DSC and MDSC Online Training

Part 2: Applications

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1

Heat Flow Signals

$$\frac{dQ}{dt} = Cp\,\frac{dT}{dt} + f(T,\ t)$$

Total Heat Flow

- All Transitions

Reversing Heat Flow

- Heat Capacity
- Glass Transition
- Melting

Non-Reversing Heat Flow

- Enthalpic Recovery
- Evaporation
- Crystallization
- Thermoset Cure
- Denaturation
- Decomposition
- Some Melting
- Chemical Reactions

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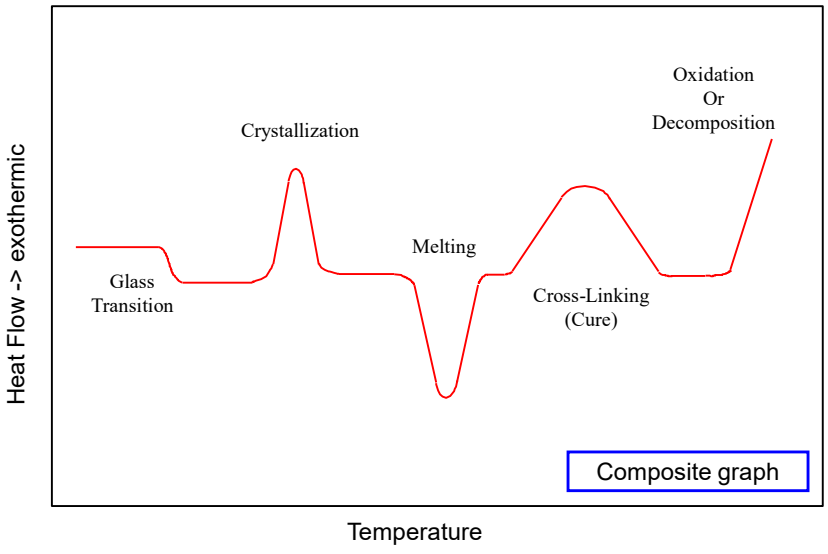
Applications Agenda

- Oxidation Induction Time
- Heat Capacity (Cp)
- The Glass Transition Temperature (Tg)
- Melting and Crystallization Analysis
- Thermosets: Curing and Crosslinking
- Pharmaceuticals

3

Typical DSC Transitions



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Oxidative Stability (OIT) Method

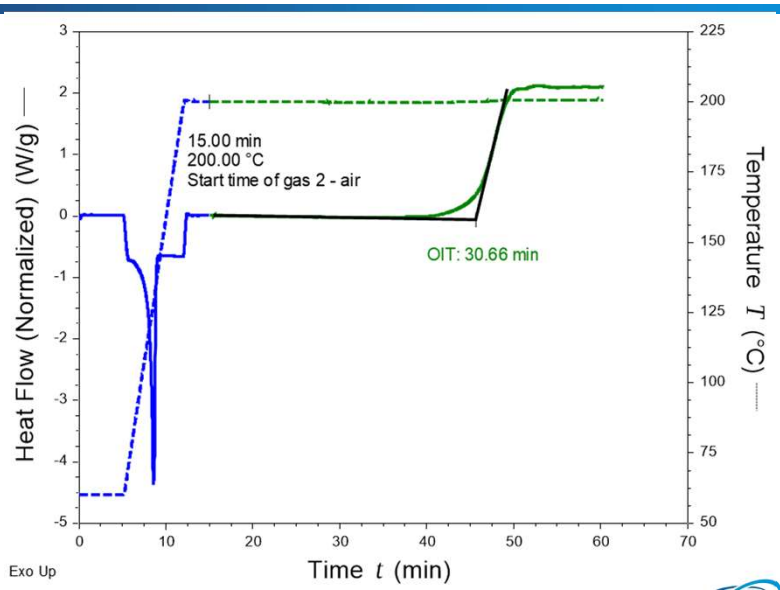
The OIT test characterizes the thermo-oxidative stability of a sample by using DSC

ASTM D3895-19 – OIT for polyolefins
ASTM D1858-08 – OIT for hydrocarbons

An OIT Method (polyolefin)

1) Isothermal for 5.00 minutes in Nitrogen
2) Ramp 20°C/min. to 200°C
3) Isothermal for 5.00 minutes
4) Select gas: 2 (air or oxygen)
5) Isothermal for 100.00 minutes (hold to a time where the exotherm reaches a peak to get accurate OIT)

Oxidative Induction Time of Polyolefin Film



Heat Capacity (Cp)

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About Heat Capacity

- Heat capacity is the amount of heat required to raise or lower the temperature of a material by 1°C.
- Specific heat capacity (Cp) refers to a specific mass and temperature change for the material (J/g°C).
- Heat capacity is directly related to molecular mobility
- Cp increases as molecular mobility increases
  - Higher Cp = More Mobility
  - Lower Cp = Less Mobility
- Quantitative indicator of structure and stability

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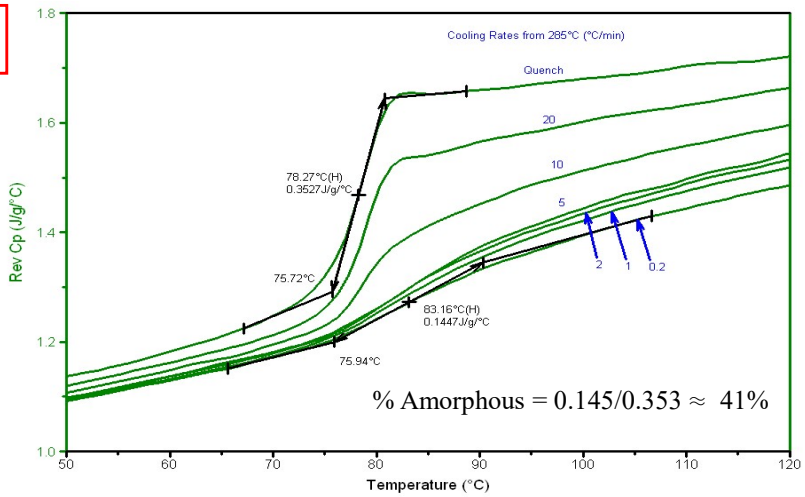
Change in Cp at Tg is a Measure of Amorphous Structure

PET
9.43mg



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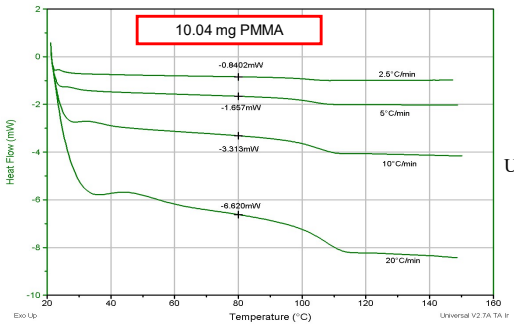
Calculating heat capacity from heat flow data



$$dH/dt = Cp\, dT/dt + f(T, t)$$

$$\text{Uncal. Cp}\left(\frac{J}{g.\,°C}\right) = \frac{\text{Heat flow}\left(\frac{mJ}{sec}\right)}{\text{Heat Rate}\left(\frac{°C}{min}\right) \text{ x wt (mg)}} \text{ x } 60\left(\frac{sec}{min}\right)$$

Heating rate (°C/min)	Heat flow (mW)	Uncalibrated Cp (J/g°C)	Actual Cp = Apparent Cp x K (the heat capacity calibration constant)
2.5	0.8402	2.008	
5	1.657	1.980	
10	3.313	1.980	
20	6.620	1.978	

