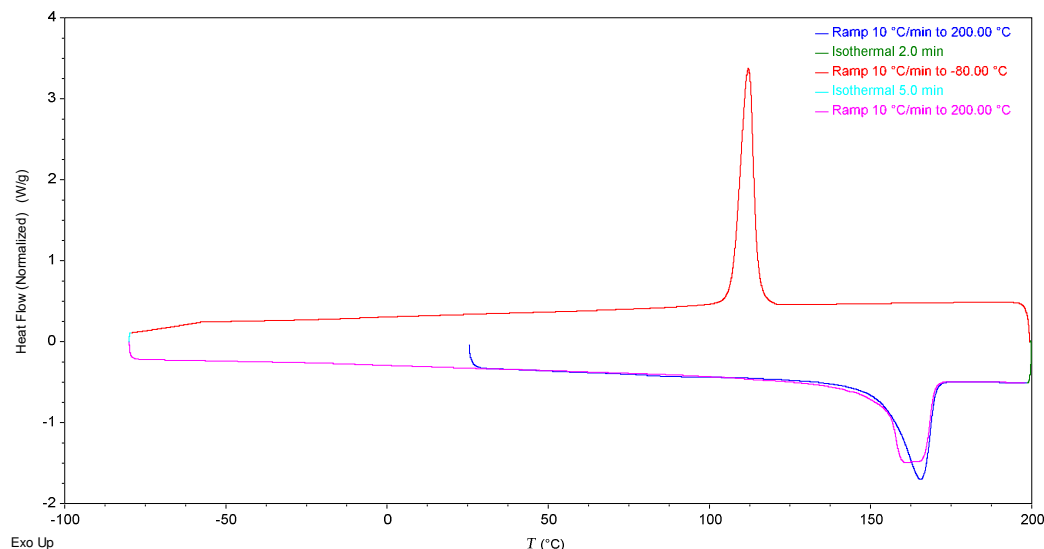
Standby 40.00 °C

RCS 90 On

Ramp & Iso segment automatically trigger data collection

# Heat-Cool-Reheat Method

- 1) Ramp 10°C/min to 200°C
- 2) Isothermal for 2.00 min
- 3) Ramp 10°C/min to -80°C
- 4) Isothermal for 5.00 min
- 5) Ramp 10°C/min to 200°C





# Method Design Rules

- **Start Temperature**

Generally, the baseline should have 2 minutes to completely stabilise prior to the transition of interest. Therefore, at 10°C/min, start at least 20°C below the transition onset temperature.

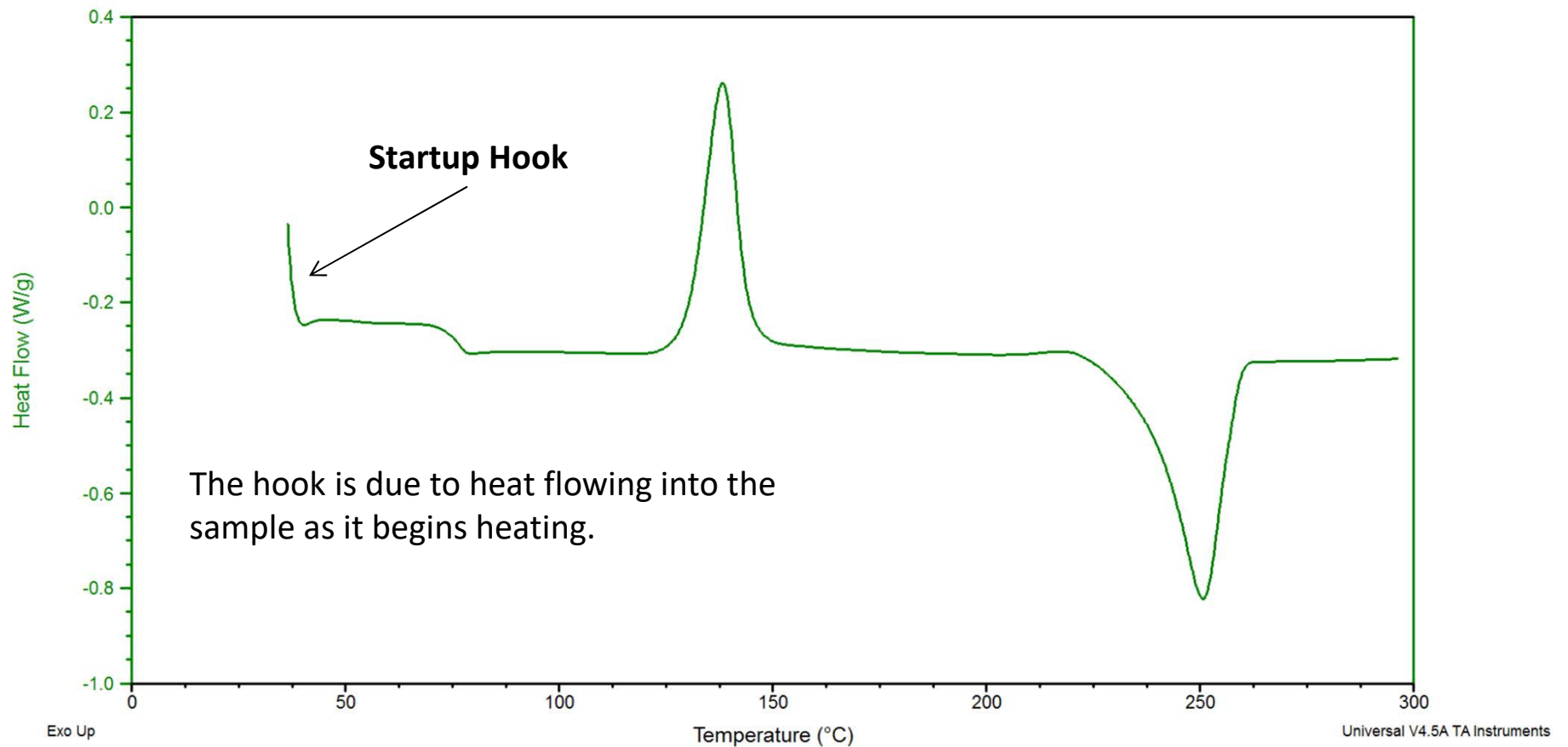
- **End Temperature**

Allow a 2 minute baseline after the transition of interest in order to correctly select integration or analysis limits.

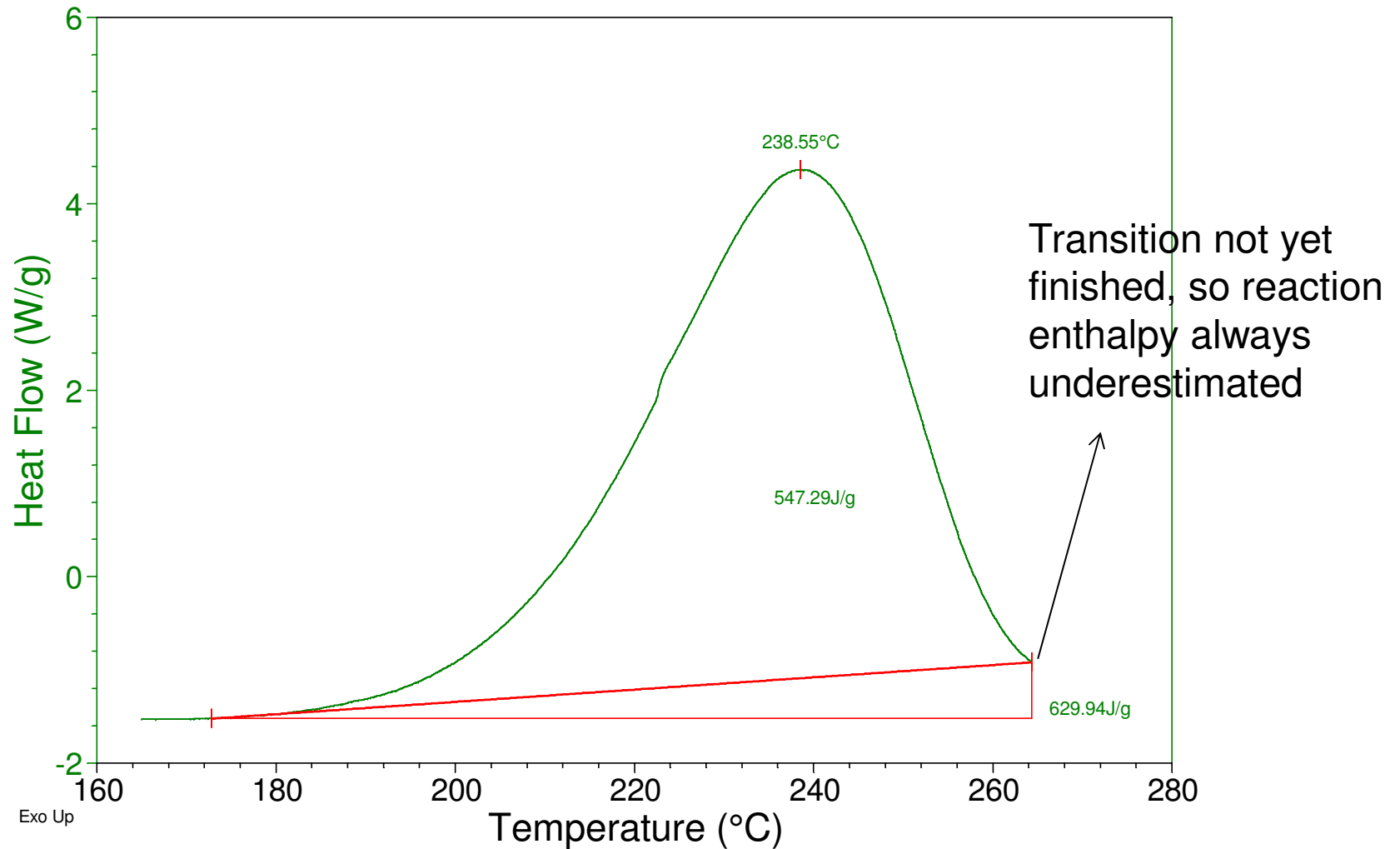
**But do not decompose the sample in the DSC cell !**

# Startup Hook in DSC

All DSC data will have an endothermic shift (on heating) at the beginning of the experiment. This is commonly referred to as the “Startup Hook”, or more specifically, the “Approach to Steady State”



# Why have 2 min of baseline after ?



# Calibration & Verification

Tzero or Baseline

Heat Flow

Temperature



# Calibration Overview

- **Tzero calibration (Tzero DSC)**

1

- Discovery DSC, DSC 2500, DSC 250  
in T4P or T4 mode

OR

- **Baseline calibration (Heat Flux DSC)**

- DSC 25
- (Tzero DSC if run in T1 mode)

AND

- **Heat flow: cell constant**

2

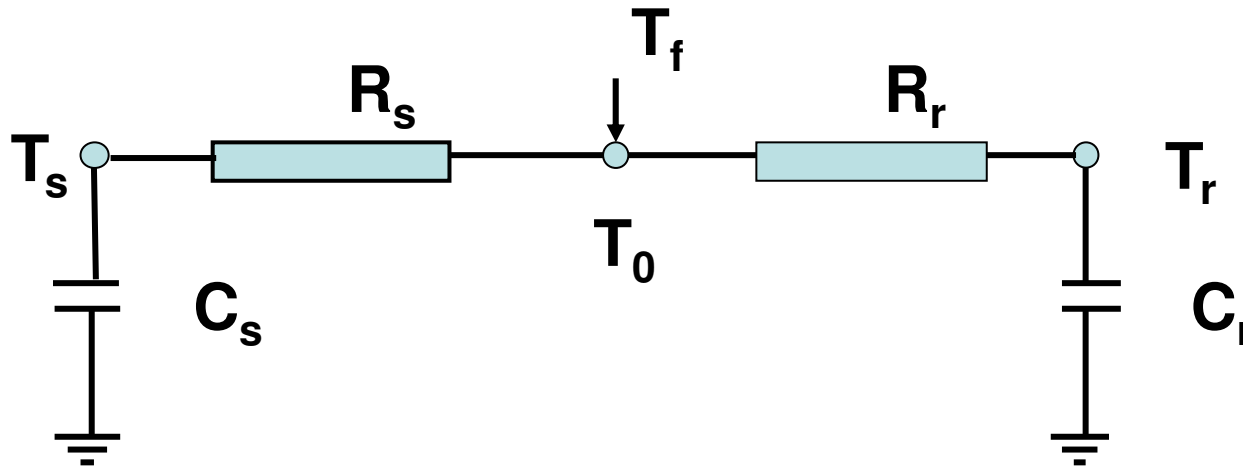
AND

- **Temperature**

Always verify first before you decide to recalibrate !

# Tzero™ Calibration for DSC 250(0) & Discovery

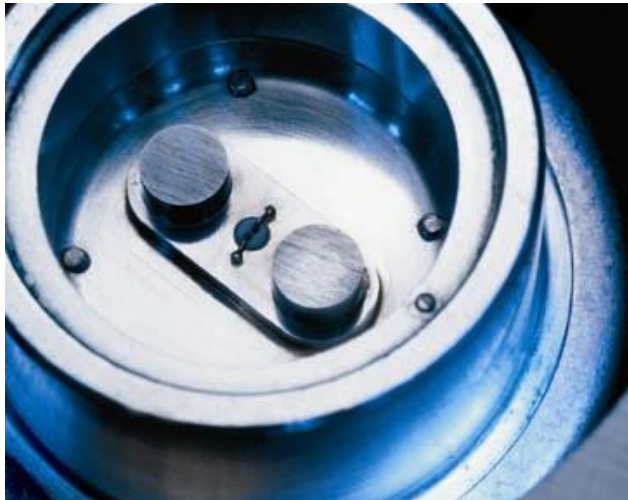
- Tzero calibration measures the C's & R's



$$q = -\frac{\Delta T}{R_r} + \Delta T_0 \left( \frac{1}{R_s} - \frac{1}{R_r} \right) + (C_r - C_s) \frac{dT_s}{dt} - C_r \frac{d\Delta T}{dt}$$

# Measuring the C's & R's

- Performed using calibration setup
  1. Run Empty Cell
  2. Run Sapphire on both Sample & Reference side



# Tzero Calibration – DSC 2500 & DSC 250

- Might be good to cycle the cell several times between min/max temp to be used for calibration (baseline conditioning) prior to Tzero calibration
- Choice of temperature range for Tzero calibration: at least as broad as desired experimental range
- Do NOT run it at a heating rate lower than 10°C/min, by preference 20°C/min
- After Tzero calibration the temperature and enthalpy calibration is mandatory performed, e.g. with indium



# Discovery DSC Tzero Calibration

2500 DSC2A-00118 (172.23.165.240) : TA Instruments Trios v4.1.1.33073

File Manager: Calibration Setup

Calibration Experiment Setup

Cell Conditioning  Tzero  Temperature  Reversing Heat Capacity

Baseline Conditioning  Cell Constant/Temperature  Direct Heat Capacity

Tzero Cell Constant/Temperature

Sample Name: Tzero Calibration

	Pan Number	Sample Mass	Pan Mass
Sample	53	94.900 mg	0.000 mg
Reference	54	97.990 mg	0.000 mg

Pan Type: None (T4P only)

Ramp Rate: 20 °C/min

Lower Temperature: -90.00 °C

Upper Temperature: 400.00 °C

Isothermal: 10.0 min

Perform Verification Run After Calibration (Baseline verification)

File Path: C:\ProgramData\TA Instruments\TRIOS\Data

Run All Queue All Schedule All

Control Panel: Idle 39.95 °C

Signals [16 of 16]	Value	Units
Method Time	0.00 min	
Remaining Segment Time	0.00 min	
Remaining Time	0.00 min	
Cell Purge	50.00 mL/min	
Temperature	39.95 °C	
Flange Temperature	-73.36 °C	
Heat Flow	-19.147 μW	
Set Point Temperature	40.00 °C	
Power Delivered	33.6712 W	
Base Purge	291.73 mL/min	
Tzero Temperature Unfit	39.96 °C	
Heat Capacity	0.00000 J/°C	
Reference Junction Temperature	46.64 °C	
Heater Temp	41.00 °C	

General

Temperature: 39.95 °C

Flange Temperature: -73.36 °C

Cooler Selection: RCS 90

Standby: 40.00 °C

RCS 90: On

Gas 1 Nitrogen: 50.00 mL/min

Base Purge: 291.73 mL/min

Gas 1: Nitrogen

Flow Rate: 50 mL/min

Auto Sampler

# Discovery DSC Calibration

2500 DSC2A-001118 (172.23.165.240) - TA Instruments Trios v4.1.1.33073

File Manager: Calibration

Calibration Experiment Setup

Cell Conditioning  Tzero  Temperature  Reversing Heat Capacity

Baseline Conditioning  Cell Constant/Temperature  Direct Heat Capacity

Tzero **Cell Constant/Temperature**

Pan Number Sample Mass Pan Mass Pan Type  
Reference 46 0.000 mg 50.910 mg Tzero Aluminum

Operator: ev  
Project: calibration

Notes:

Insert Isothermal 1.0 min  
Ramp 5 °C/min

Premelt	Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input checked="" type="checkbox"/>	Indium	156.598	131.59	171.59	52	3.850	50.710

Calibration  Perform Verification after Calibration

Verification Criteria: Temperature ± 0.1 °C Enthalpy ± 2 %

Premelt	Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input type="checkbox"/>	Indium	156.598	131.59	171.59	52	3.850	50.710

File Path: C:\ProgramData\TA Instruments\TRIOS\Data

Run All Queue All **Schedule All**

TA Instruments TRIOS - Add New Schedule

Scheduled Sequence Name: \_\_\_\_\_

Frequency: Weekly

Time: 01:00 AM

Begin Date: 21/01/2015

End Date: 21/02/2015

No end date

Apply Cancel

Control Panel: Idle 39.99 °C

Signals [16 of 16]	Value	Units
Method Time	0.00	min
Remaining Segment Time	0.00	min
Remaining Time	0.00	min
Cell Purge	50.00	mL/min
Temperature	39.99	°C
Flange Temperature	-81.93	°C
Heat Flow	-4.283	µW
Set Point Temperature	40.00	°C
Power Delivered	37.5483	W
Base Purge	291.84	mL/min
Tzero Temperature Unfit	40.00	°C
Heat Capacity	0.00000	J/°C
Reference Junction Temperature	46.64	°C
Heater Temp	40.49	°C

General

Temperature 39.99 °C

Flange Temperature -81.93 °C

Cooler Selection RCS 90

Standby 40.00 °C

RCS 90 On

Gas 1 Nitrogen: 50.00 mL/min

Base Purge 291.84 mL/min

Gas 1: Nitrogen

Flow Rate 50 mL/min

General

Auto Sampler

Ready: Run Status Idle End Status NotRun Cortex GDM FEP FCM Auto Sampler 11:37:58 AM

# Discovery DSC Calibration

2500 DSC2A-001118 (172.23.165.240) - TA Instruments Trios v4.1.1.33073

File Manager: Calibration

Calibration Experiment Setup

Cell Conditioning  Tzero  Temperature  Reversing Heat Capacity

Baseline Conditioning  Cell Constant/Temperature  Direct Heat Capacity

Tzero Cell Constant/Temperature

Reference: 46 0.000 mg 50.910 mg Tzero Aluminum

Operator: ev

Project: calibration

Notes:

Insert Isothermal 1.0 min

Ramp: 5 °C/min

Calibration Experiments

Premelt	Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input checked="" type="checkbox"/>	Indium	156.598	131.59	171.59	52	3.850	50.710

Calibration  Perform Verification after Calibration

Verification  Perform Calibration if Verification fails

Verification Criteria: Temperature  $\pm$  0.1 °C Enthalpy  $\pm$  2 %

Verification Experiments

Premelt	Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input type="checkbox"/>	Indium	156.598	131.59	171.59	52	3.850	50.710

File Path: C:\ProgramData\TA Instruments\TRIOS\Data

Run All Queue All Schedule All

Control Panel: Idle 39.99 °C

Signals [16 of 16]	Value	Units
Method Time	0.00 min	
Remaining Segment Time	0.00 min	
Remaining Time	0.00 min	
Cell Purge	50.00 mL/min	
Temperature	39.99 °C	
Flange Temperature	-81.93 °C	
Heat Flow	-4.283 $\mu$ W	
Set Point Temperature	40.00 °C	
Power Delivered	37.5483 W	
Base Purge	291.84 mL/min	
Tzero Temperature Unfilt	40.00 °C	
Heat Capacity	0.00000 J/°C	
Reference Junction Temperature	46.64 °C	
Heater Temp	40.49 °C	

General

base Purge 291.84 mL/min

Gas 1: Nitrogen

Flow Rate 50 mL/min

Auto Sampler

Ready: Run Status Idle End Status NotRun Cortex GDM FEP FCM Auto Sampler 11:37:58 AM

for running calibration immediately, first send to queue to check if it looks OK before starting it

# Discovery DSC Calibration

2500 DSC2A-00118 (172.23.165.240) : TA Instruments Trios v4.1.1.33073

File Manager Experiments Control Panel

Experiments

Incomplete Queue (0)

Running Queue (5)

- Run 1 - [Pan 0 - Tzero]
- Run 2 - [Pan 53 - Tzero]
- Run 3 - [Pan 0 - Baseline]
- Run 4 - [Pan 52 - Cell Constant/Temperature]
- Run 5 - [Pan 52 - Verify Cell Constant/Temperature]

Design View (0)

Create New Runs

Load Sequence File

Experiments

History

Results

Calibration

Run 1 in Running Queue

Sample

Sample Name: Tzero Calibration

Pan Number	Sample Mass	Pan Mass
0	0.000 mg	0.000 mg

Reference: 0, 0.000 mg, 0.000 mg

Edit Tray Configuration

Pan Type: None

Operator: ev

Project: calibration

Notes:

File Name: C:\ProgramData\TA Instruments\TRIOS\Data\Tzero Calibration.tri

Procedure

Test: Tzero

Name: Tzero

Template Segments

Ramp Rate	20	°C/min
Lower Temperature	-90.00	°C
Upper Temperature	400.00	°C
Isothermal	10.0	min

Change file names in the running queue to more comprehensive ones and then start it; e.g.