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Measuring Heat Capacity

- In a DSC experiment, heat capacity is measured as the absolute value of the heat flow, divided by the heating rate, and multiplied by a calibration constant.

$$dH/dt = Cp\, dT/dt$$

Sample Heat Capacity

$$Cp = \left[\frac{dH/dt}{dT/dt} \right] \times K$$

Heat Flow

Heating Rate

Calibration constant

Calculating Heat Capacity

- Depending on the DSC that you have there are three different ways to calculate Cp
  - ASTM E1269 (Three Run Method)
    - Applicable to all DSC's
  - Direct Cp – Single Run Method
    - Applicable to DSC 2500, Discovery DSC, Q2000/1000 only
    - Fastest determination
  - MDSC® - Single Run Method
    - Any DSC w/ MDSC option
    - Most accurate determination

Conventional Method for Cp by DSC (ASTM E1269)

- ASTM E1269: Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry
  - Requires three discreet experiments
    - Baseline
    - Sapphire
    - Sample
  - Method
    1: Equilibrate at XX.00°C
    2: Isothermal for 10.00 min
    3: Ramp 20.00°C/min to XX.00°C
    4: Isothermal for 10.00 min

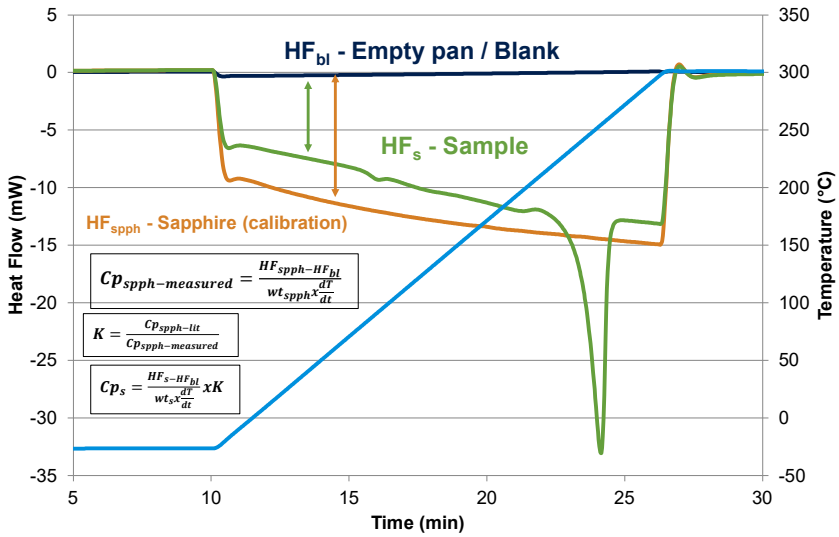
The sample weight must be constant throughout the ramp.

ASTM Method E1269 (Three-Run Method)

- Three experiments are run over a specific temperature range
  - Allow 10 minute isothermal at start and end
  - Use 10-20°C/min heating rate

1. Empty pan run (Baseline run)
   - Match pan/lid weights to ± 0.05 mg
   - Used to establish a reference baseline (absolute heat flow)
2. Sapphire run
   - Used to determine calibration constant
   - Use same weight of pan/lid as above ± 0.05 mg
   - Typical weight is 20 – 25 mg
3. Sample run
   - Typical weight is 10 – 15 mg
   - Use same weight of pan/lid as above ± 0.05 mg

ASTM E1269 "3-Run" Method for Determining Cp



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Direct Cp Measurement

- Available in DSC's with T4P Heat flow mode: DSC 2500, Discovery DSC, Q2000
- Sapphire used as a calibration standard
- Typical Method
  - Heat @10-20°C/min
- Sample Size ~10mg
- For best results – use lowest mass pans possible

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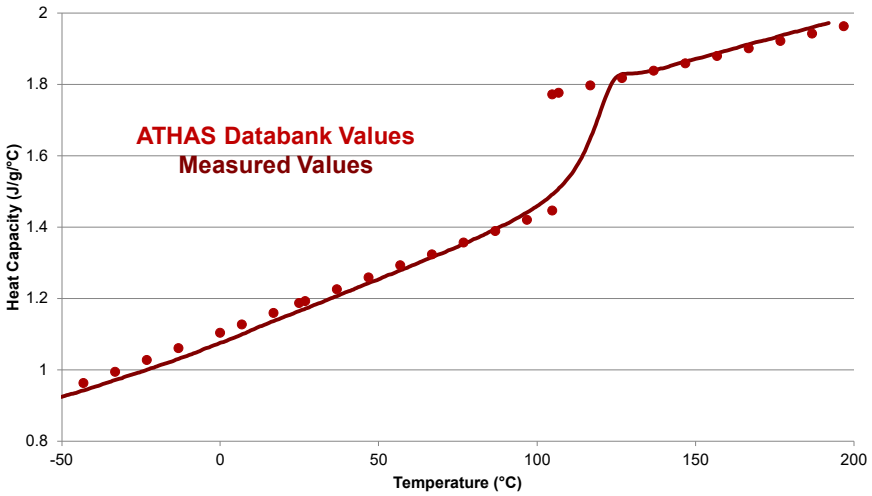
Direct Cp of PMMA – DSC 2500



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Heat Capacity Measurement using Modulated DSC® (MDSC)

- Uses sinusoidal temperature ramp overlaid upon linear ramp
- Separates Heat Capacity and Kinetic transitions
- Increases sensitivity to Heat Capacity changes (e.g. Tg)
- Can determine Cp directly in a single run
- Best available measurement of Heat Capacity
- Accurate & repeatable to within 1-2% or better

Recommended Test Conditions

- Period
  120 seconds
- Heating rate
  2-3°C/min
- Amplitude
  ±1°C

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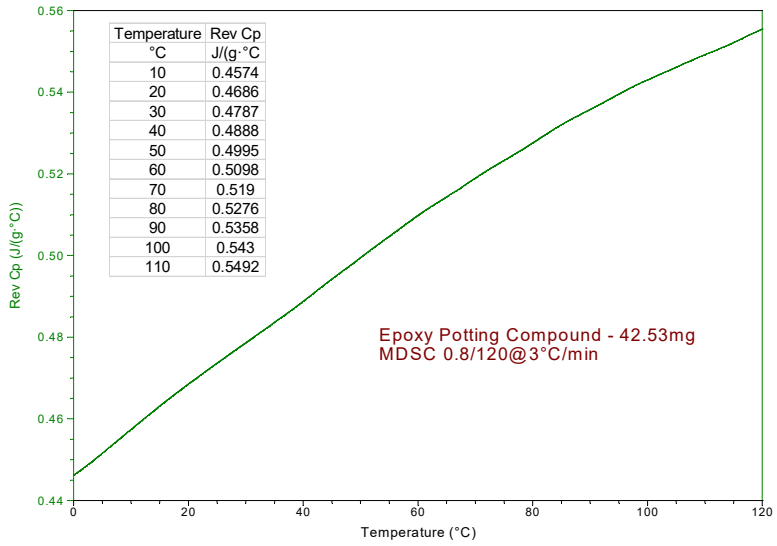
Heat Capacity by MDSC

Temperature	Rev Cp
°C	J/(g·°C
10	0.4574
20	0.4686
30	0.4787
40	0.4888
50	0.4995
60	0.5098
70	0.519
80	0.5276
90	0.5358
100	0.543
110	0.5492