1. Tzero empty
2. Tzero sapphire
3. empty cell verification
4. Indium calibration
5. Indium verification

Control Panel

Idle 40.00 °C

Signals [16 of 16]	Value	Units
Method Time	0.00	min
Remaining Segment Time	0.00	min
Remaining Time	0.00	min
Cell Purge	50.00	mL/min
Temperature	40.00	°C
Flange Temperature	-82.53	°C
Heat Flow	-2.040	µW
Set Point Temperature	40.00	°C
Power Delivered	39.0512	W
Base Purge	291.84	mL/min
Tzero Temperature Unfit	40.00	°C
Heat Capacity	0.00000	J/°C
Reference Junction Temperature	46.64	°C
Heater Temp	40.30	°C

General

Temperature 40.00 °C

Flange Temperature -82.53 °C

Cooler Selection RCS 90

Standby 40.00 °C

RCS 90 On

Gas 1 Nitrogen: 50.00 mL/min

Base Purge 291.84 mL/min

Gas 1: Nitrogen

Flow Rate 50 mL/min

General

Auto Sampler

Run Status: Idle End Status: NotRun Cortex GDM FEP FCM Auto Sampler 11:40:59 AM

# Viewing Tzero Calibration Results

The screenshot displays the 'T Zero Calibration' software interface. The window title is 'TZeroCalib...628\_1942'. The main content is divided into several sections:

- Calibration Source Information:** Contains fields for 'Calibration name' (TZeroCalibration\_DRC-00071\_20120628\_1942, circled in red), 'Description' (From -90.38 °C to 395.80 °C at 19.954°C/min), 'Cell serial number' (DRC-00071), 'Baseline run' (T0 Calibration(12)), and 'Sapphire run' (T0 Calibration(13)). There are 'Save Results' and 'Save & Apply' buttons.
- Method Details:** Includes 'Lower temp' (-90.38 °C), 'Upper temp' (395.8 °C), 'Heating rate' (19.95 °C/min), 'Pan type' (None), 'Purge gas' (Nitrogen), 'Cooler type' (RCS 90 Cooler), and 'Analysis' (Sapphire no pans).
- Parameters:** A table showing calibration parameters:

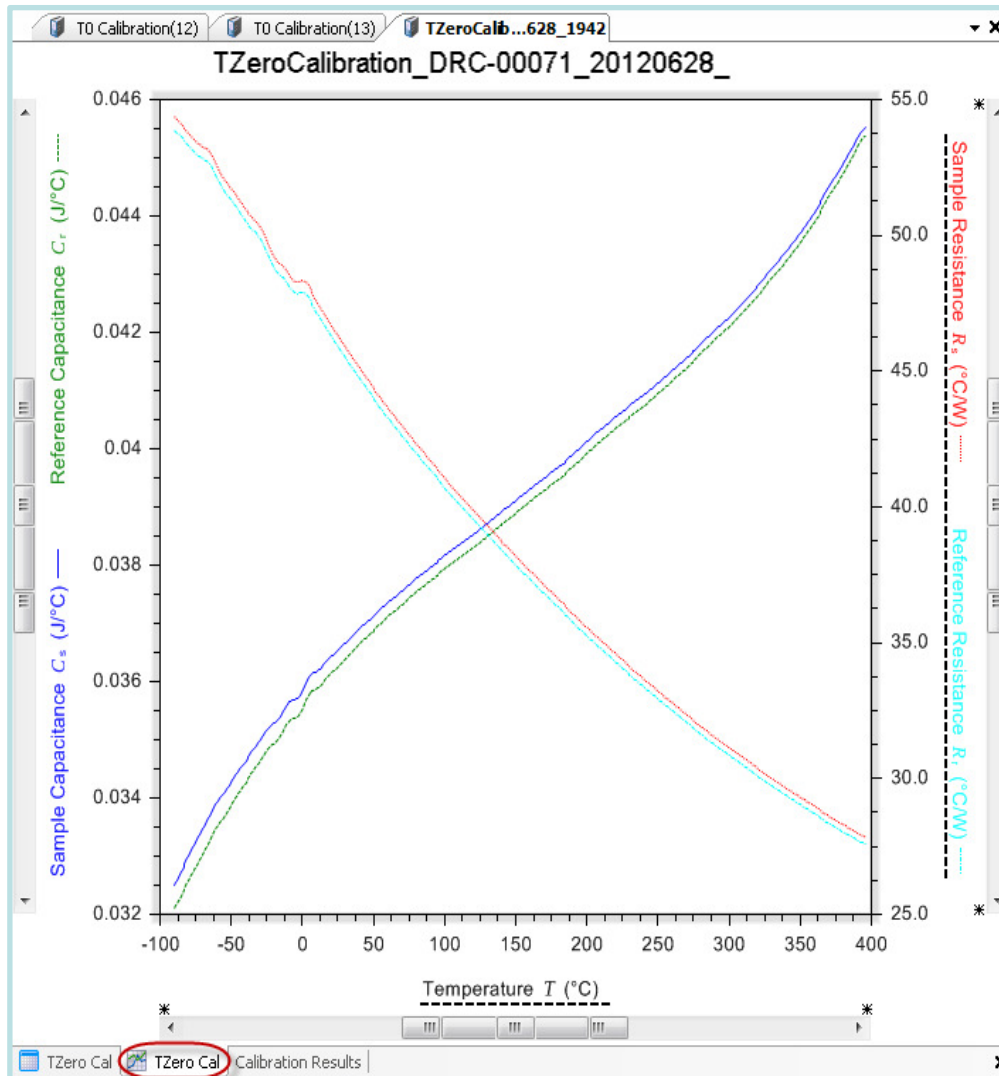
	Slope	Offset
Delta T	-1.336e-03 $\mu\text{V}/^\circ\text{C}$	-10.635838 $\mu\text{V}$
Delta TZero	0.01057952 $\mu\text{V}/^\circ\text{C}$	13.0938287 $\mu\text{V}$

At the bottom, a taskbar shows three tabs: 'TZero Cal', 'TZero Cal', and 'Calibration Results'. The 'Calibration Results' tab is circled in red, with a red arrow pointing to it from the text 'Click to see graph'.

Note that calibration results are automatically saved to the equipment

Click to see graph

# Viewing Tzero Calibration Results



Characteristics of the thermal resistances and heat capacities:

- Both curves should be smooth, with no steps, spikes or inflection points.
- Thermal resistances should always have negative slope that gradually decreases.
- Heat capacities should always have positive slope.

This cell is very well balanced. It is acceptable and usual to have larger differences between sample and reference.



# Viewing Existing Tzero Calibration

The screenshot displays the TA Instruments software interface for viewing calibration data. The main window is titled "Calibration" and shows three calibration entries:

- TZero Calibration 4/24/2017 4:06 PM (Applied)**
  - Name: TZeroCalibrationDSC2A-00118\_20170424\_1605.tri
  - Description: From -88.352 °C to 397.543 °C at 20.000 °C/min
  - Temperature Range: Lower Temperature: -88.352 °C, Upper Temperature: 397.543 °C, Heating Rate: 20.000 °C/min
  - Slope and Offsets: Delta T: -0.0016 0.003, Delta TZero: 0.0121 14.789
  - Source Files: C:\TA\Users\TA\DSC\calibration\april 24 2017\Tzero Calibration empty\_ev\_4242017.tri, C:\TA\Data\DSC\calibration\april 24 2017\Tzero Calibration sapphire\_ev\_4242017.tri
- Cell Constant Calibration 4/24/2017 4:42 PM (Applied)**
  - Name: TempCalibrationDSC2A-00118\_20170424\_1642.tri
  - Heating Rate: 10 °C/min
  - KCell Information: Standard: Indium, Applied KCell: 1.052
- Temperature Calibration 4/24/2017 4:42 PM (Applied)**
  - Name: TempCalibrationDSC2A-00118\_20170424\_1642.tri
  - Heating Rate: 10 °C/min
  - Temperature Offset: TMelt (Ref): 156.598, TMelt (meas): 158.408

Red annotations highlight the "Calibration Data" tab and the "Source Files" section. A red text overlay reads: "Graphs of the original datafiles, no C&R".

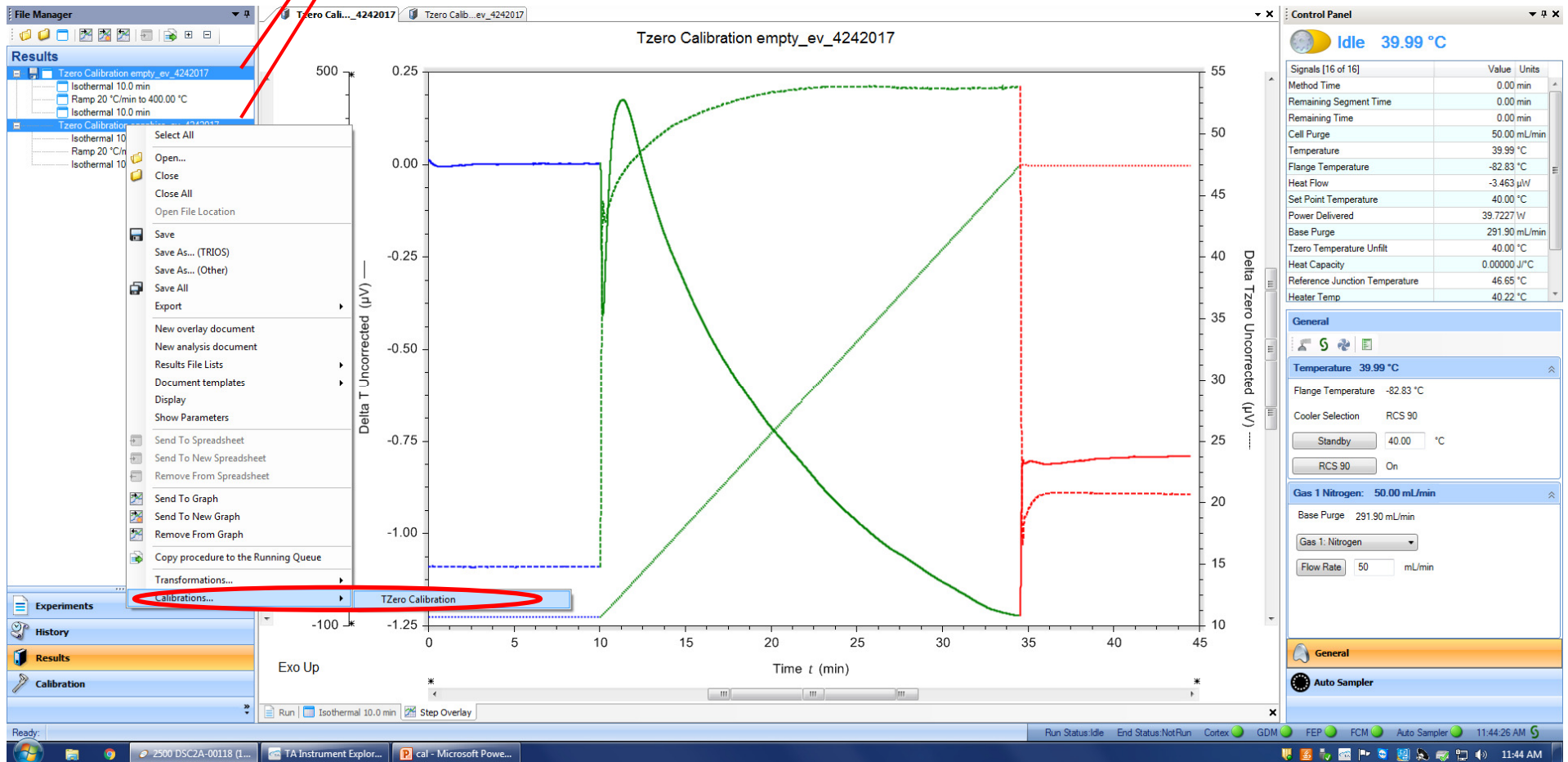
The right-hand side of the interface shows a "Control Panel" with a "Signals [16 of 16]" table:

Signal	Value	Units
Method Time	0.00	min
Remaining Segment Time	0.00	min
Remaining Time	0.00	min
Cell Purge	49.99	mL/min
Temperature	40.00	°C
Flange Temperature	-82.60	°C
Heat Flow	-3.208	µW
Set Point Temperature	40.00	°C
Power Delivered	37.6080	W
Base Purge	291.87	mL/min
Tzero Temperature Unfilt	40.00	°C
Heat Capacity	0.00000	J/°C
Reference Junction Temperature	46.65	°C
Heater Temp	40.24	°C

The bottom status bar shows the system is ready and the time is 11:43 AM.

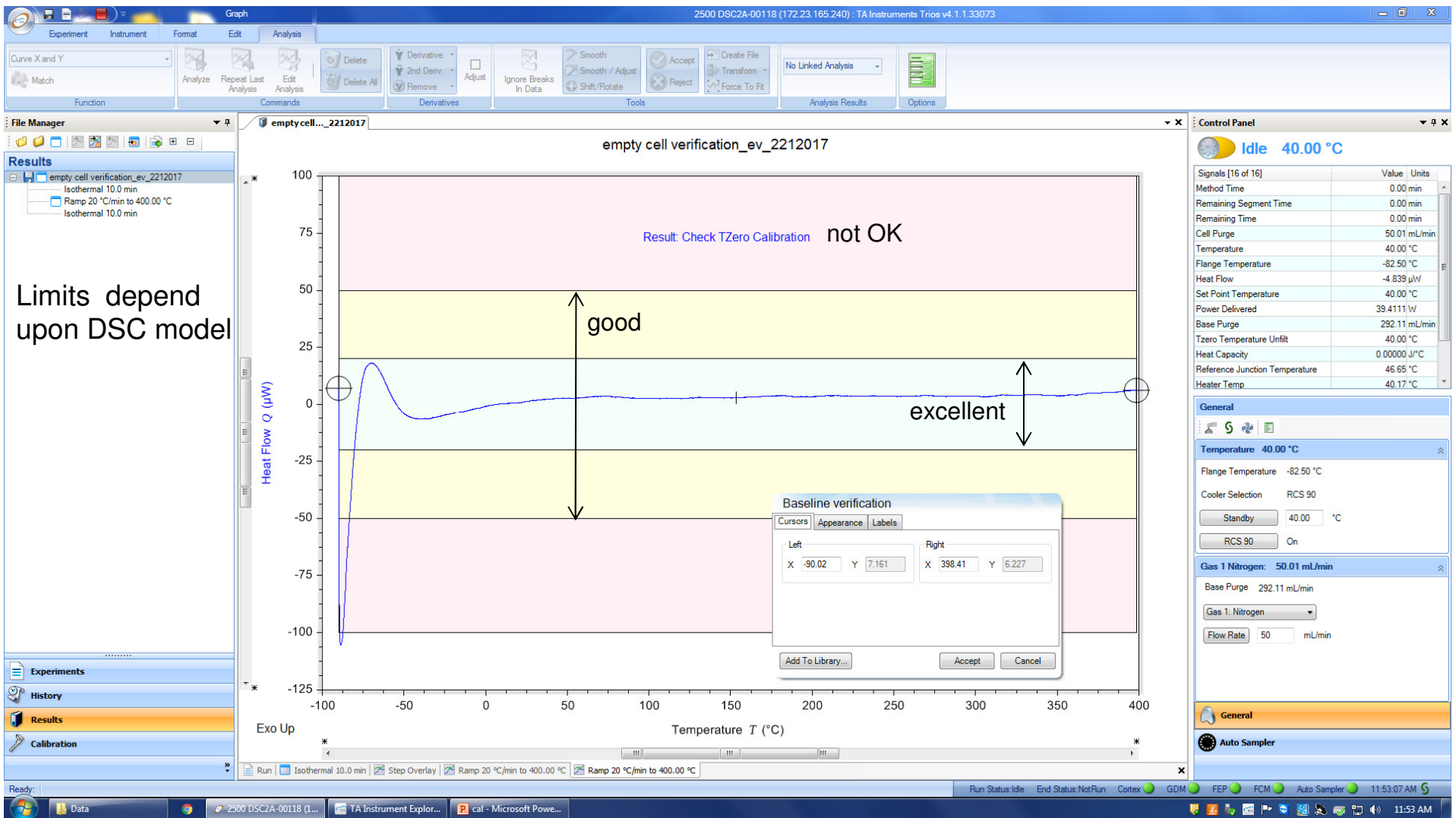
# Viewing Existing Tzero Calibration

Highlight both files simultaneously, right click, calibrations, Tzero calibration, C&R are calculated, do not apply