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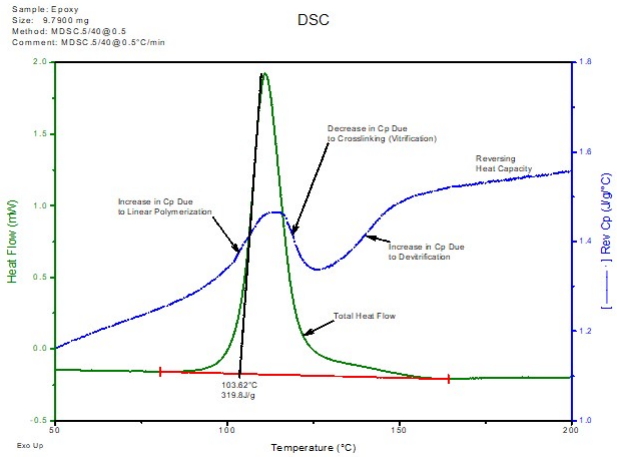
Change in Cp During Cure Is Not Reversible

Sample: Epoxy
Size: 9.79 mg
Method: MDSC at 0.5°C/min



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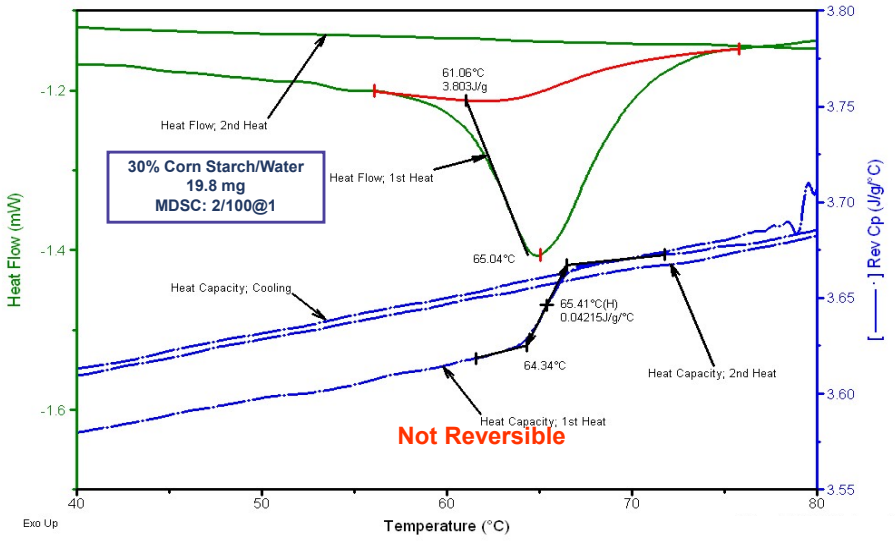
Change in Cp During Starch Gelatinization



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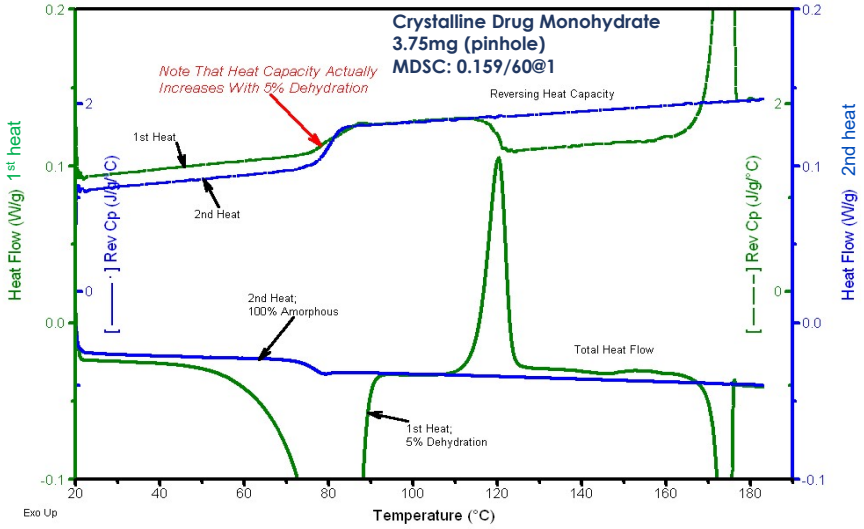
Cp Signals Show Changes in Structure – Pharmaceutical Material



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Quasi-Isothermal Heat Capacity

- MDSC® can also measure heat capacity isothermally (quasi-isothermally) – standard DSC can't do this
- Benefit is to measure structure changes over time

Changes in heat capacity

↓

Changes in mobility

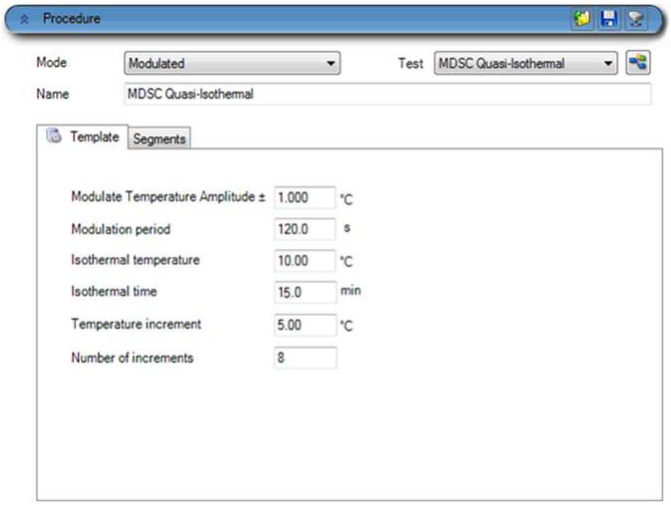
↓

Changes in structure

Quasi Isothermal MDSC for Specific Heat Capacity - Procedure

1. Accurately weigh the mass of sapphire and pan
2. Accurately weigh the mass of a reference pan of the same type
3. Set the following conditions
   - Amplitude: +/- 1 °C
   - Modulation Period: 120 s
   - Isothermal Temperature: ***Set to your desired lower temperature***
   - Isothermal Time: 15 min (this is a good starting point)
   - Temperature Increment: ***Set to the desired temperature interval***
   - Number of Increments: ***Set to the number of intervals you need***

Quasi Isothermal MDSC for Specific Heat Capacity – Procedure in TRIOS



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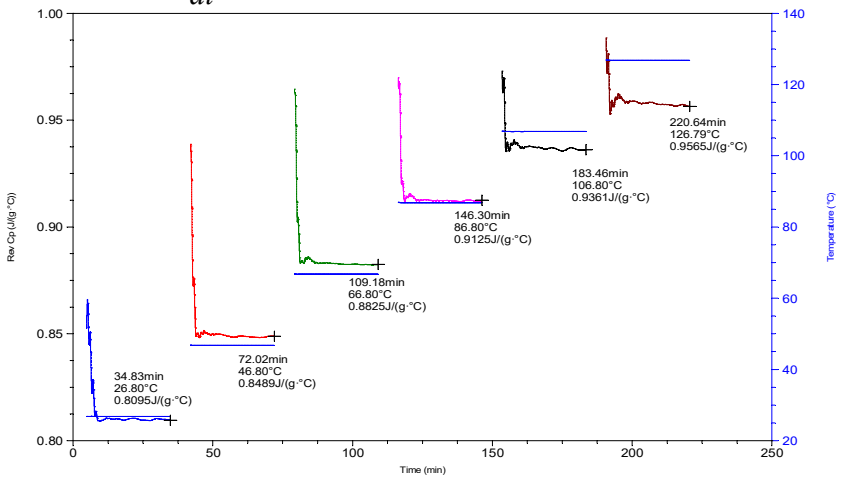
Cp of Sapphire Standard: Determining KCp (reversing)

Sample: Sapphire
Size: 24.6900 mg

$$\frac{dQ}{dt} = Cp\left(\omega A_T \cos \omega t\right)$$



App note TA 432

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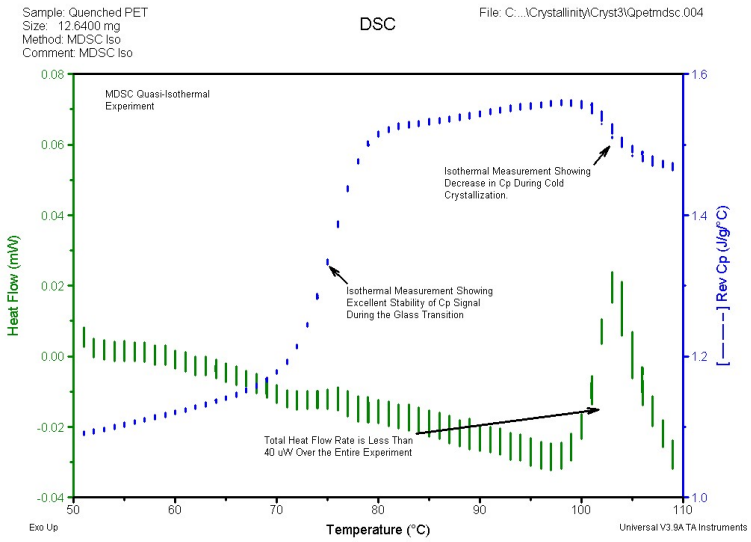
Quasi-Isothermal MDSC®



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KCp (reversing): Temperature Dependence

Temperature (°C)	Experimental Cp (J/(g·°C))	Literature Cp (J/(g·°C))	KCp (Lit. Cp/ Exp. Cp)
26.85	0.8095	0.7788	0.96
46.85	0.8489	0.8188	0.96
66.85	0.8825	0.8548	0.96
86.85	0.9125	0.8871	0.97
106.85	0.9361	0.9161	0.97

$$KCp = \frac{Cp^{Theo.}}{Cp^{Meas.}}$$

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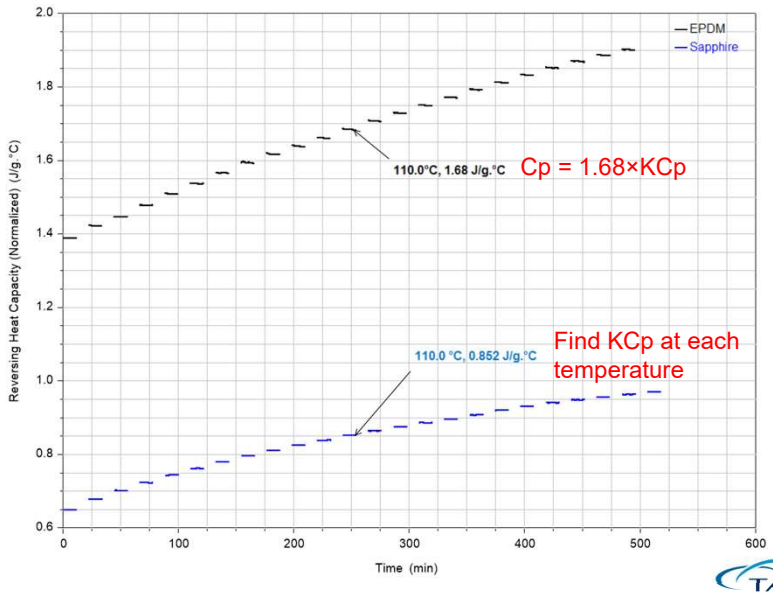
Quasi Isothermal MDSC® for Specific Heat Capacity



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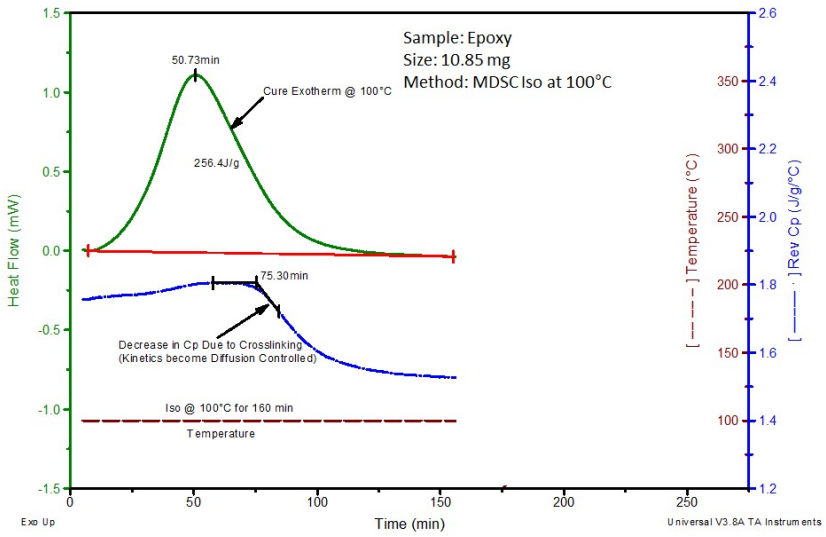
Epoxy Cure with Quasi-Isothermal MDSC®



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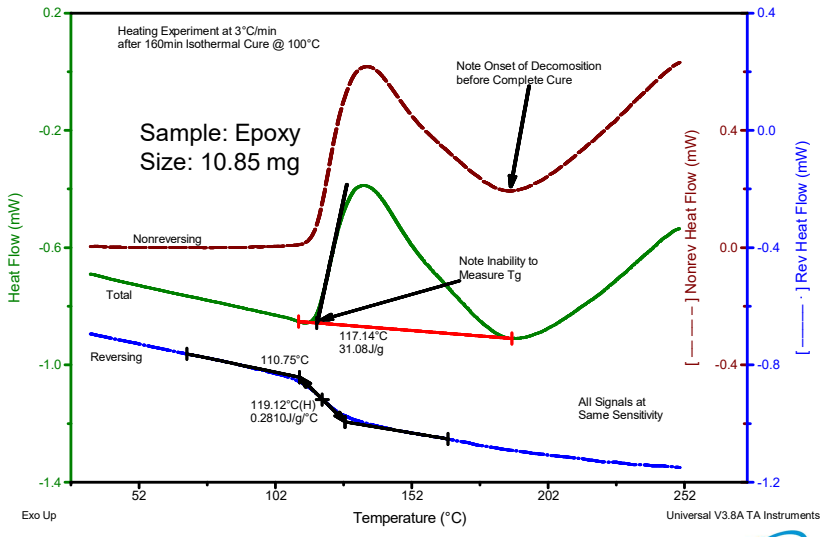
Advantage of MDSC® for Post Cure Scan



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What Affects the Specific Heat Capacity?

- Amorphous Content
- Aging
- Side Chains
- Polymer Backbone
- Copolymer Composition

- Anything that affects the mobility of the molecules, affects the Heat Capacity

- Amorphous *Cp* is greater than Crystalline *Cp*

- Amorphous Content increases Specific Heat Capacity

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The Glass Transition Temperature
(Amorphous Structure)

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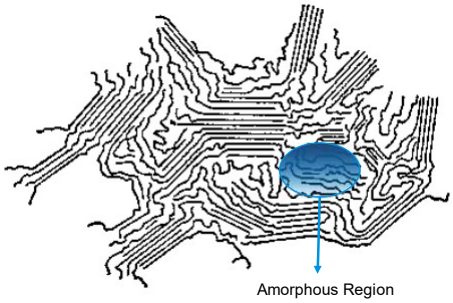
Characterization of Amorphous Structure

- Amorphous Structure
  - Randomly oriented molecules
  - No long-range order
  - Liquids, glassy or rubbery solids
  - Most polymers are either amorphous or semi-crystalline



- Glass Transition (Tg)
  - Due to amorphous (non-crystalline) structure
  - Due to macro-molecular motion (translational); i.e., the entire molecule is free to move relative to adjacent molecules.
  - Extremely important transition because the significant change in molecular mobility at Tg causes significant changes in physical and reactive properties.