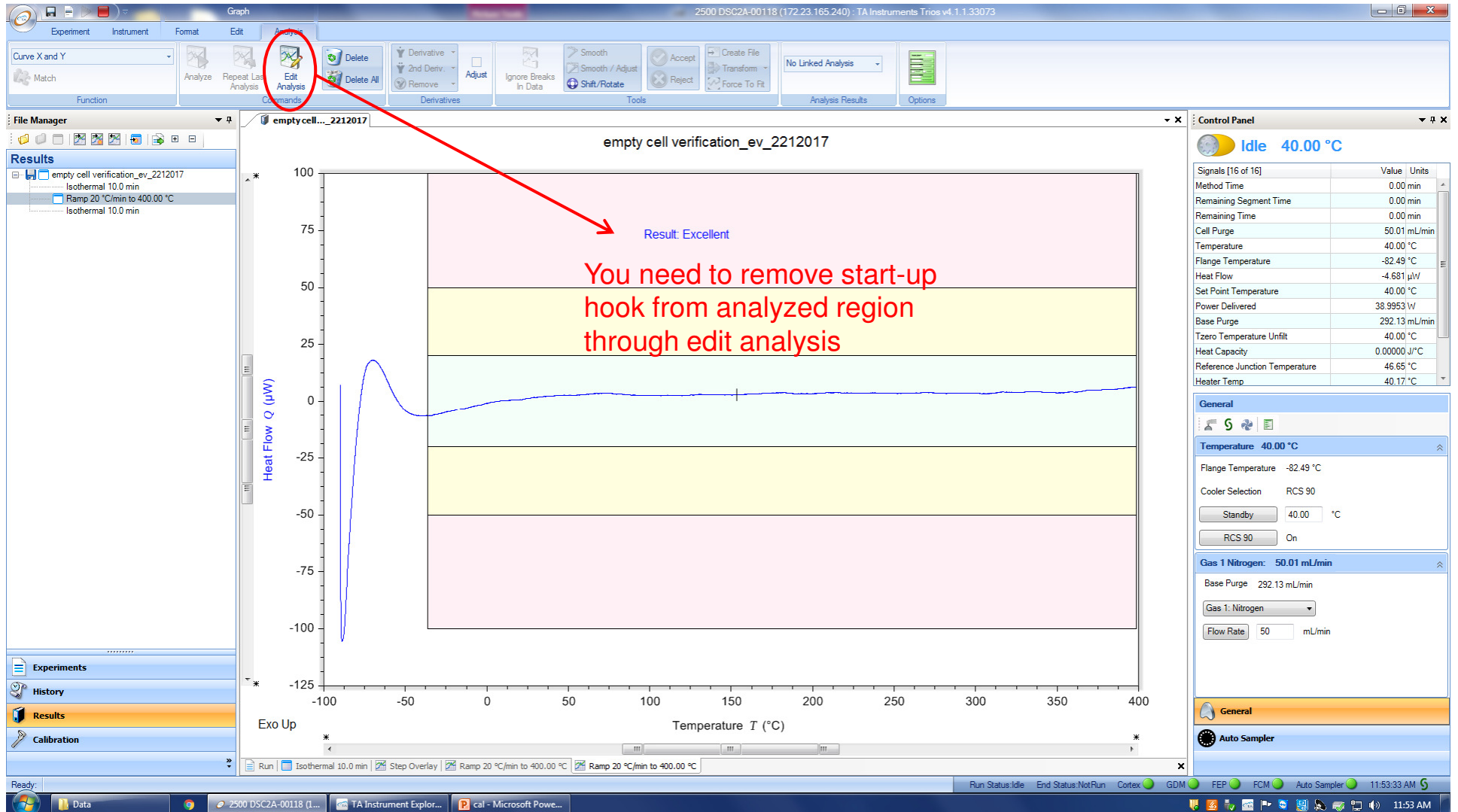


# Baseline Verification



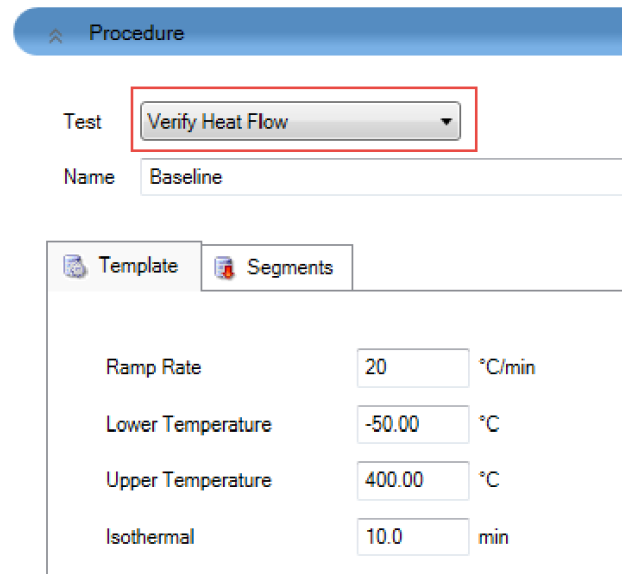
Limits depend upon DSC model

# Baseline Verification



# Tzero Calibration & Baseline Verification

- Always verify first the baseline before deciding to recalibrate Tzero !



Procedure

Test: Verify Heat Flow

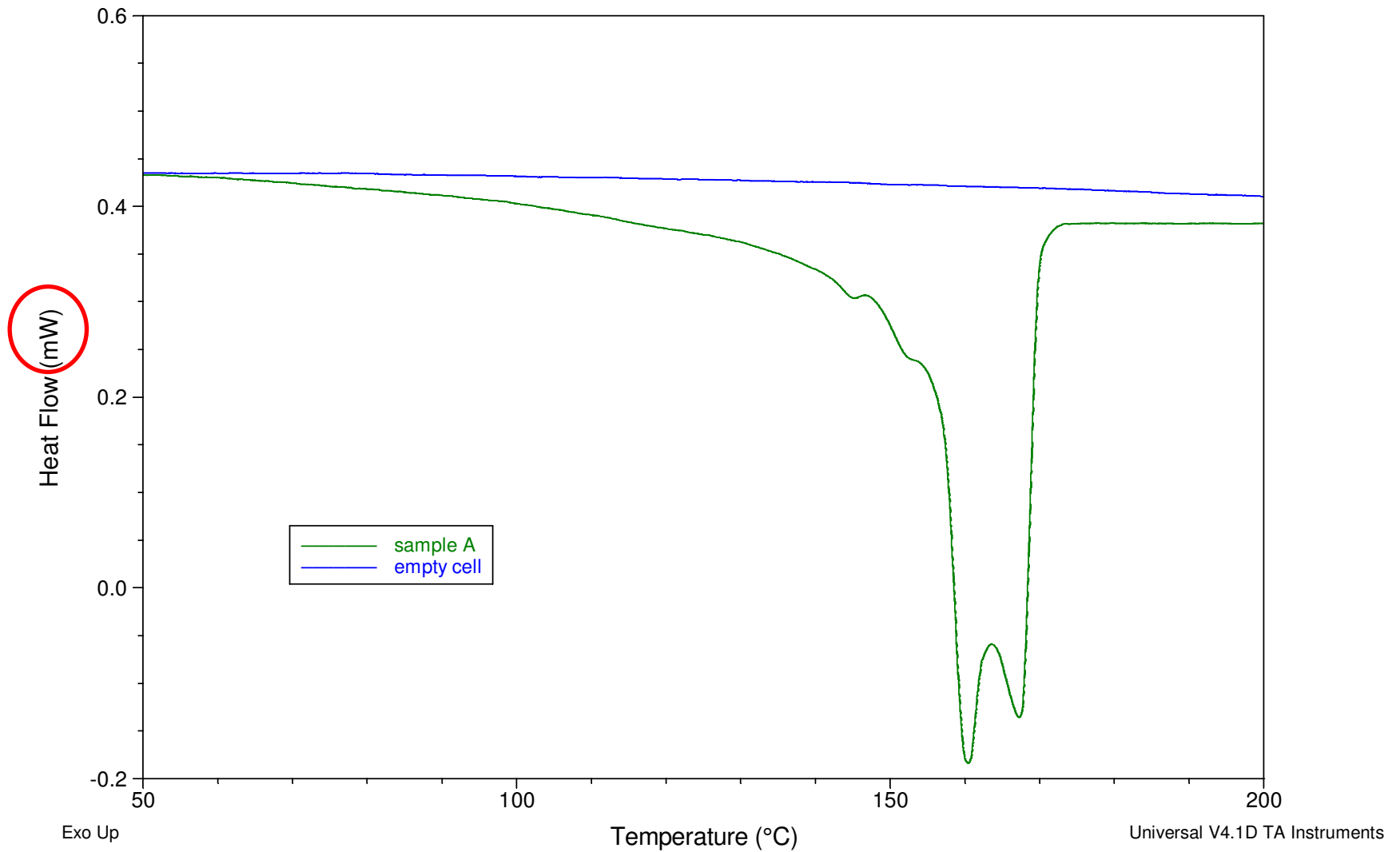
Name: Baseline

Template Segments

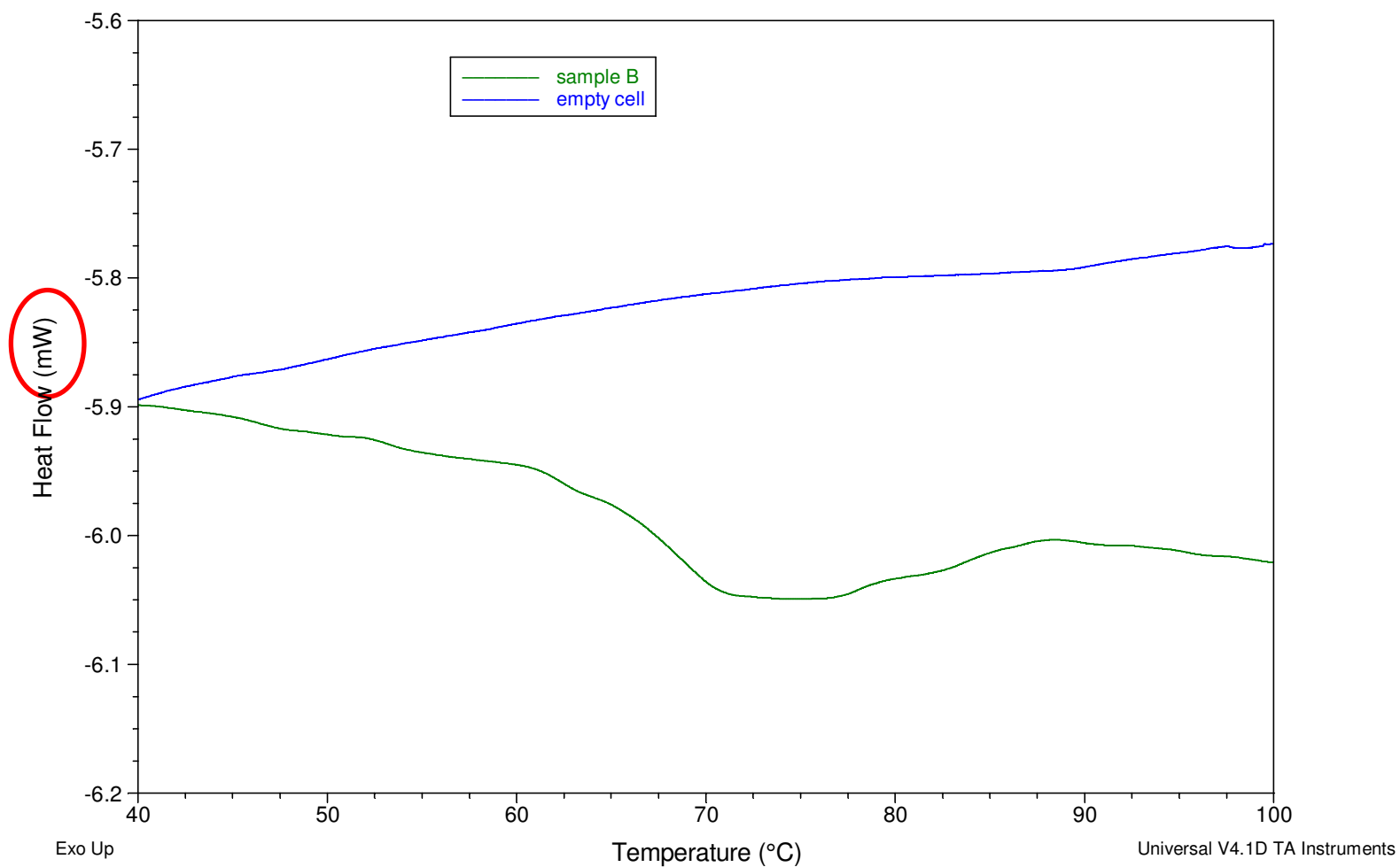
Ramp Rate	20	°C/min
Lower Temperature	-50.00	°C
Upper Temperature	400.00	°C
Isothermal	10.0	min

- Even if the verification is not OK, compare with sample transitions to check if flattest baseline is really required (plot overlay in mW)