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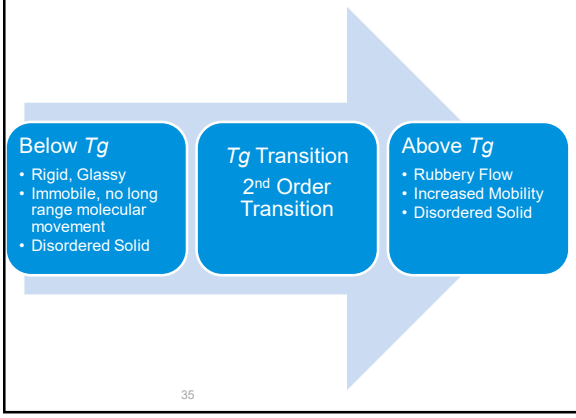
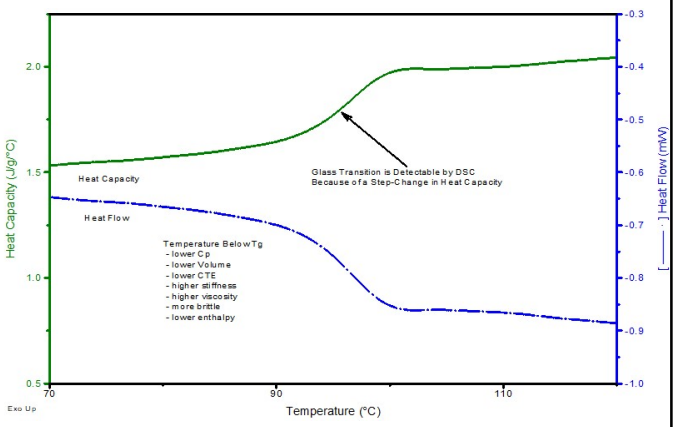
The Glass Transition (*Tg*)

- The glass transition is a change in the free volume and molecular mobility in the amorphous phase of a material that results in a step change in heat capacity.





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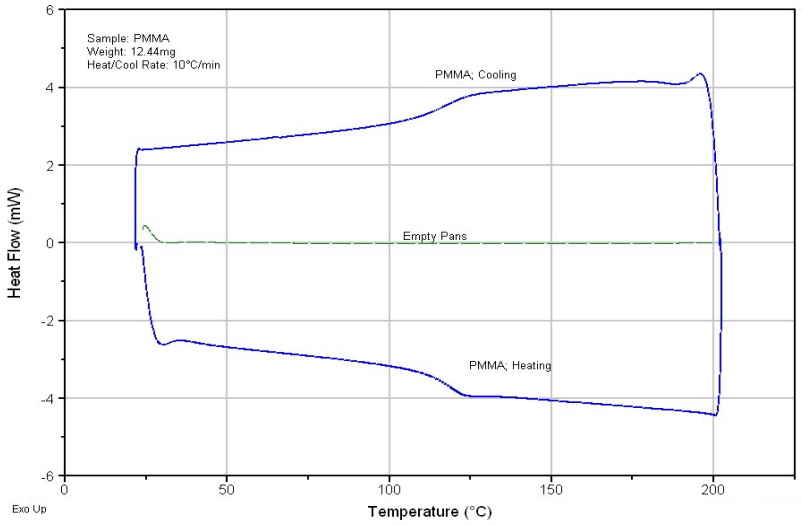
A Glass Transition is Reversible



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Factors affecting Glass Transition and Other Tg measurement Techniques

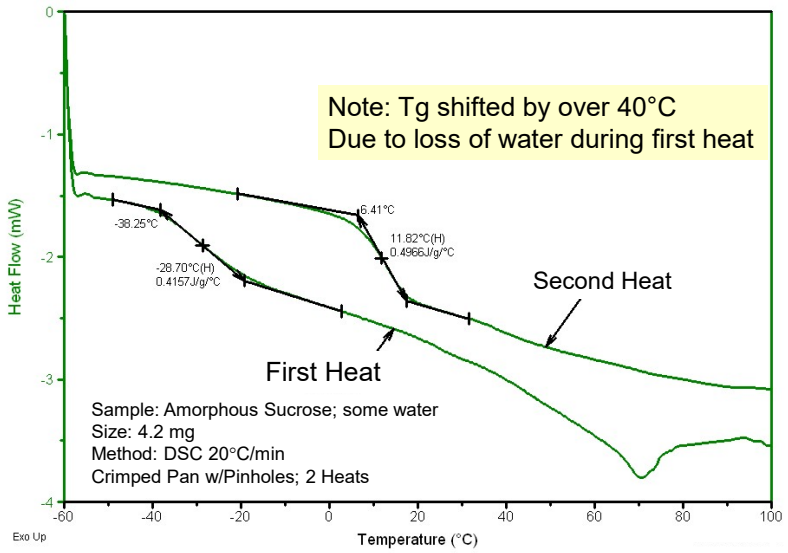
- Heating Rate
- Heating & Cooling
- Aging
- Molecular Weight
- Plasticizer
- Filler
- Crystalline Content
- Copolymers
- Side Chains
- Polymer Backbone
- Hydrogen Bonding

Anything that affects the mobility of the molecules, affects the Heat Capacity and, in turn, the Glass Transition

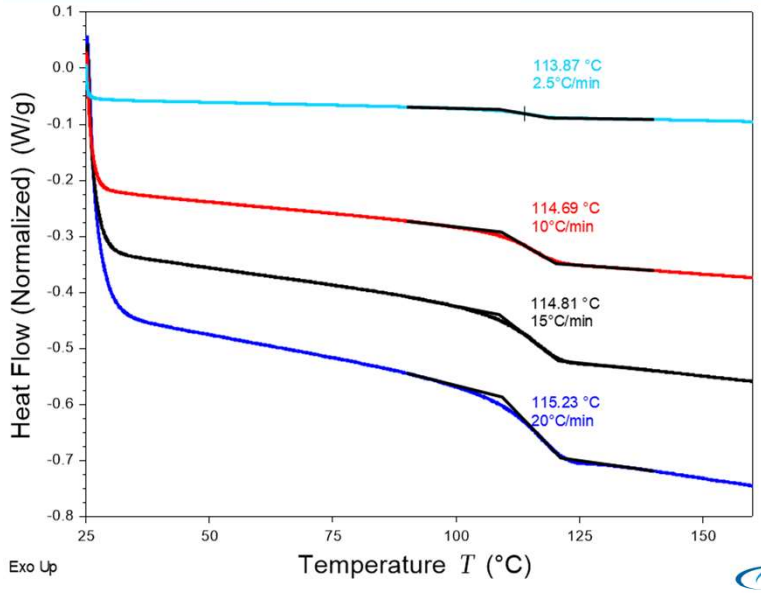
- The *Tg* can also be measured by other techniques apart from the standard DSC
  - Using Modulated DSC (MDSC)
  - Thermomechanical Analysis (TMA)
  - Dynamic Mechanical Analysis (DMA)

- Sensitivity of the technique to detect a glass transition:
  - Standard DSC < MDSC < TMA < DMA