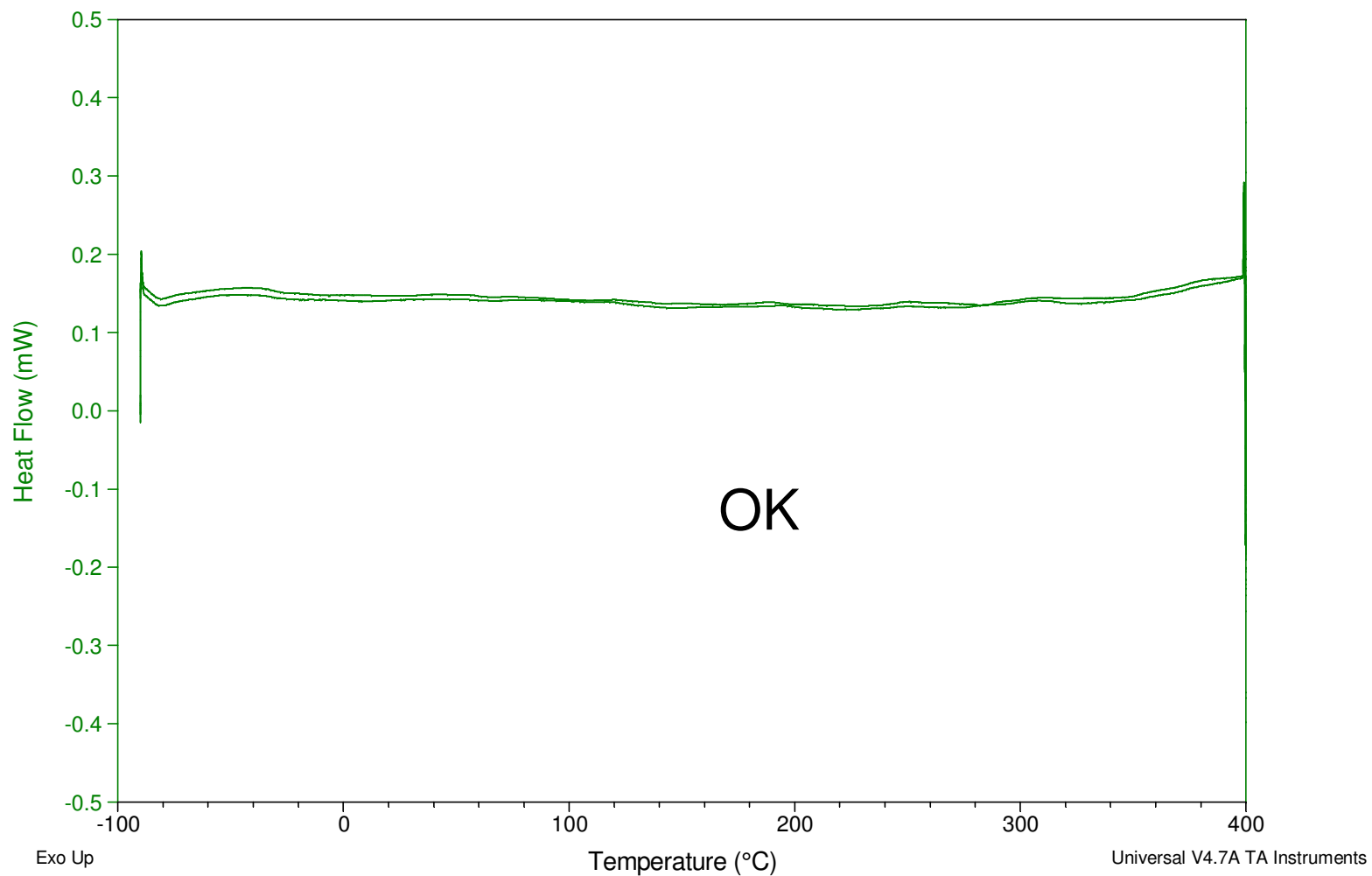
# When to recalibrate ? **NO**



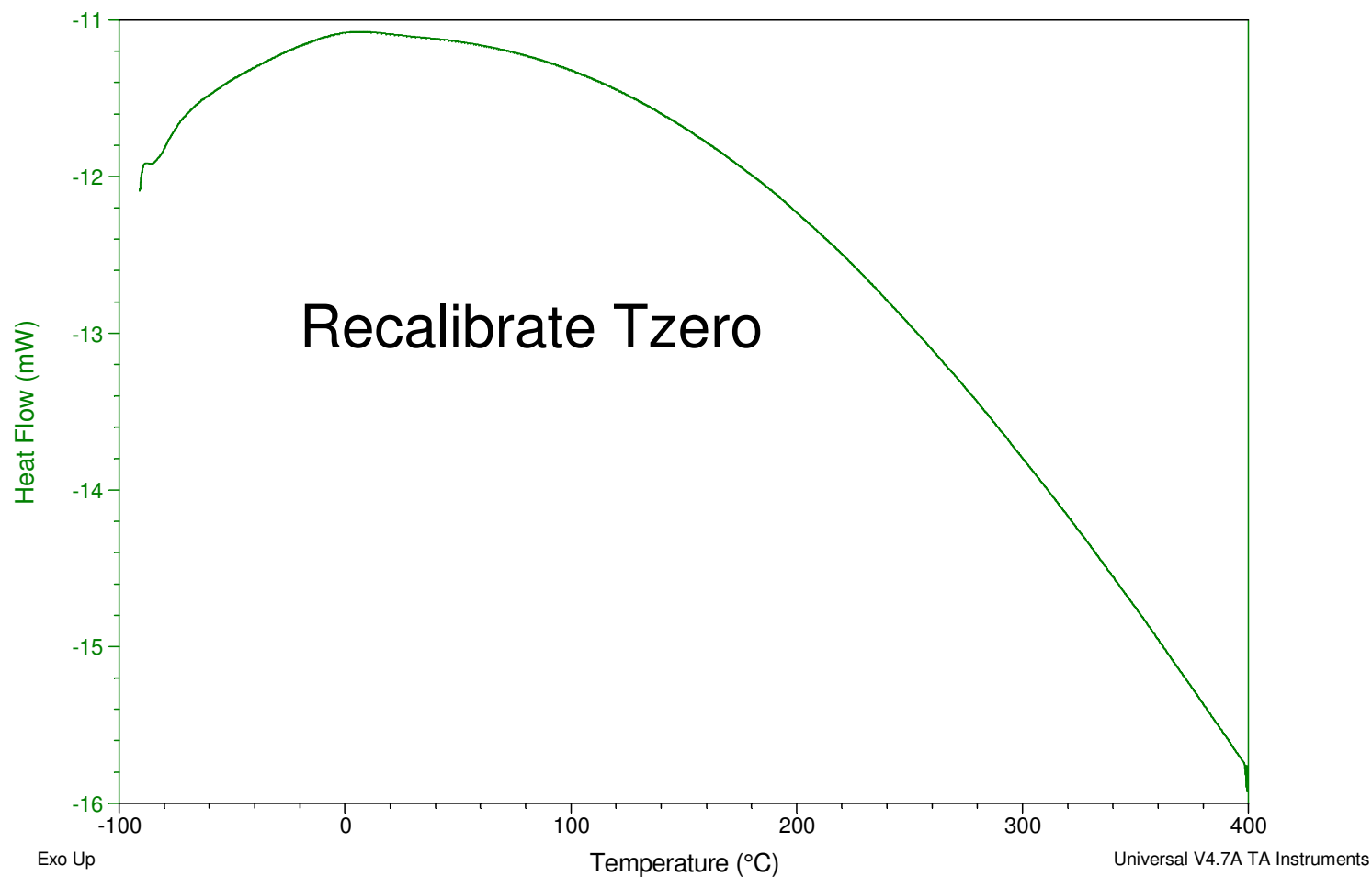
# When to recalibrate ? **YES**



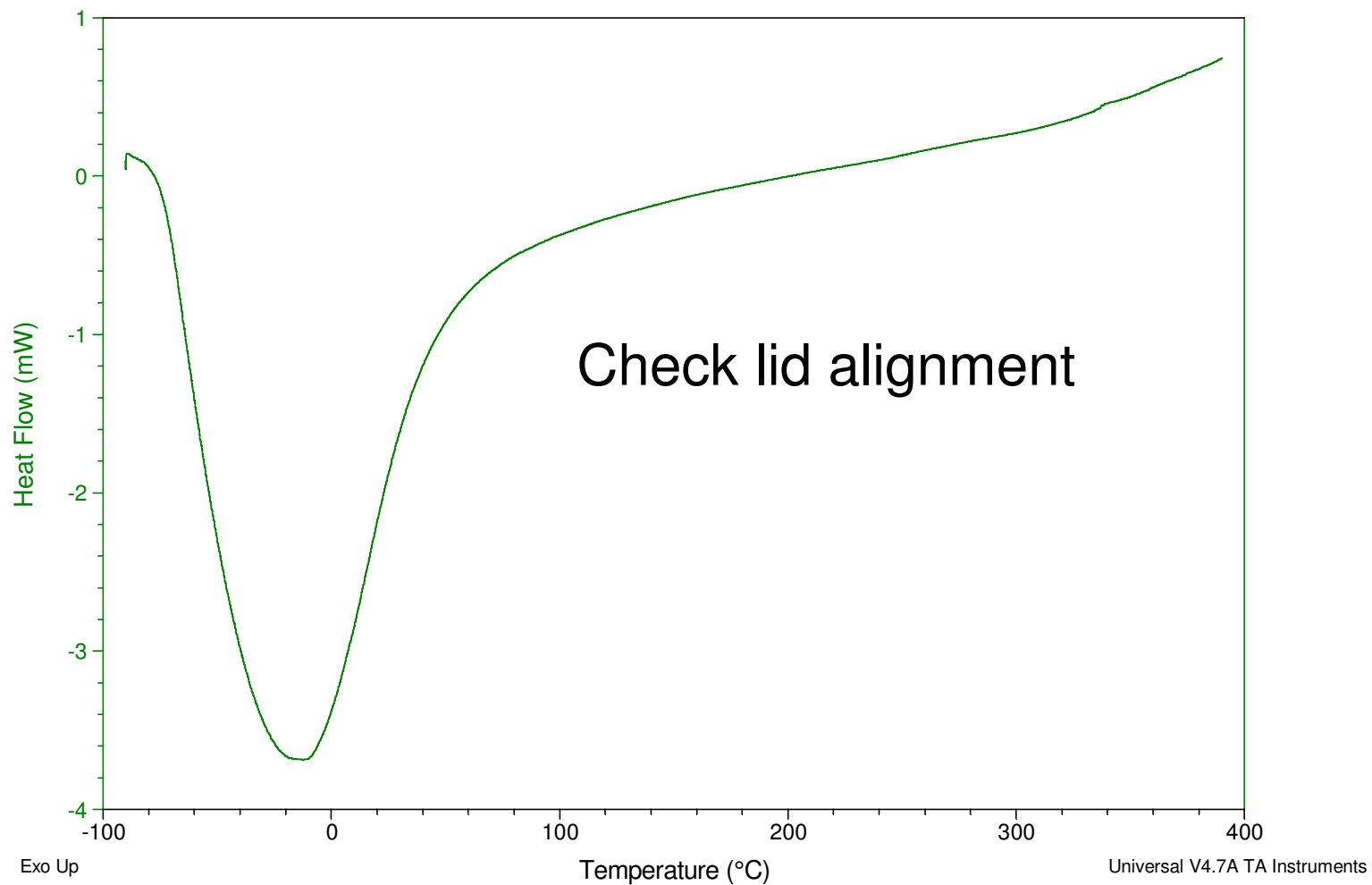
# Baseline OK ? If not which action ?



# Baseline OK ? If not which action ?

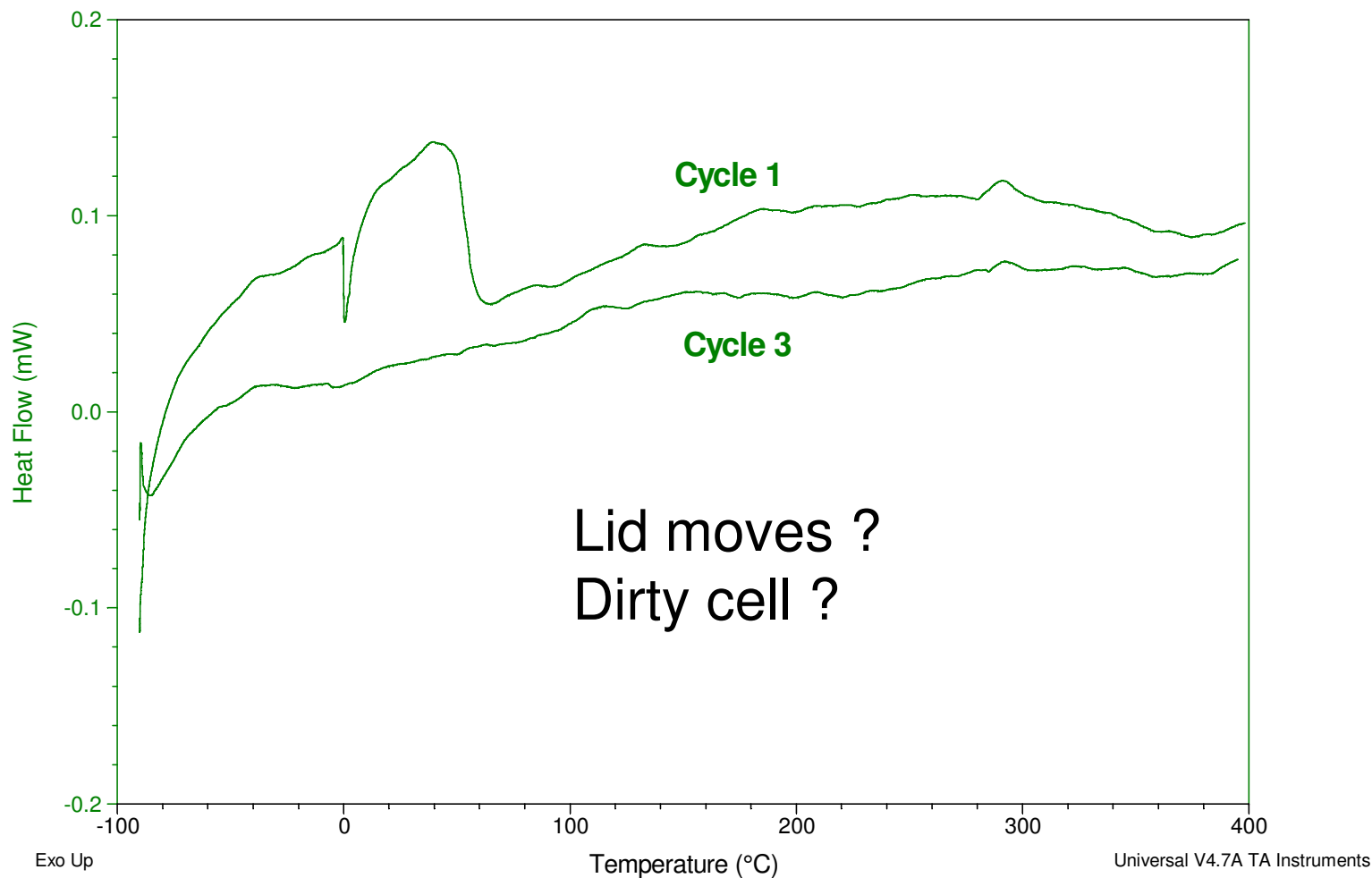


# Baseline OK ? If not which action ?





# Baseline OK ? If not which action ?



# Baseline Calibration – DSC 25

The screenshot shows the TA Instruments TRIOS software interface for DSC 25 calibration. The main window is titled "Calibration Experiment Setup". In the "Calibration Setup" tab, the "T1 Baseline" checkbox is selected and circled in red. Other options include "Cell Conditioning", "Temperature", "Reversing Heat Capacity", "Baseline Conditioning", and "Cell Constant/Temperature". The "T1 Baseline" section contains the following parameters:

Parameter	Value	Units
Sample Name	T1 baseline	
Operator	ev	
Project		
Notes		
Ramp Rate	20	°C/min
Lower Temperature	20.00	°C
Upper Temperature	200.00	°C
Isothermal	10.0	min
Perform Verification Run After Calibration	<input checked="" type="checkbox"/>	

At the bottom of the "T1 Baseline" section, there are buttons for "Run All", "Queue All", and "Schedule All". The "File Path" is set to "C:\ProgramData\TA Instruments\TRIOS\Data".

On the right side, the "Control Panel" shows the current temperature as "Idle 39.99 °C". Below this, there is a table of signals:

Signals [15 of 15]	Value	Units
Method Time	0.00	min
Remaining Segment Time	0.00	min
Remaining Time	0.00	min
Cell Purge	50.01	mL/min
Temperature	39.99	°C
Flange Temperature	-79.38	°C
Heat Flow	168.633	µW
Set Point Temperature	40.00	°C
Power Delivered	39.4094	W
Base Purge	292.72	mL/min
Tzero Temperature Unfilt	40.00	°C
Reference Junction Temperature	46.63	°C
Heater Temp	40.17	°C
Power Request on Power Supply	55.6184	W

Below the signals table, there are sections for "General", "Temperature 39.99 °C", "Gas 1 Nitrogen: 50.01 mL/min", and "Auto Sampler".

At the bottom of the software window, there is a taskbar with several open applications: "training", "2500 DSC2A-00118 (1...", "TA Instrument Explor...", and "cal - Microsoft Powe...". The system tray shows the time as 3:18 PM.

Overlaid on the bottom center of the screenshot is the following text:

Calibration: 1 run of empty cell (no cups)  
Verification: 1 run of empty cell (no cups)

# Baseline Calibration – DSC 25

The screenshot displays the TA Instruments software interface for DSC 25. The main window is titled "T1 Calibration" and shows the following details:

- Name:** T1CalibrationDSC2A-00118\_20170428\_1556.tri
- Description:** From 69.854 °C to 149.871 °C at 20.000 °C/min
- Temperature Range:**
  - Lower Temperature: 69.854 °C
  - Upper Temperature: 149.871 °C
  - Heating Rate: 20.000 °C/min
- Slope and Offsets:**
  - Delta T Slope: -0.003 µV/°C
  - Delta T Offset: -0.589 µV
  - Initial Temperature: 69.854 °C

Red annotations highlight the "Name" field, the "Adjust Analysis" button, and the "T1 baseline calibration" tab in the bottom taskbar.

**Control Panel:** Shows the system is "Running" at 198.92 °C with an "Isothermal 10.0 min" segment. A progress bar indicates 00:08:26 remaining.

Signals [15 of 15]	Value	Units
Method Time	22.34 min	
Remaining Segment Time	8.44 min	
Remaining Time	8.44 min	
Cell Purge	50.01 mL/min	
Temperature	198.92 °C	
Flange Temperature	-66.08 °C	
Heat Flow	155.982 µW	
Set Point Temperature	200.00 °C	
Power Delivered	84.9166 W	
Base Purge	292.69 mL/min	

No.	Description
1	Equilibrate 20.00 °C
2	Isothermal 10.0 min
3	Ramp 20 °C/min to 200.00 °C
4	Isothermal 10.0 min

**Running Method List:**

**General:** Auto Sampler

**Running Method List:**

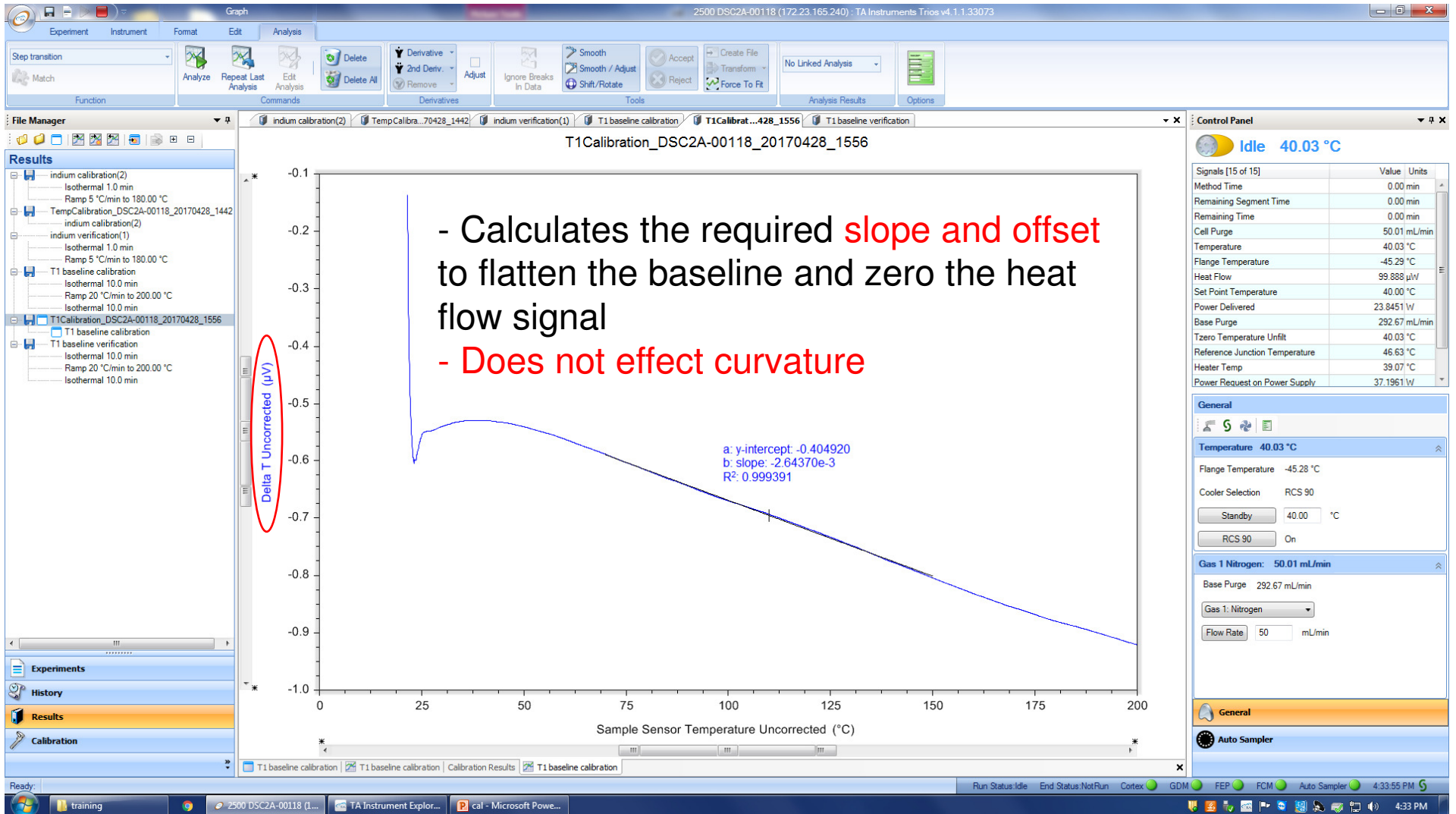
**Taskbar:** Shows "T1 baseline calibration" and "Calibration Results" tabs.

Note that calibration result is automatically saved to the equipment

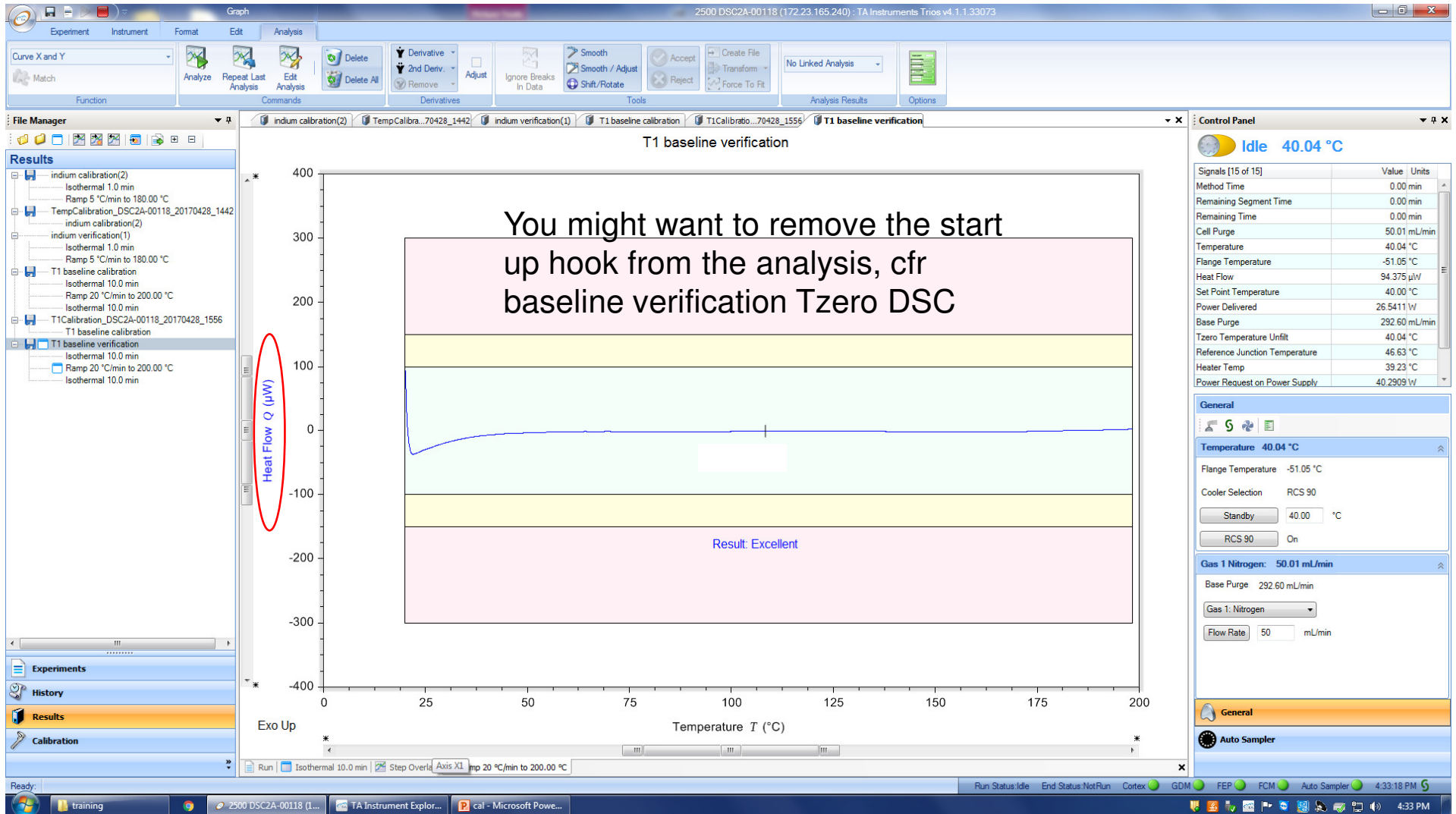
If you want to make changes to the analysis (change temp region)