Effect of Moisture on Tg by DSC



10mg PMMA Sample at Different Heating Rates



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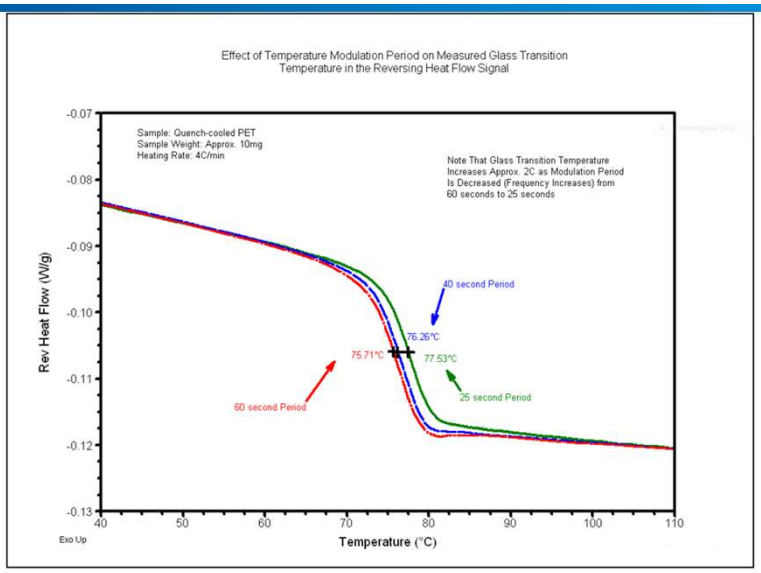
MDSC: Effect of Frequency on *Tg*



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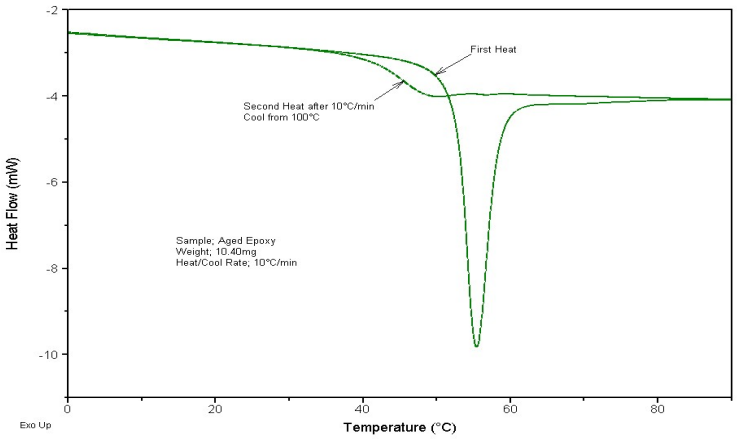
Aged Epoxy: The *Tg* On The First Heat Cycle



Depending on the thermal history of amorphous (glassy) polymers, the glass transition can appear as a simple step in the baseline or one that has a substantial *endothermic peak* that can be misinterpreted as a melting peak.

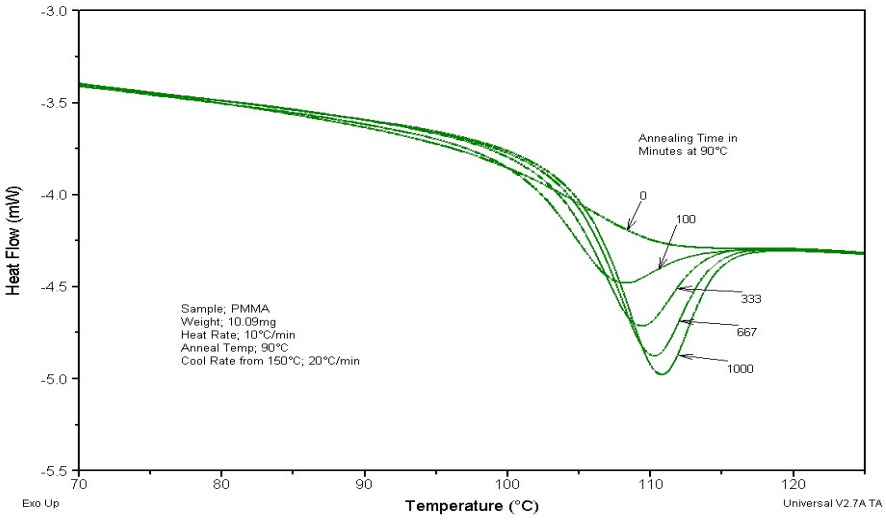
Enthalpic Recovery

- By heating a sample above the glass transition temperature and then cooling it back to room temperature, the previous thermal history is erased.
  - The second heat typically shows the true properties of the material rather than the material properties with some processing effects
- The endothermic peak that develops in the glass transition with aging at temperatures below the glass transition temperature is due to “enthalpic relaxation.” This peak is known as enthalpic recovery.
  - It is due to the fact that amorphous materials are not in thermodynamic equilibrium but, with time, do relax and move towards equilibrium.

Enthalpic Relaxation and Recovery

- Enthalpic Relaxation
  - The process of a meta-stable glass relaxing towards equilibrium at a temperature below *Tg*
  - Occurs as the sample is being cooled to temperatures below *Tg*
  - Occurs as the sample is being stored at temperatures below *Tg*
- Enthalpic Recovery
  - The recovery of energy (J/g) lost during Enthalpic Relaxation. It (peak in DSC data @ *Tg*) occurs as the sample is heated to a temperature above *Tg*

Effect of Annealing on the Tg



Measuring Tg in Complex Samples with MDSC®

- Complex samples can have overlapping transitions which make it difficult to detect or measure Tg
- MDSC experimental conditions which provide some cooling during temperature modulation are recommended – adds sensitivity
- Use an underlying heating rate that is slow enough to provide 4 or more modulation cycles over the transition of interest in order to improve separation of overlapping events (resolution)

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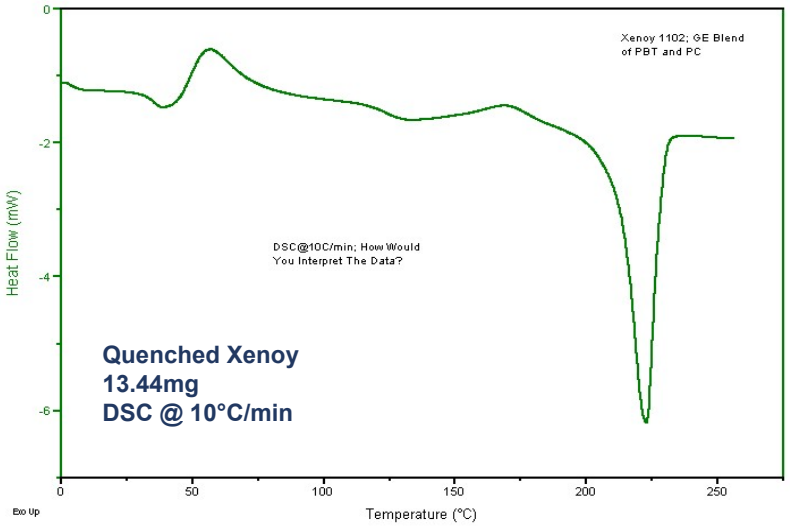
DSC of Complex Polymer Blend



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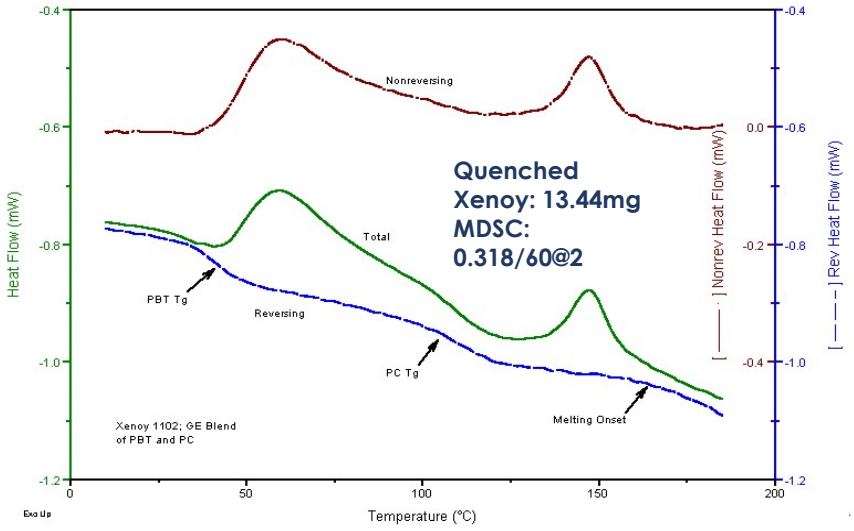
MDSC® of Complex Polymer Blend



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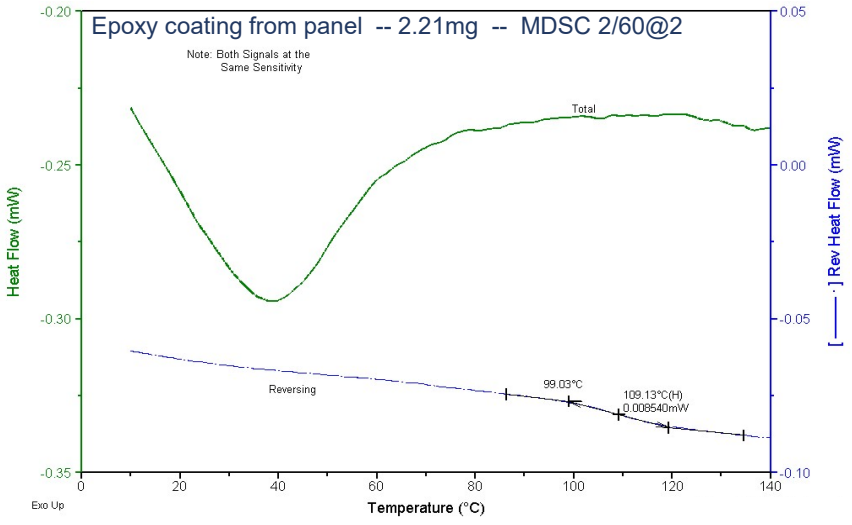
MDSC® of Weak Tg



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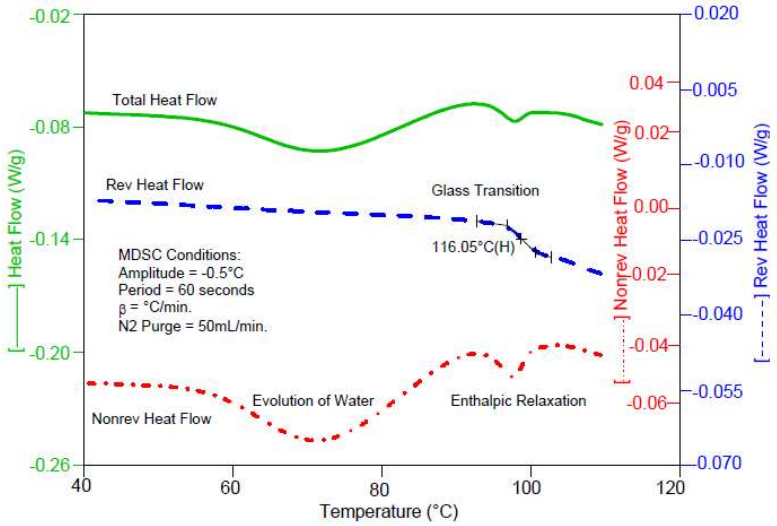
Glass Transition of Lactose by MDSC®



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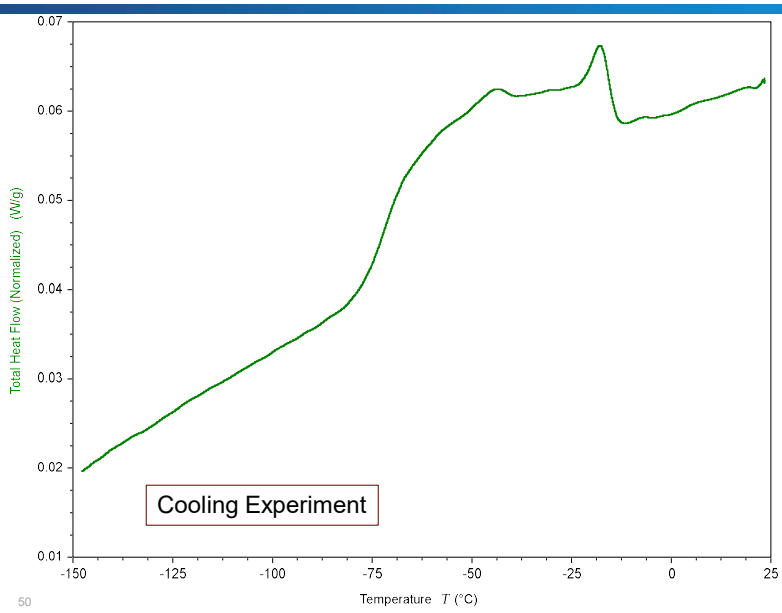
MDSC® of a Process Oil – Low Temperature Transitions in a Process Oil



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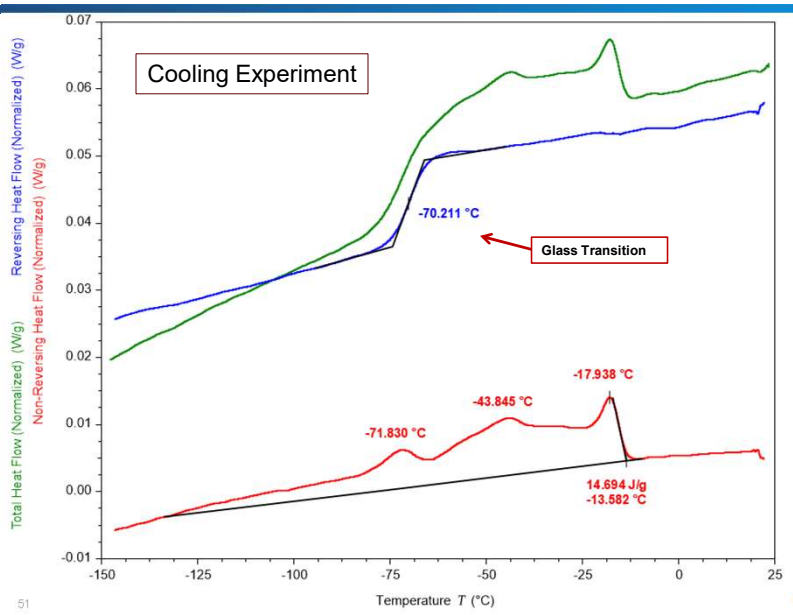
MDSC® of a Process Oil – Low Temperature Transitions in a Process Oil