To see the analyzed graph

# Baseline Calibration – DSC 25



# Baseline Verification – DSC 25



# DSC Calibrations: Cell Constant & Temperature

- Cell constant is the calorimetric calibration which corrects for non-adiabatic heat transfer (heat lost to the surroundings)
- It is performed using a well-known melting standard such as indium

$$\text{Cell Constant} = \frac{\Delta H_f (\textit{literature})}{\Delta H_f (\textit{measured})}$$

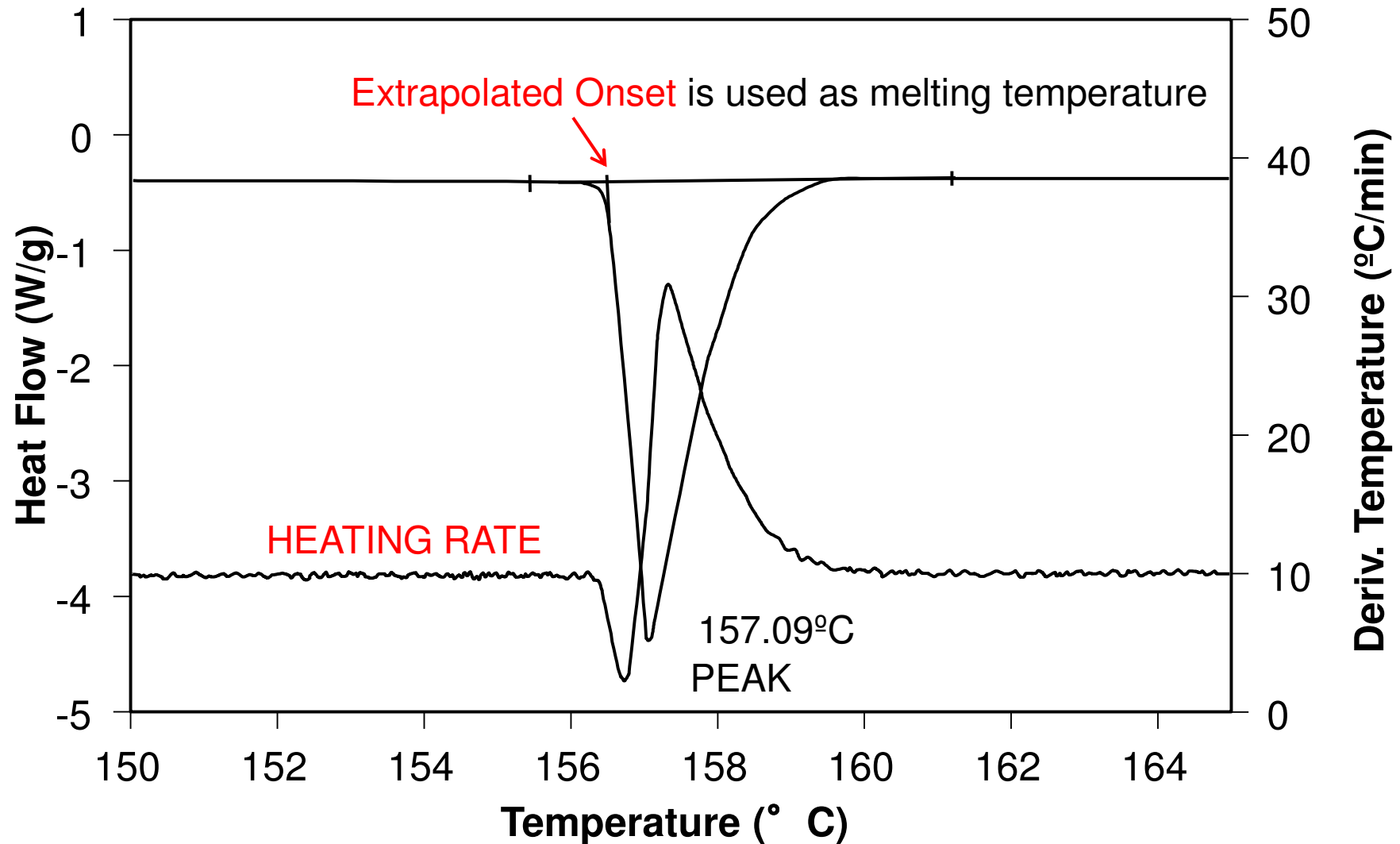
Cell constant is used as multiplication factor for heat flow; typically cell constant is close to 1.

- Temperature Calibration points are determined by comparing the measured melting onset temperature to the literature value
- TA Instruments software allows for up to 5 temperature calibration points
  - Generally, these should bracket the temperature range of interest for subsequent samples

# Heat Flow Calibration (Cell Constant)

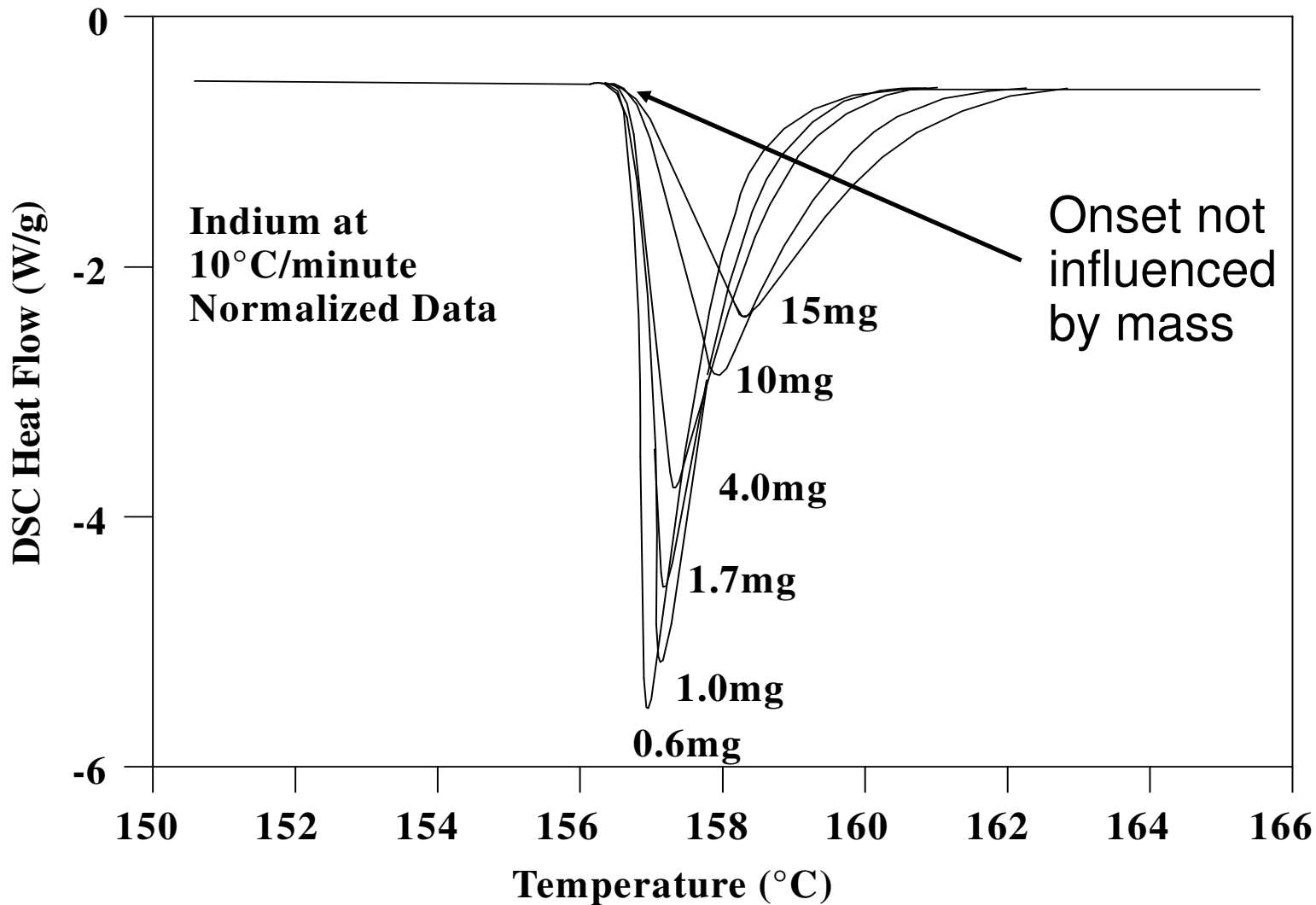
- Heat Flow Calibration of Differential Scanning Calorimeters – ASTM E-968
- Enthalpy Calibration
- One Run -> Indium metal
  - Sample Weight 1-5 mg
  - Flatten and pre-melt sample the first time you run it
  - Re-use sample max 10 times
  - Heating rate of 10°C/min recommended (heat flow calibration does not depend upon heating rate, but slow rates give more noise)
  - Dependent upon purge gas/flow rate and pan type

# Temperature Calibration





# Influence of Sample Mass



# Temperature Calibration

- ASTM E967 method
  - Pure metals typically used
  - Calibration depends upon purge gas/flow rate and pan type
  - Calibration changes a little with heating rate
  - **Temperature calibration (1 point) is automatically included when calibrating cell constant**
  - Instead of 1 single temperature multiple calibration points (up to a maximum of five) can be used (specify temperature calib then)
  - Choose the calibration standards so that their transition temperatures span the interesting measuring region
  - Use tin, lead, gallium and zinc one time only

# Temperature Calibration

- If you want to calibrate temperature with more than 1 standard choose temperature calibration and include all standards (also indium, despite the fact that indium has already been run for enthalpy calibration)
- If you want to run calibration with indium at a lower rate than 10°C/min, by preference first perform cell constant calib at 10°C/min, and subsequently temp calib only at the lower heating rate

# Temperature and Enthalpy Standards

Enthalpy  
(cell constant)

- Benzoic acid (147.3 J/g)  $T_m = 123^\circ \text{C}$
- Urea (241.8 J/g)  $T_m = 133^\circ \text{C}$
- Indium (28.71 J/g)  $T_m = 156.6^\circ \text{C}$
- Anthracene (161.9 J/g)  $T_m = 216^\circ \text{C}$
- Adamantane  $-65.54^\circ \text{C}$  20.57 J/g

Temperature

- Cyclopentane\*  $-150.77^\circ \text{C}$
- Cyclopentane\*  $-135.09^\circ \text{C}$
- Cyclopentane\*  $-93.43^\circ \text{C}$
- Cyclohexane#  $-83^\circ \text{C}$
- Water#  $0^\circ \text{C}$
- Gallium#  $29.76^\circ \text{C}$
- Phenyl Ether#  $30^\circ \text{C}$
- p-NitrotolueneE  $51.45^\circ \text{C}$
- NaphthaleneE  $80.25^\circ \text{C}$
- Indium#  $156.60^\circ \text{C}$
- Tin#  $231.95^\circ \text{C}$
- Lead\*  $327.46^\circ \text{C}$
- Zinc#  $419.53^\circ \text{C}$

\* GEFTA recommended  
*Thermochim. Acta*, 219 (1993) 333.

# ITS 90 Fixed Point

E Zone refined organic compound  
(sublimes)

see TN-11

# Traceable Calibration Materials

- Certified materials used to establish traceability of instrument calibration
- NIST: US, Gaithersburg, MD 20899
  - <http://www.ts.nist.gov/srm>
- Laboratory of the Government Chemist, UK
  - <http://www.lgc.co.uk/>
- ISO certification often requires third party calibration of instruments:
  - Service provided by TA Instruments service representative using certified materials
  - Certificate of Calibration issued showing traceability of calibration to a national laboratory

# Verifying Heat Flow & Temperature

- Run Indium as a sample
- Analyze melt and record melt onset and heat of fusion
- Compare to known values
  - Melting of In 156.598°C
  - Heat of Fusion 28.71J/g
- Always verify first before you decide to recalibrate

# Calibration & Verification of Heat Flow & Temperature, All DSC's

The screenshot displays the TA Instruments TRIOS software interface for DSC calibration and verification. The main window is titled "Calibration Experiment Setup" and is divided into several sections:

- Calibration Setup:** Includes checkboxes for "Cell Conditioning", "Tzero", "Temperature", "Reversing Heat Capacity", "Baseline Conditioning", "Cell Constant/Temperature" (highlighted with a red circle), and "Direct Heat Capacity".
- Cell Constant/Temperature:** A sub-section containing a table for calibration experiments and verification criteria.
- Calibration Experiments Table:**

Premelt	Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input checked="" type="checkbox"/>	Indium	156.598	131.59	171.59	1	4.120	51.600
- Verification Experiments Table:**

Premelt	Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input type="checkbox"/>	Indium	156.598	131.59	171.59	1	4.120	51.600
- Verification Criteria:** A text field showing "Verification Criteria: Temperature ± 0.1 °C Enthalpy ± 2 %" (highlighted with a red circle).
- Buttons:** "Run All", "Queue All", and "Schedule All" buttons are located at the bottom of the setup window.

The right-hand side of the interface features a "Control Panel" showing the current temperature as "Idle 39.99 °C" and a "Signals" table with various parameters and their values. The bottom status bar shows the system is "Ready" and provides information about the instrument and the current time (12:00 PM).

# Calibration of Heat Flow & Temperature, All DSC's

2500 DSC2A-00118 (172.23.165.240) : TA Instruments Trios v4.1.1.33073

Experiment Instrument

File Manager

Results

- indium calibration(2)
  - Isothermal 1.0 min
  - Ramp 5 °C/min to 180.00 °C
- TempCalibration\_DSC2A-00118\_20170428\_1442
  - indium calibration(2)
    - Isothermal 1.0 min
    - Ramp 5 °C/min to 180.00 °C
  - indium verification(1)
    - Isothermal 1.0 min
    - Ramp 5 °C/min to 180.00 °C

Control Panel

Idle 40.00 °C

Signals [16 of 16]

Signal	Value	Units
Method Time	0.00 min	
Remaining Segment Time	0.00 min	
Remaining Time	0.00 min	
Cell Purge	50.00 mL/min	
Temperature	40.00 °C	
Flange Temperature	-78.52 °C	
Heat Flow	3.068 µW	
Set Point Temperature	40.00 °C	
Power Delivered	36.3640 W	
Base Purge	292.61 mL/min	
Tzero Temperature Unfit	40.00 °C	
Heat Capacity	0.00000 J/°C	
Reference Junction Temperature	46.63 °C	
Heater Temp	40.27 °C	

General

Temperature 40.00 °C

Flange Temperature -78.52 °C

Cooler Selection RCS 90

Standby 40.00 °C

RCS 90 On

Gas 1 Nitrogen: 50.00 mL/min

Base Purge 292.61 mL/min

Gas 1: Nitrogen

Flow Rate 50 mL/min

General

Auto Sampler

Ready

Run Status: Idle End Status: NotRun Cortex GDM FEP FCM Auto Sampler 3:07:30 PM

training 2500 DSC2A-00118 (1... TA Instrument Explor... cal - Microsoft Powe...

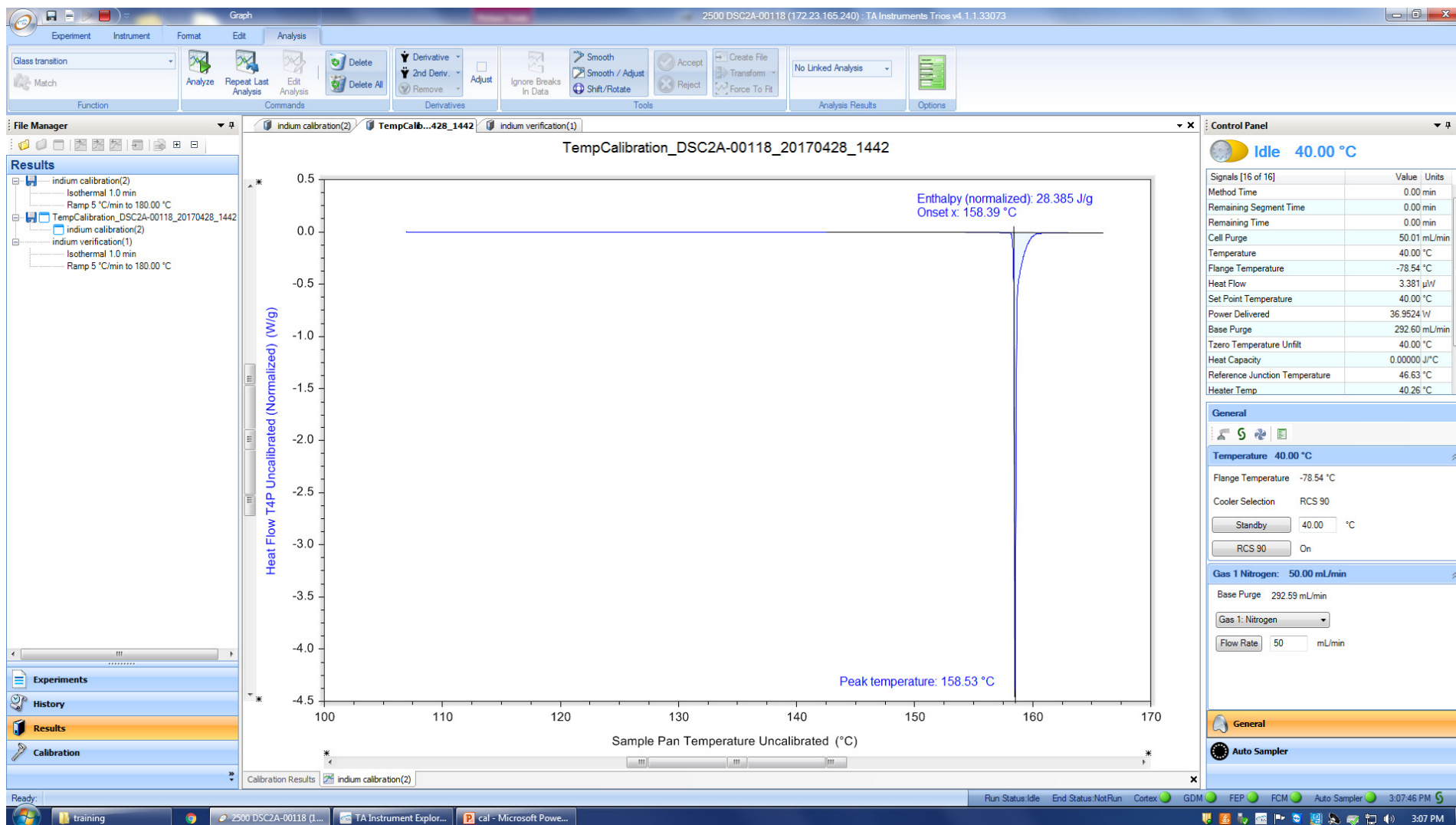
3:07 PM

Note that calibration result is automatically saved to the equipment

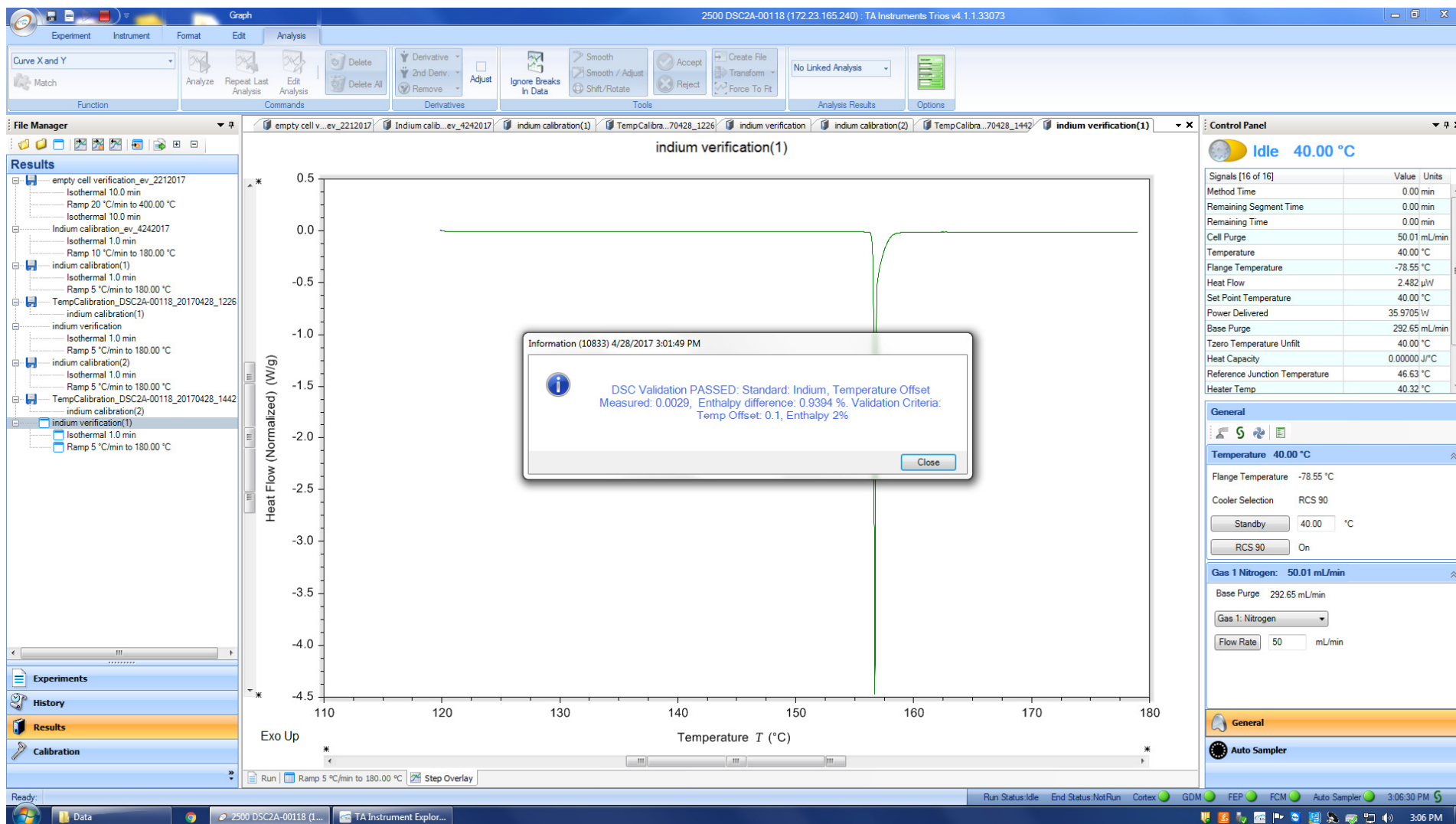
To see the analyzed result



# Calibration of Heat Flow & Temperature, All DSC's



# Verification of Heat Flow & Temperature, All DSC's



# Verification of Heat Flow & Temperature, All DSC's

The screenshot displays the TA Instruments software interface. The main plot shows Heat Flow (Normalized) (W/g) on the y-axis (ranging from -4.5 to 0.5) versus Temperature T (°C) on the x-axis (ranging from 110 to 180). A sharp endothermic peak is visible at approximately 158°C. The plot is titled "indium verification(1)".

Two red text annotations are overlaid on the plot area:

- For the verification run you cannot see the analysis, have to analyze yourself
- You can also see the validation info from the log, click on information and search for validation

The log table at the bottom shows the following data:

Code	Log Time	Description	Machine ID	User ID
10833	4/28/2017 3:01:48.989	DSC Validation PASSED. Standard: Indium, Temperature Offset Measured: 0.0029, Enthalpy diff.: 2500 DSC2A-001.. NTSERV1labbe		
10833	4/28/2017 12:45:39.470	DSC Validation FAILED Standard: Indium, Temperature Offset Measured: 5.9789, Enthalpy Diffe.: 2500 DSC2A-001.. NTSERV1labbe		

The log table also includes a search bar with "validation" entered and a red box around the "Information" icon in the first row.

# Heat Flow & Temperature Calibration: Temperature Calibration with **Multiple** Points

Calibration Experiment Setup

Cell Conditioning     Tzero     Temperature     Reversing Heat Capacity  
 Baseline Conditioning     Cell Constant/Temperature     Direct Heat Capacity

Cell Constant/Temperature    Temperature

Pan Number	Sample Mass	Pan Mass	Pan Type
Reference: 46	0.000 mg	50.910 mg	Tzero Aluminum

Operator: ev  
Project: calibration

Insert Isothermal: 1.0 min  
 Ramp: 10 °C/min

Premelt Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input checked="" type="checkbox"/> Tin	231.93	181.93	261.93	2	3.720	51.110
<input checked="" type="checkbox"/> Indium	156.598	106.59	186.59	1	3.870	50.710

Calibration     Perform Verification after Calibration  
 Verification     Perform Calibration if Verification fails  
 Verification Criteria: Temperature ± 0.2 °C

Premelt Reference Material	Melt Temp	Lower Limit	Upper Limit	Pan Number	Sample Mass	Pan Mass
<input checked="" type="checkbox"/> Tin	231.93	181.93	261.93	2	3.720	51.110
<input checked="" type="checkbox"/> Indium	156.598	106.59	186.59	1	3.870	50.710

File Path: C:\ProgramData\TA Instruments\TRIOS\Data

Control Panel: Idle 40.00 °C

Signals [16 of 16]    Value    Units  
 Method Time: 0.00 min  
 Remaining Segment Time: 0.00 min  
 Remaining Time: 0.00 min  
 Cell Purge: 49.99 mL/min  
 Temperature: 40.00 °C  
 Flange Temperature: -82.21 °C  
 Heat Flow: -4.445 μW  
 Set Point Temperature: 40.00 °C  
 Power Delivered: 37.8499 W  
 Base Purge: 292.28 mL/min  
 Tzero Temperature Unfit: 40.00 °C  
 Heat Capacity: 0.00000 J/°C  
 Reference Junction Temperature: 46.65 °C  
 Heater Temp: 40.17 °C

General  
 Temperature: 40.00 °C  
 Flange Temperature: -82.21 °C  
 Cooler Selection: RCS 90  
 Standby: 40.00 °C  
 RCS 90: On  
 Gas 1 Nitrogen: 49.99 mL/min  
 Base Purge: 292.28 mL/min  
 Gas 1: Nitrogen  
 Flow Rate: 50 mL/min

Experiments  
 History  
 Results  
 Calibration

Run Status: Idle    End Status: NotRun    Cortex    GDM    FEP    FCM    Auto Sampler    11:58:00 AM

Have to run indium twice, once for the cell constant and once for the multi temperature cal !

2 point temp cal table will be created: indium + tin

# Reloading Older Cell Constant/Temperature Calibration

Most to the left = latest calibration  
Click on other bullets to reload older calibrations (e.g. with different pan type)

Value	Units
Idle	40.00 °C
Time	0.00 min
ng Segment Time	0.00 min
ng Time	0.00 min
ge	50.02 mL/min
ture	40.00 °C
emperature	-25.14 °C
w	20.293 µW
t Temperature	40.00 °C
elivered	21.2065 W
rge	292.80 mL/min
emperature Unfilt	40.00 °C
capacity	0.00000 J/°C
se Junction Temperature	46.63 °C
emp	40.60 °C

T Melt (Ref)	T Melt (meas)
156.598	158.393



# Discovery DSC 2<sup>nd</sup> Generation: Multiple Calibration Sets

The screenshot displays the software interface for the Discovery DSC 2<sup>nd</sup> Generation. The main window is titled "Experiments" and shows a "Procedure" configuration page. The "Test" is set to "Custom". Under the "Segments" section, there is a table with columns "No." and "Description". Below this, the "Advanced" section contains several options:

- Use Standby Temperature (Load Window)
- Discard pan in waste bin at end of test (End of Test)
- Use Standby Temperature (End of Test)
- Use default calibrations (Selected Calibrations)

The "Selected Calibrations" option is circled in red. A red text overlay points to this option, stating: "calibration selected on the calibration data page is used".

On the right side, the "Control Panel" shows the system is in "Idle" mode at 39.99 °C. A table of signals is visible:

Signals [15 of 15]	Value	Units
Remaining Time	0.00 min	
Cell Purge	50.00 mL/min	
Temperature	39.99 °C	
Flange Temperature	-79.35 °C	
Heat Flow	79.350 µW	
Set Point Temperat	40.00 °C	
Power Delivered	38.6373 W	
Base Purge	297.13 mL/min	
Tzero Temperature	40.00 °C	
Reference Junction	46.61 °C	
Heater Temp	40.35 °C	
Power Request on I	55.5931 W	
Delta Tzero Unfilt	0.01 °C	

The bottom of the interface shows a taskbar with several open applications, including "Skype for Business", "TA Instrument Explor...", "2500 DSC2A-00118 (1...", "0550-0039 (172.23.16...", "5500-0056 (172.23.16...", and "DSC2S T1 cal - Micro...". The system tray shows the time as 12:04 PM.

# Discovery DSC: Multiple Calibration Sets

The screenshot displays the TA Instruments TRIOS software interface. The main window is titled "Calibration" and shows a list of calibration sets. The "T1 Calibration" set is selected, indicated by a blue dot. A dialog box titled "TA Instruments TRIOS" is open, showing the "Add New Calibration Set" form. The "Calibration Set Name" field contains "T1 first ca" and the "Calibration Data" field contains a list of calibration data points. A red circle highlights the "Save Selected as a Set" button in the top right corner of the main window, with a red arrow pointing to the dialog box. A red text overlay at the bottom of the dialog box reads: "The calibration selected by the blue dot is now saved as a set, you can do that for multiple calibration sets".

Calibration Data

Displayed Calibration Set: No Set Selected

Save Selected as a Set: Save

**T1 Calibration 8/10/2017 3:50 PM (Applied)**

Name: T1CalibrationDSC2A-00118\_20170810\_1550.tri  
Description: From 29.771 °C to 199.859 °C at 20.000 °C/min  
Temperature Range: Lower Temperature: 29.771 °C, Upper Temperature: 199.859 °C, Heating Rate: 20.000 °C/min  
Slope and Offsets: Delta T Slope: -0.002 μV/°C, Delta T Offset: -0.353 μV, Initial Temperature: 29.771 °C  
Quadratic Coefficients: Quadratic A: -0.048, Quadratic B: 0.000, Quadratic C: 0.000

Instrument Parameters  
Source Files

**Cell Constant Calibration 8/10/2017 4:47 PM (Not Applied)**

Name: TempCalibrationDSC2A-00118\_20170810\_1647.tri  
Heating Rate: 10 °C/min  
KCell Information: Standard: Indium, Applied KCell: 0.959  
Instrument Parameters  
Source Files

**Temperature Calibration 8/10/2017 4:47 PM (Not Applied)**

Name: TempCalibrationDSC2A-00118\_20170810\_1647.tri  
Heating Rate: 10 °C/min  
Temperature Offset: TMelt (Ref), TMelt (meas)

TA Instruments TRIOS

Add New Calibration Set

Calibration Set Name: T1 first ca

Calibration Data: Izero 1/1/0001 12:00:00 AM, T1 Heat Flow 8/10/2017 3:50:08 PM, Temperature 8/10/2017 4:47:03 PM, K Cell 8/10/2017 4:47:04 PM

Save Cancel

General

Temperature: 39.99 °C

Flange Temperature: 41.52 °C  
Cooler Selection: RCS 90  
Standby: 40.00 °C  
RCS 90: On

Gas 1 Nitrogen: 50.00 mL/min  
Base Purge: 297.21 mL/min  
Gas 1: Nitrogen  
Flow Rate: 50.00 mL/min

Auto Sampler

# Discovery DSC: Multiple Calibration Sets

The screenshot displays the TA Instruments Discovery DSC software interface. The main window is titled "Experiments" and shows a "Procedure" configuration screen. In the "Advanced" section, the "Selected Calibrations" dropdown menu is highlighted with a red circle and contains the option "T1 first cal".

Calibration set includes: baseline, cell constant temperature, heat capacity. It does not include Tzero.

Redoing Tzero calibration removes previous calibration sets !

You can now choose for every experiment which calibration set to use

The right-hand side of the interface shows a "Control Panel" with various parameters and a "Signals" table.

Signals [15 of 15]	Value	Units
Method Time	0.00	min
Remaining Segme	0.00	min
Remaining Time	0.00	min
Cell Purge	49.99	mL/min
Temperature	39.99	°C
Flange Temperatu	41.52	°C
Heat Flow	94.658	µW
Set Point Tempera	40.00	°C
Power Delivered	1.1270	W
Base Purge	297.22	mL/min
Tzero Temperature	40.00	°C
Reference Junction	46.64	°C
Heater Temp	41.86	°C
Power Request on	1.2172	W

General  
Temperature: 39.99 °C  
Flange Temperature: 41.52 °C  
Cooler Selection: RCS 90  
Standby: 40.00 °C  
RCS 90: On  
Gas 1 Nitrogen: 49.99 mL/min  
Base Purge: 297.22 mL/min  
Gas 1: Nitrogen  
Flow Rate: 50.00 mL/min



# Optimization of Experimental Conditions



# Instrument Preparation

- **Cooling Accessories**
  - They should be operating and equilibrated prior to runs (so do not turn off between runs !)
- **Warm-up Time/Environment**
  - Electronics should be given at least one hour to stabilize for important samples if the instrument has been turned off (typically leave instrument always on)
  - Electronics are effected by ambient temperature. Avoid areas such as hoods or near an air conditioner
- **Purge Gas**
  - Make sure cylinder is not empty or moisture condensation will occur if cooler is on or still cold

# Understanding Your Sample

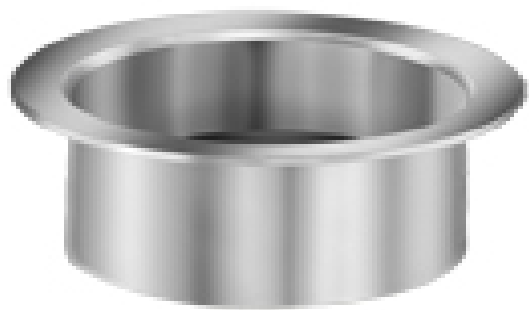
- Find out all you can about the sample
  - Form
  - Are there any volatiles (TGA) ?
  - Decomposition temp (TGA)
  - What transitions to expect ? In what temperature range ?
- This will help you choose
  - Max Temperature Range
  - Heating Rate
  - Sample Pans
  - Sample Size

# Effective Sample Preparation

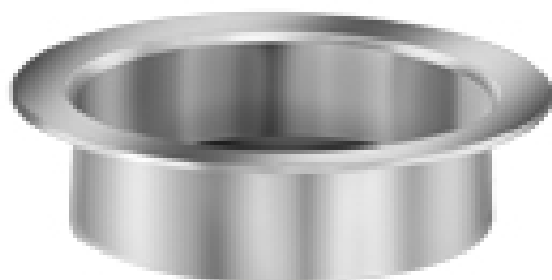
- **Good sample preparation relies on two aspects:**
  - Choose the proper pan
  - Load your sample effectively
- **DSC Pan types:**
  - **Standard Pans:** Appropriate for most solid samples (films, powders, polymer granule, etc)
  - **Hermetic Pans:** Designed primarily for samples which may evolve a volatile during heating
    - Can be used for small quantities of liquids as well
  - **Specialty Pans**
    - **High Volume Pans:** Used for larger quantities of liquids and solutions and for temperatures above boiling point

# TA Instruments Tzero Pans

**Tzero Pan**

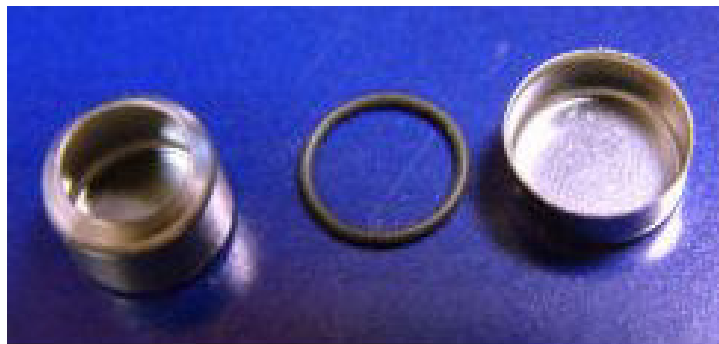


**Tzero Low-Mass Pan**



- The Tzero pan has been engineered to have a perfectly flat bottom and not to deform during crimping. This ensures the optimal contact between pan and sensor, minimizing the contact resistance and improving resolution.
- The Tzero Pan can be configured for crimped or hermetic use.
- The Tzero Low-Mass Pan is designed for the highest sensitivity when sample mass is limited (crimped use only).

# TA Instruments High Volume Pans



- The high volume pan is designed for measuring diluted solutions (larger volume improves sensitivity)
- The high volume pan allows measuring liquids above their boiling point (aluminum hermetic pan is limited to a pressure of 2-3 bar)

Pressure capability	3.8 MPa gauge (550 psig)
Temperature limit for aqueous solutions	250°C
Pan Volume Material	100 $\mu$ L (max) 302 SST
O-ring material	Viton

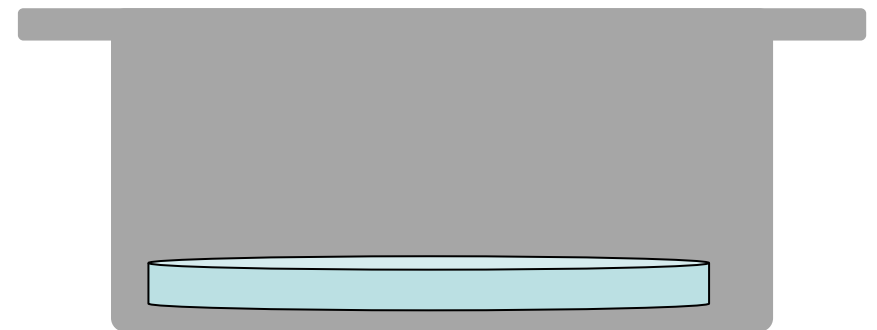
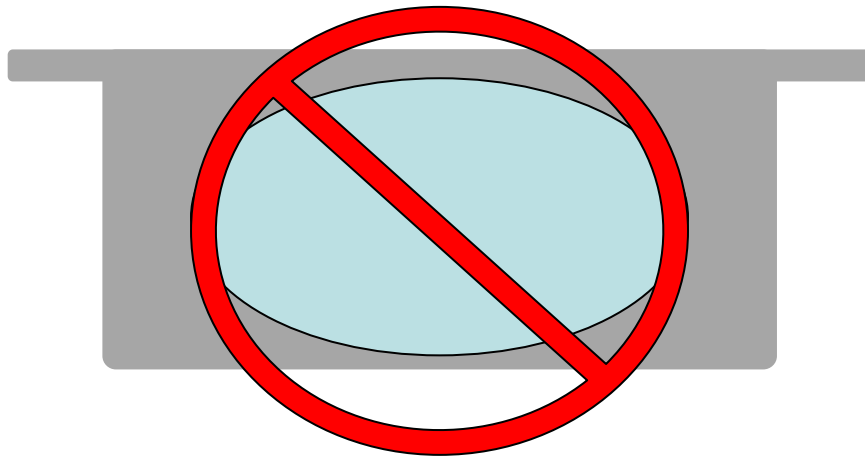
# Sample Shape: Solids

- Keep sample thin
- Cover as much as the bottom of pan as possible



# Sample Shape: Solids

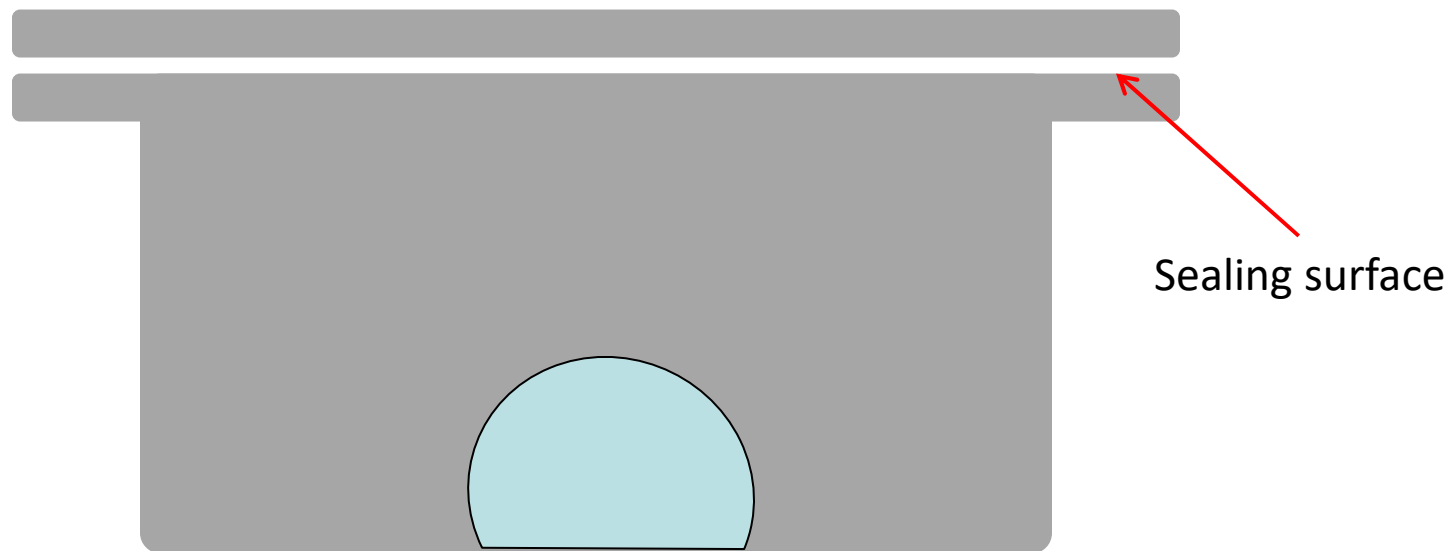
- Cut sample to make thin, don't crush (can introduce thermal history)
- If sample is a pellet, cut a cross section





# Sample Shape: Liquids

- Use a pipette to place a single drop of liquid carefully in the middle of the pan
- Do not allow the liquid to wet the sealing surface of the pan !
- Use a hermetic lid



# DSC: Sample Size

- In a DSC experiment, sample size is a trade-off between sensitivity and resolution
  - Larger samples = more sensitivity
  - Smaller samples = more resolution
- In general, it is best to use the smallest sample which does not compromise the required sensitivity
  - melting of metal or chemical < 5mg
  - melting or Tg of polymer : about 10 mg
  - composites or blends >10 mg
- For accurate enthalpy measurements the sample weight must be determined with an accuracy of 0.01 mg

# Heating/Cooling Rate

- High rates increase sensitivity

$$dQ/dt = C_p \cdot dT/dt + f(t, T)$$

- Low rates increase resolution by providing more time at any temperature
- **Good starting point is 10 or 20°C/min**

# Keeping the DSC Cell Clean

One of the first steps to ensuring good data is to keep the DSC cell clean

How do DSC cells get dirty ?

- Decomposing samples during DSC runs
  - Run TGA to determine the decomposition temperature, stay below that temperature !  
Just because the instrument has a max temperature range of 550°C (w/ RCS) doesn't mean you need to heat every sample to 550°C !
- Samples spilling out of the pan
  - Use lids
  - Use hermetic pans if necessary
- Transfer from bottom of pan to sensor
  - Make sure bottom of pans stay clean

# Help !?

- Software :
  - Case sensitive help, help topics, manual
- E-training (basic)
  - [www.tainstruments.com](http://www.tainstruments.com)
- TATechTips & Webinars
  - [www.tainstruments.com](http://www.tainstruments.com)
- Application library
  - [www.tainstruments.com](http://www.tainstruments.com)

Help and data analysis from your laptop ? Simply install the S/W from CD or download it !

www.tainstruments.com

The screenshot shows the TA Instruments website's Support page. The navigation bar includes 'Products', 'About TA Instruments', 'Videos', 'Support', 'Training', 'News & Events', 'Careers', and 'Contact'. A search bar is also present. The 'Support' menu is expanded, showing four main categories: 'Service Support', 'Application Support', 'Software Downloads & Support' (highlighted with a red circle), and 'Support Plans'. The 'Software Downloads & Support' category includes links for 'Downloads', 'Instruments sorted by software', 'Software Sorted by Instruments', 'Report a Bug', and 'Request a Feature'. The 'Support Plans' category includes 'Lifetime Support Plan', 'Premium Support Plan', 'Plus Support Plan', 'Basic Support Plan', 'Performance Maintenance Visit (PMV)', and 'Academic Support Plan'. A 'Quick Links' sidebar on the left lists 'Applications Support', 'Service Support', 'Support Plans', 'Software Downloads & Support', and 'Parts and Accessories'. A banner image below the menu features the words 'SECURE', 'HELP', 'SUPPORT', and 'ADVICE' over a keyboard background. At the bottom, four service tiles are displayed: 'Service Plans' (with a technician image), 'Service Support' (with a network diagram), 'Application Support' (with a customer service team), and 'Software Downloads' (with a download icon).

Service Support	Application Support	Software Downloads & Support	Support Plans
Service Support Helpline	Applications Support Helpline	Downloads	Lifetime Support Plan
Instrument Relocation	Tech Tips	Instruments sorted by software	Premium Support Plan
Installation Requirements & Repairs	Applications Notes Library	Software Sorted by Instruments	Plus Support Plan
The IQ/OQ Product Offering	Training	Report a Bug	Basic Support Plan
Calibration with Certified Standards		Request a Feature	Performance Maintenance Visit (PMV)
Safety Data Sheets			Academic Support Plan
Supported Instruments			

# Help on Desktop

## TA Instruments

### Thermal Discovery Series Manuals

To view the desired manual using Acrobat Reader, click the name in the list below:

*TA Manual Supplement*  
(Contains important information applicable to all manuals.)

#### Instrument Documentation

Discovery DSC Getting Started Guide

Discovery DSC Getting Started Guide for DSC 25, DSC 250, DSC 2500

Discovery TGA Getting Started Guide

Discovery TGA Getting Started Guide for TGA 55, TGA 550, TGA 5500 - **NEW!**

#### Accessory Documentation

DSC High Pressure Capsule Kit Getting Started Guide

DSC High Volume Pan Kit Getting Started Guide

Liquid Nitrogen Pump System (LN2P) Getting Started Guide

Mass Spectrometer (MS) Getting Started Guide

Photocalorimeter Accessory (PCA) Getting Started Guide

Refrigerated Cooling System (RCS) Getting Started Guide

TGA HiRes™ Option Operator's Guide

#### Software Documentation

What's New in TRIOS Software

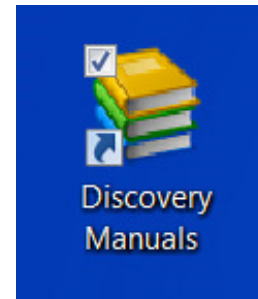
Installing TRIOS Software

#### Miscellaneous Documentation

Thermal Discovery Series Installation Requirements



Issued September 2016



# Trios

Offline-Discovery : TA Instruments Trios v...

Experiment Instrument

Offline Setup Procedure Start Stop - Test Log View Help

Help Topics, searchable & step per step description

File Manager

Experiments

Incomplete

Running Queue

Design View

Create new experiment

Run Design View Schedule

Sample

Sample Name

Sample Pan Number Size Pan Mass

Reference

Operator

You can install an offline version of Trios on your laptop





## Quick Start Guides

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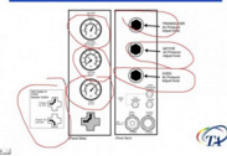
Tech Tips

## Quick Start Guides e-Training Courses

QuickStart e-Training courses are designed to teach a new user how to set up and run samples on their thermal analyzer or rheometer. These 60-90 minute courses are available pre-recorded and can be taken 24/7 (24 hours a day, 7 days a week). These pre-recorded courses are available to all users at no charge. Typically these courses should be attended shortly after installation.

### View Quick Start Guide Videos

#### Primary Things



ARES Rheometer Quickstart

#### Agenda

- Opening the file in the software
- Loading the sample
- Setting up the experiment
- Running the experiment
- Saving the data

DMA Q800 Quickstart Course – Instrument and Experimental Setup

#### Preparing & Loading the Sample



TMA Quickstart

#### Agenda

- Opening the file in the software
- Loading the sample
- Setting up the experiment
- Running the experiment
- Saving the data

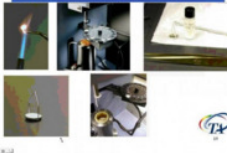
DMA Q800 – Analysis Quickstart

#### Preparing & Loading the Sample



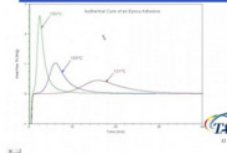
Q600 SDT Quickstart

#### Preparing & Loading the Sample

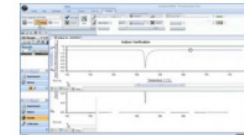


TGA Quickstart

#### Kinetic Heat Flow



MDSC Quickstart



Discovery DSC – TRIOS Data Analysis



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or on You Tube !

### Tech Tips

TA Instruments is the world leader in Thermal Analysis, Rheology, and Microcalorimetry, and we just keep getting better. On this page, you can see just some of the many products we offer to help provide you with the tools to succeed. Product videos, reviews, and side by side comparisons show we're willing to bare all. Our transparency comes from a long history of success, a willingness to better ourselves, and the desire to help you be a leader in your field.

- Baseline verification
- Powder preparation kit
- Integrating baselines
- .... and much more

### Sort Tech Tips Videos by category

View all

Microcalorimetry (TechTips)

Rheology (TechTips)

Tech tips

Thermal Applications (TechTips)



Measuring Thin Film w/  
Optical Dilatometers – TA  
TechTip



TA TechTips Year In  
Review!



Water Sorption Experiments  
w/ VTI-SA+ – TA TechTips



Running A Water Sorption  
Experiment- TA TechTips



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About TA Instruments Webinars

### Webinars

The top researchers in chemistry, materials science, and life sciences rely on TA Instruments systems for their cutting-edge research. In our TA Instruments Webinar Series we are pleased to bring this work directly to you. Learn from the experts in the field about how thermal analysis, rheology, and microcalorimetry are used to solve today's most challenging problems. These free webinars are presented live with interactive Q&A, then archived for subsequent viewing and reference. See the list of previous webinars below, and check back often for the latest information.

### Webinar Archives



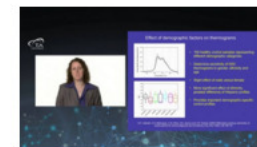
In Search of Calorimetric Answers to Events In Self Assemblies



Extensional Rheology in Polymer Processing



Uses of Isothermal Microcalorimetry in Urology



Plasma Protein Calorimetry For Clinical Diagnostics



## Support

### Quick Links

Applications Support

Service Support

Support Plans

Software Downloads & Support

Parts and Accessories

#### Service Support

- Service Support Helpline
- Instrument Relocation
- Installation
- Requirements & Repairs
- The IQ/OQ Product Offering
- Calibration with Certified Standards
- Safety Data Sheets
- Supported Instruments

#### Application Support

- Applications Support Helpline
- Tech Tips
- Applications Notes Library**
- Training

#### Software Downloads & Support

- Downloads
- Instruments sorted by software
- Software Sorted by Instruments
- Report a Bug
- Request a Feature

#### Support Plans

- Lifetime Support Plan
- Premium Support Plan
- Plus Support Plan
- Basic Support Plan
- Performance Maintenance Visit (PMV)
- Academic Support Plan

Home / Support



Service Plans



Service Support



Application Support



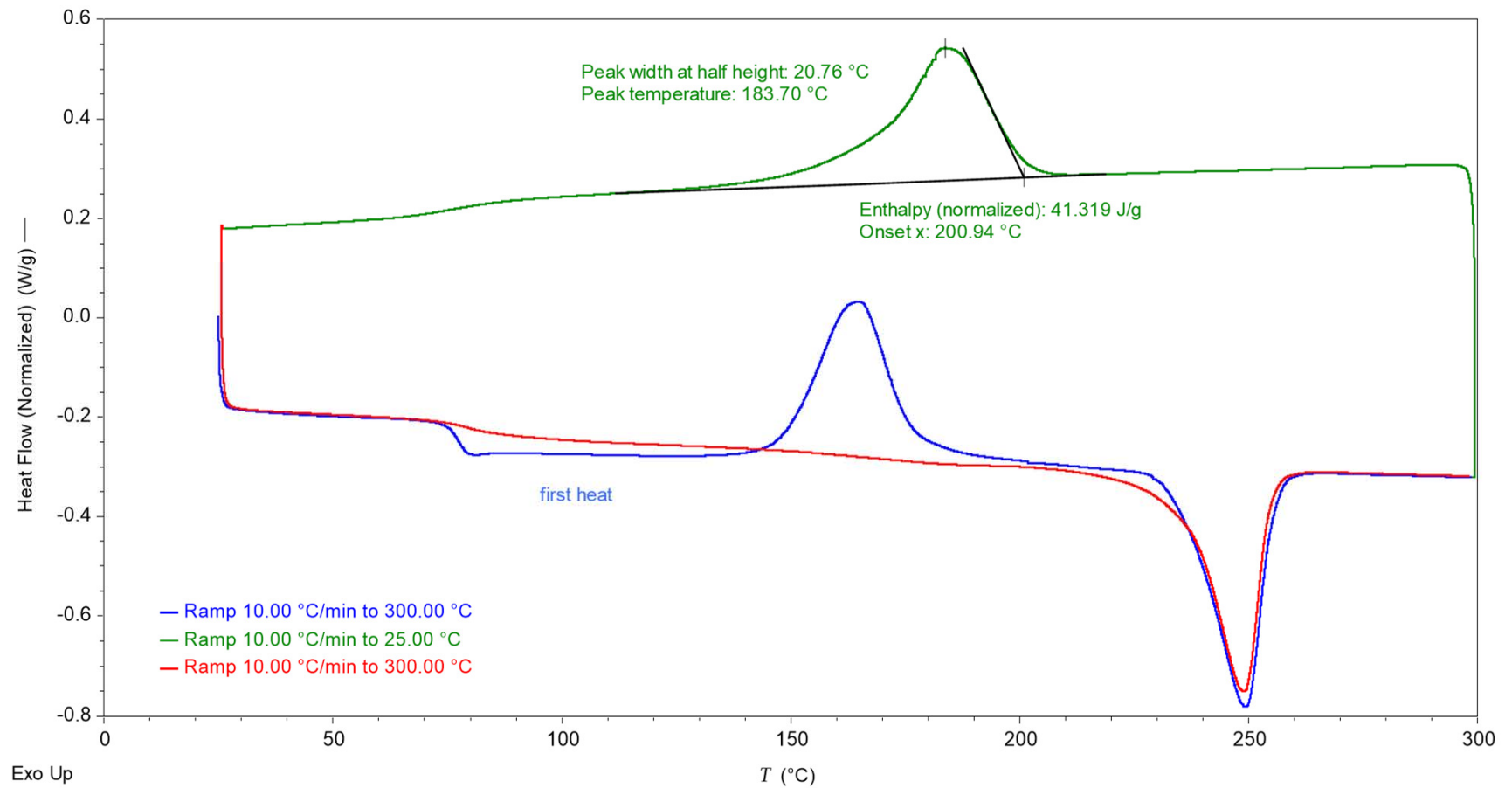
Software Downloads

# Trios Software



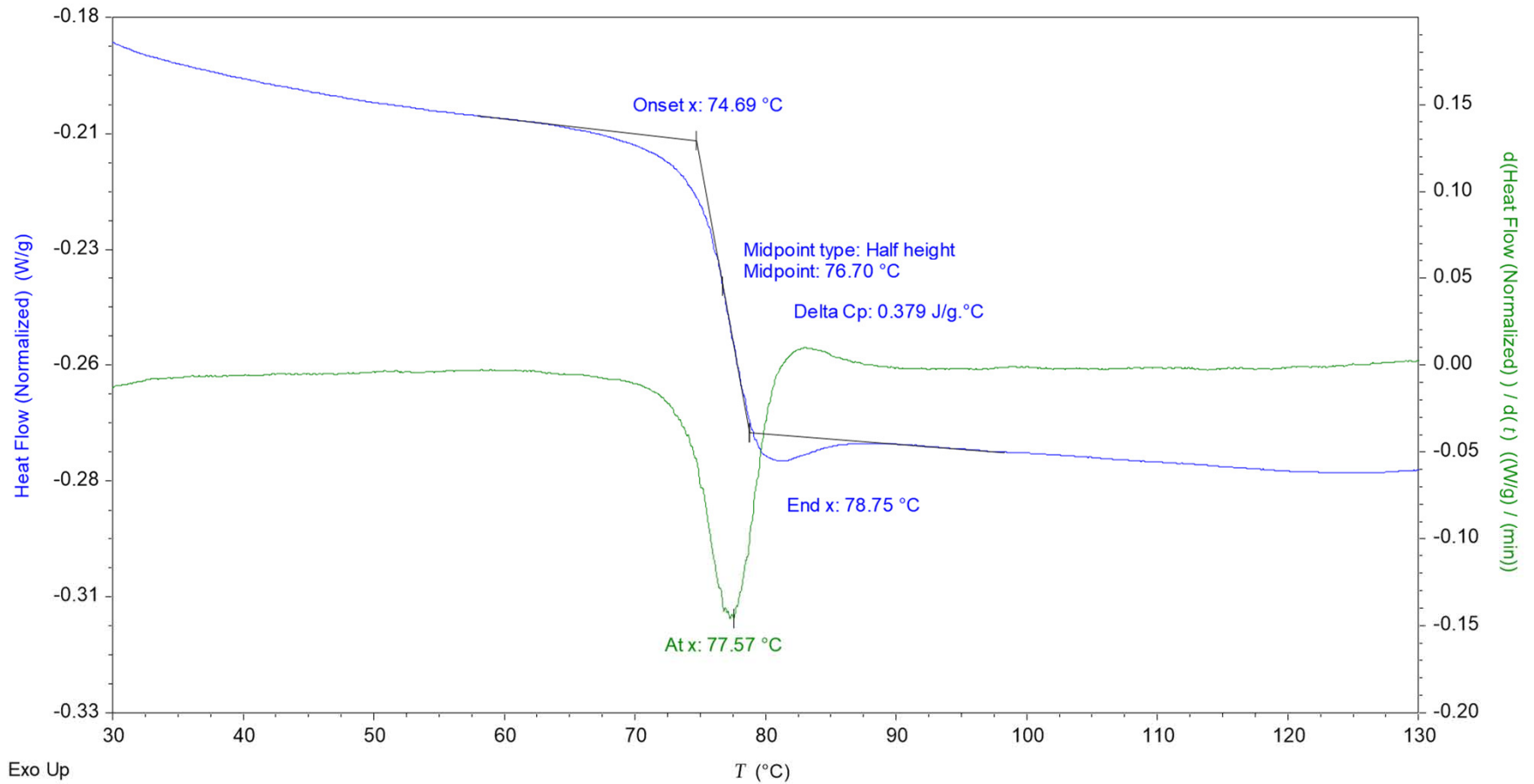
# PET (Heat-Cool-Heat)

C:/ProgramData/TAInstruments/Trios/Data/ExampleFiles/Thermal



# PET (Heat-Cool-Heat) 1<sup>st</sup> Heat

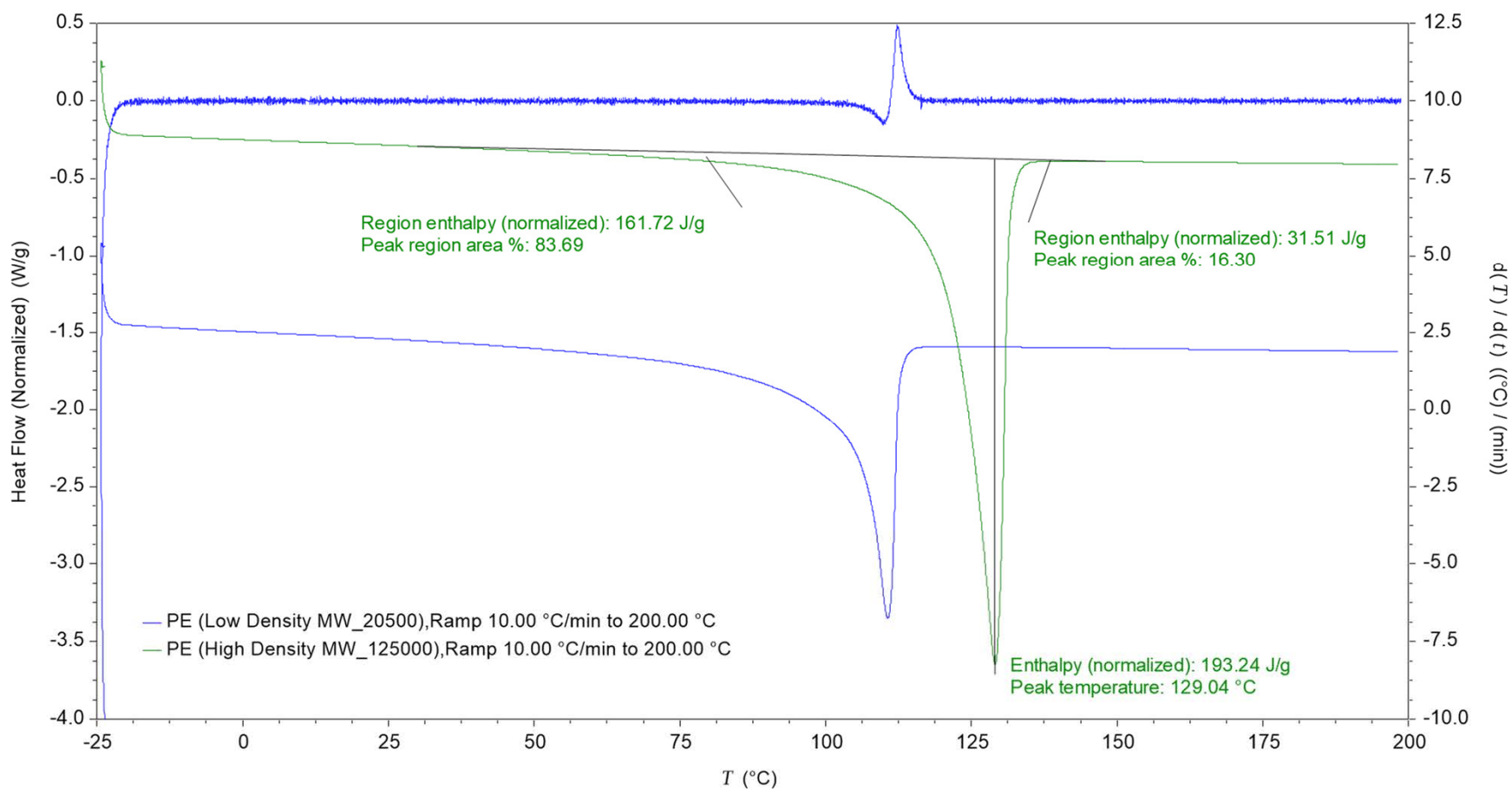
C:/ProgramData/TAInstruments/Trios/Data/ExampleFiles/Thermal





# PE (Low & High Density) 2<sup>nd</sup> Heat

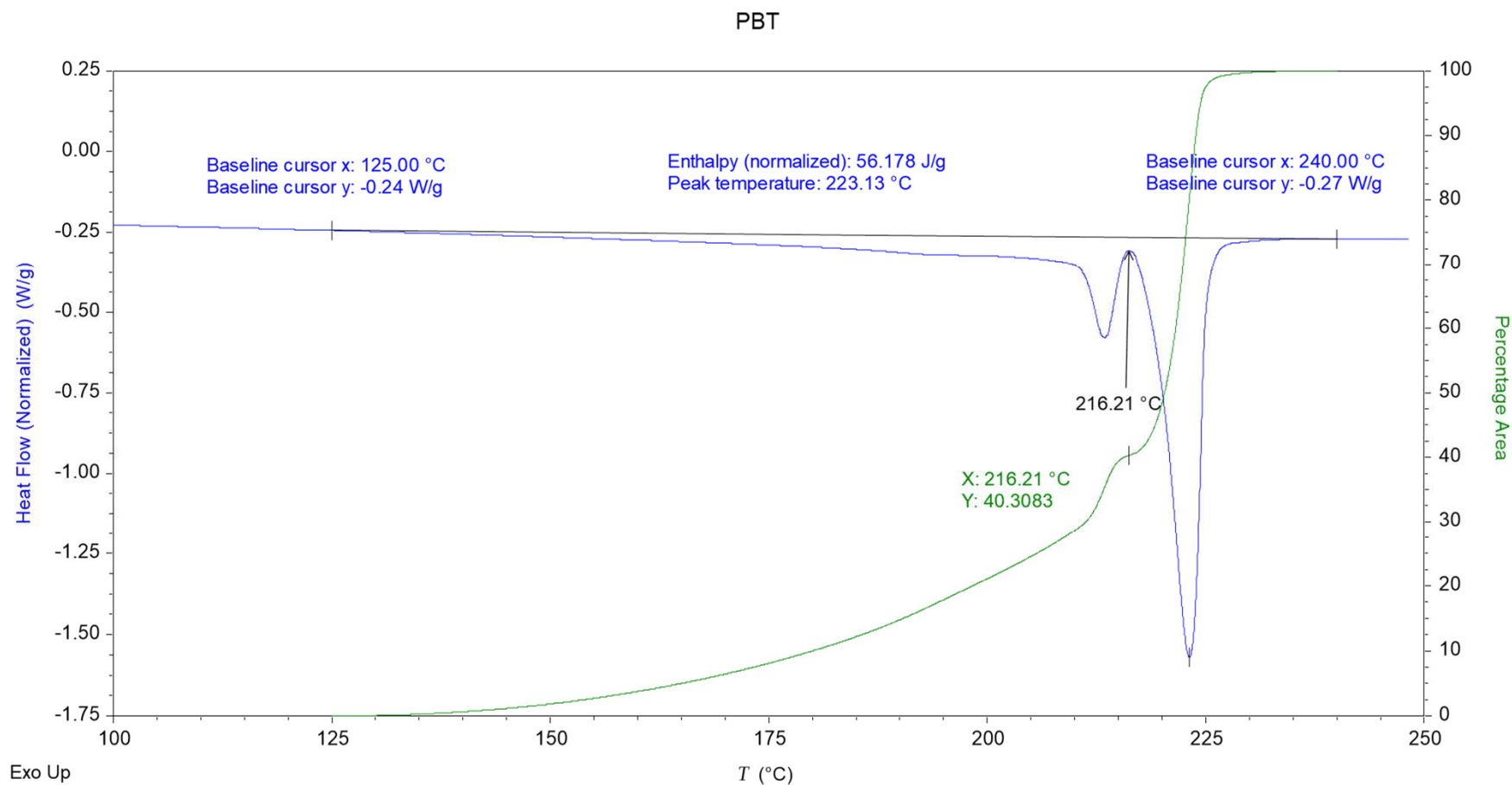
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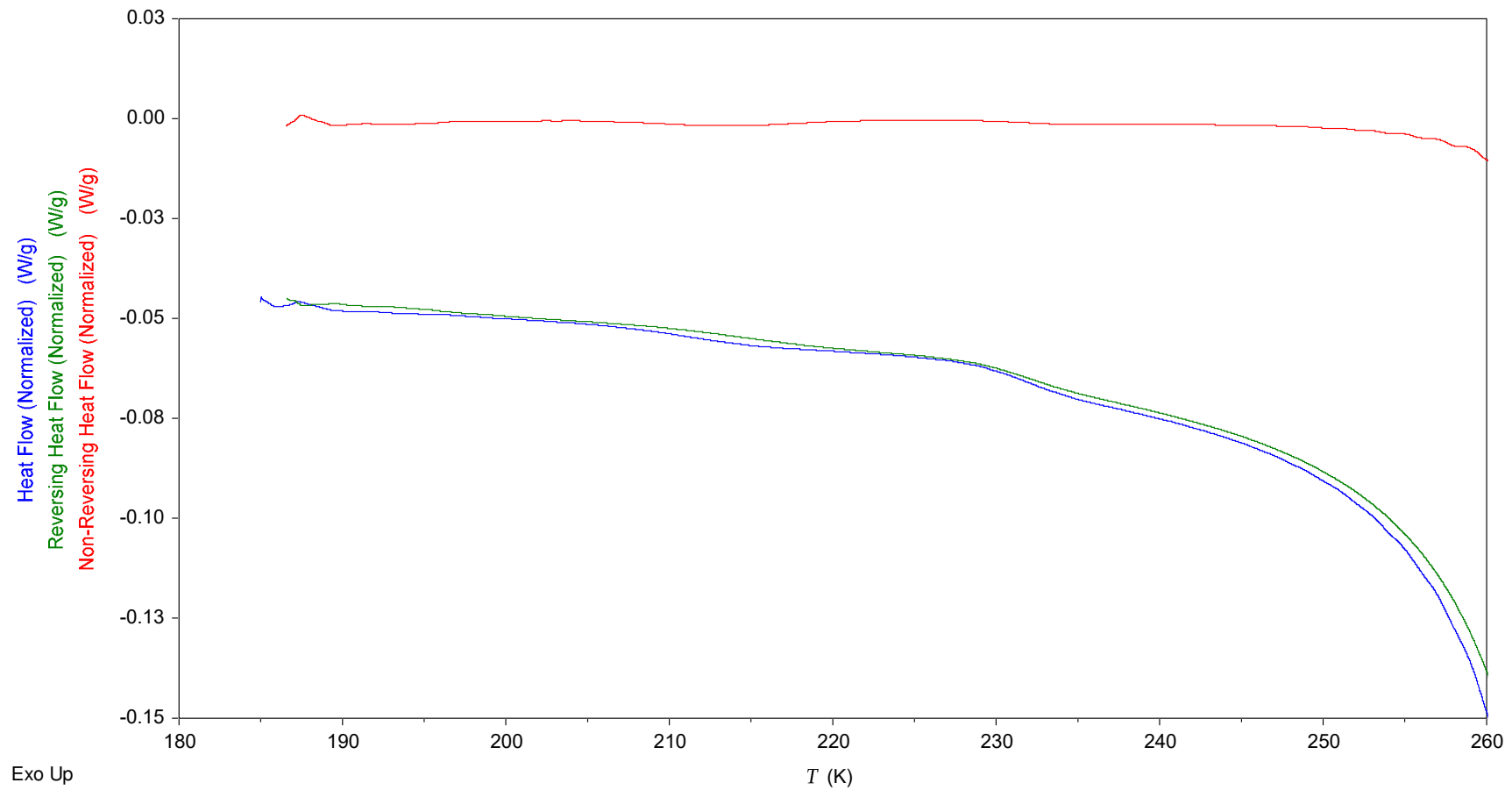


# PBT 2<sup>nd</sup> Heat

C:/ProgramData/TAInstruments/Trios/Data/ExampleFiles/Thermal

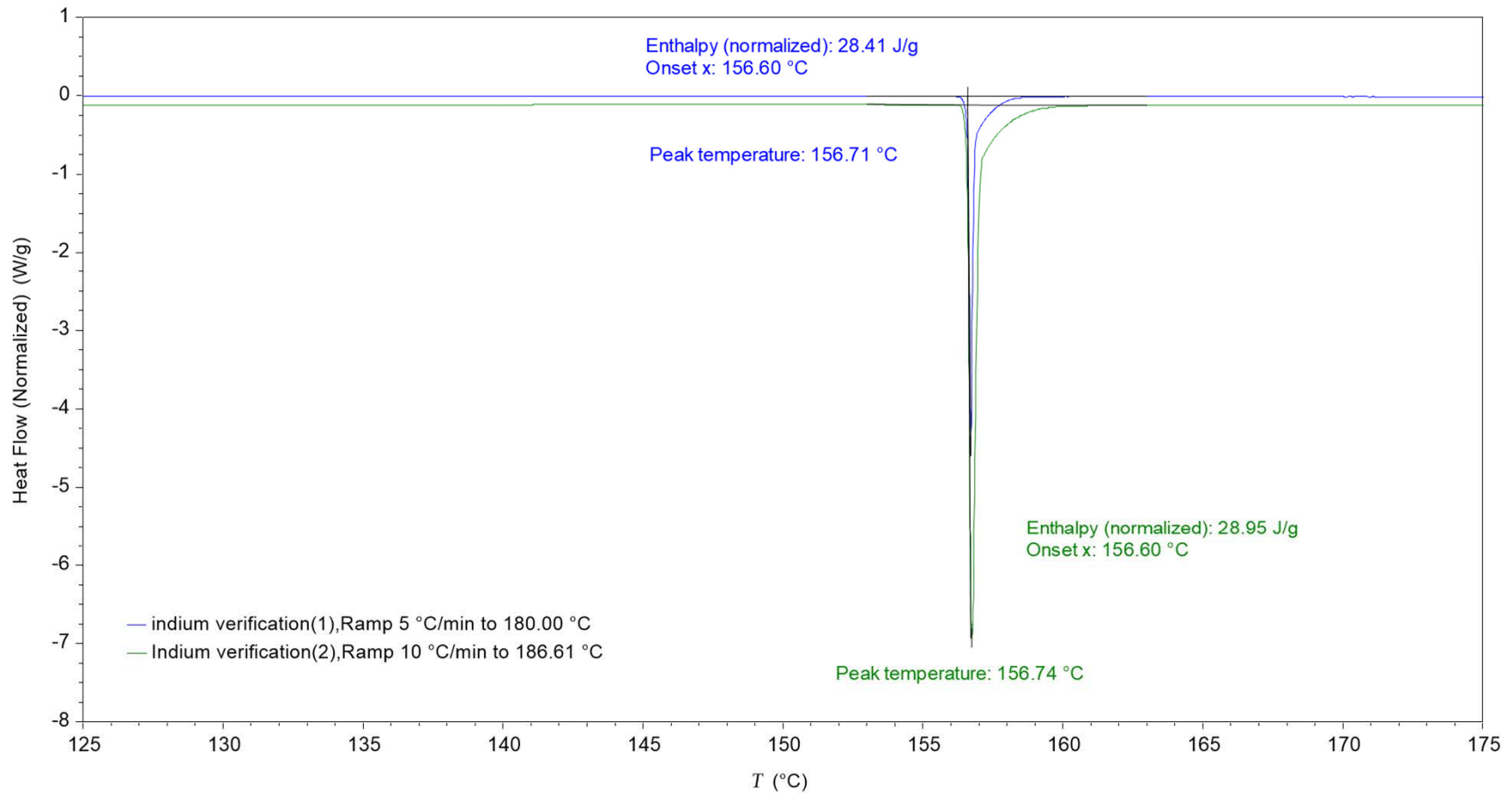


# Liquid Sample MDSC 1st Heat



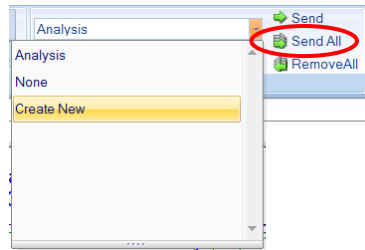
Note: temperature in K

# Indium Verification: Influence of Heating Rate



Use the **analysis library** to integrate both between 153 to 163°C

# Two Indium Verification Test: Statistics



Offline-DSC2500 : TA Instruments Trios v4.1.1.33073

File Manager: indium verification(1), indium verification(2), Overlay, Analysis

Selected step: indium

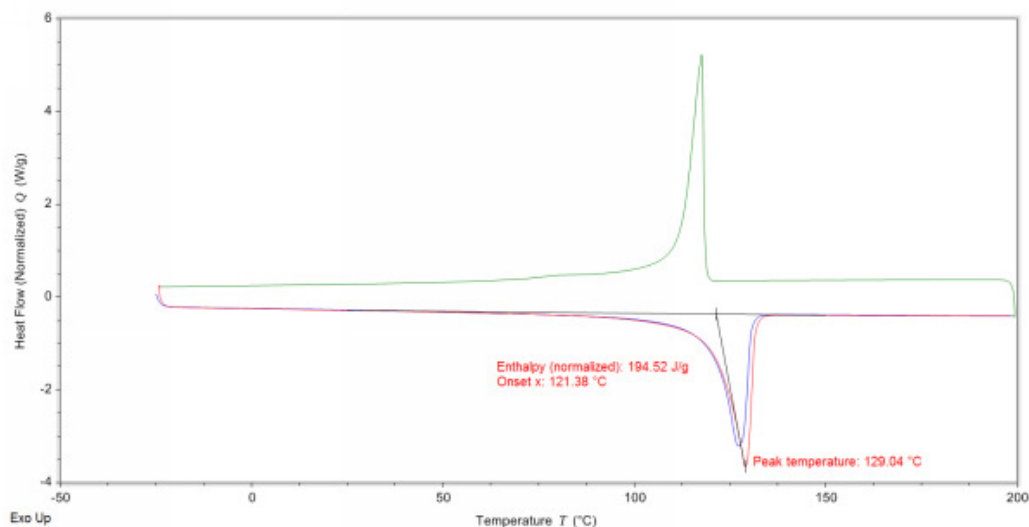
	A	B	C	D	E	F	G	H
	File Name	Step Name	Name	Run date	Enthalpy (normalized) J/g	Onset temperature °C	Peak temperature °C	Row
1	indium verification(1)	Ramp 5 °C/min to 180	Indium	4/28/2017 2:42:50 PM	28.410	156.60	156.71	1
2	Indium verification(2)	Ramp 10 °C/min to 18	Indium Cert verificatio	3/14/2017 1:13:40 PM	28.949	156.60	156.74	2

Statistics

Mean = 28.679	Mean = 156.60
SD = 0.38125	SD = 0.00
Rel. SD = 0.013294	Rel. SD = 0.00
Var. = 145.35	Var. = 0.00

indium indium

# Reporting



PE (High Density MW\_125000)  
3.8 mg

Segment 1	Equilibrate -25.00 °C
Segment 2	Ramp 10.00 °C/min to 200.00 °C
Segment 3	Mark End of Cycle
Segment 4	Ramp 10.00 °C/min to -25.00 °C
Segment 5	Mark End of Cycle
Segment 6	Ramp 10.00 °C/min to 200.00 °C
Segment 7	Mark End of Cycle

Peak Integration (enthalpy)

Enthalpy (normalized)	Peak temperature	Onset x
194.52 J/g	129.04 °C	121.376 °C

<https://triosvideos.s3.amazonaws.com/Report%20View.mp4>

# Thank You !

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