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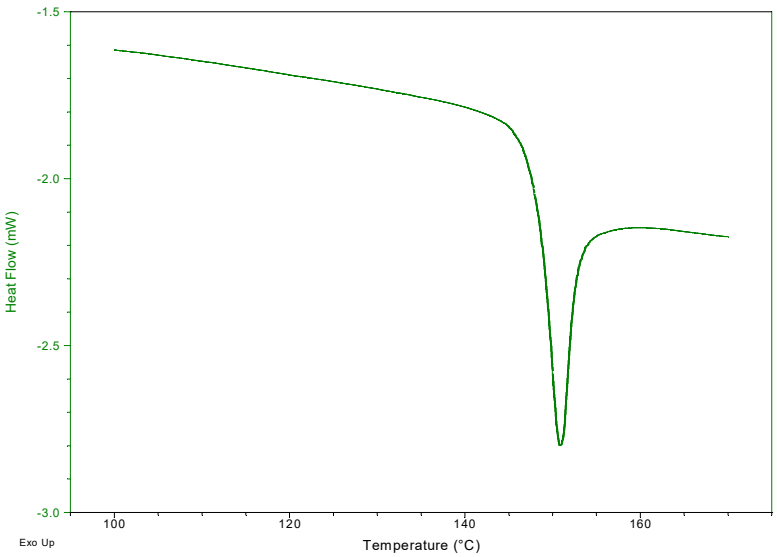
Is this a Tg or a Melt?



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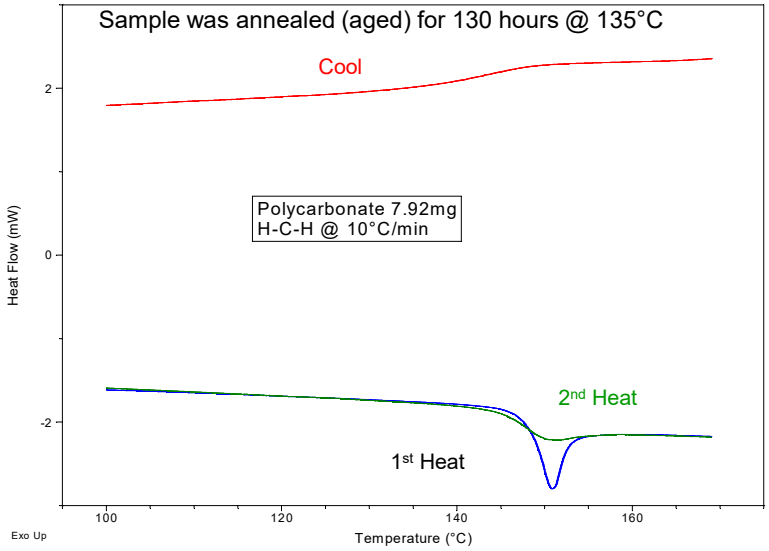
Now – Is this a Tg or a Melt?



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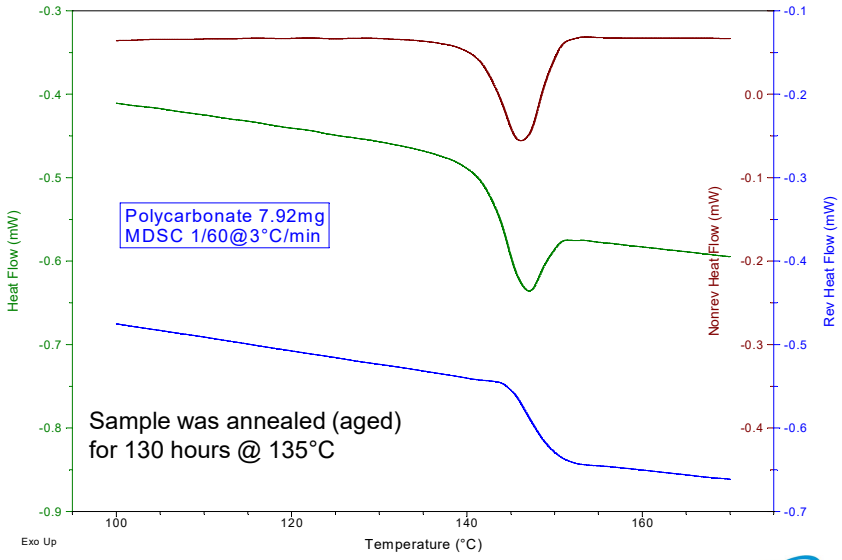
MDSC of Aged Polycarbonate



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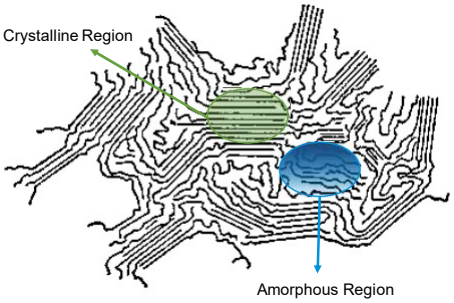
Is it a Tg?

- If not sure if a transition is a Tg
  - Run Heat-Cool-Heat (H-C-H)
    - If transition is a Tg then it should be present on cooling curve and 2nd heat
  - Run MDSC
    - A Tg will always show up in the Reversing Curve of a MDSC experiment
    - Use “conventional MDSC” template
    - Period: 60 seconds
    - Amplitude: 1°C
    - Rate: 2-3°C/min
  - Run TMA or DMA

Melting and Crystallization Analysis
(Crystalline Structure)

Semi-Crystalline Polymers

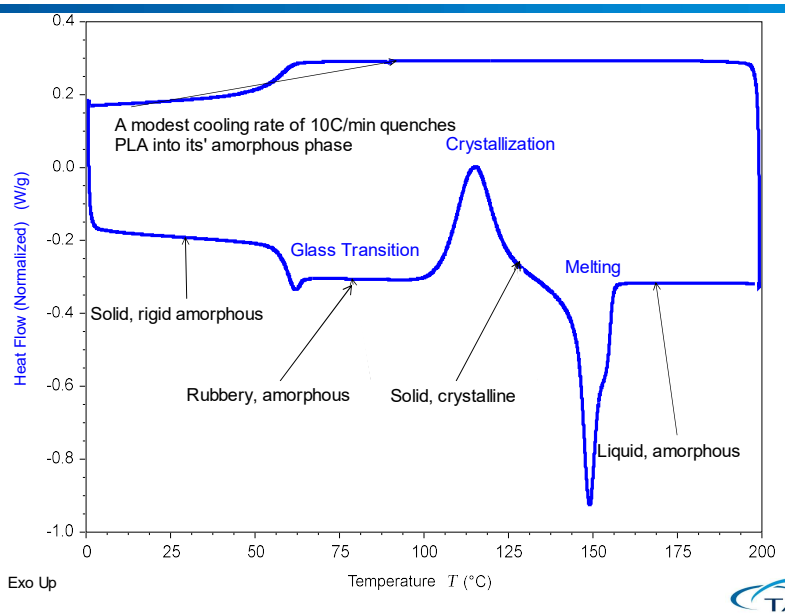
- Crystalline Structure –
  - Molecules arranged in well defined structures
  - Consists of repeating units
  - Polymers can have crystalline phases
    - Length of molecules prevents complete crystallization
- Semi-crystalline Polymers –
  - Both amorphous & crystalline solid phases
  - Examples are most common thermoplastics
    - Polyethylene, Polypropylene, etc



Study of melting/crystallization using a DSC

- Melting is the process of converting solid, crystalline structure (lower energy) to a liquid amorphous structure (higher energy).
- Melting:
  - low energy state → high energy state; requires input of energy; Endothermic peak
- Crystallization – The process of converting either solid amorphous structure (cold crystallization on heating) or liquid amorphous structure (cooling) to a more organized solid crystalline structure
- Crystallization:
  - high energy state → low energy state; releases energy; Exothermic peak
- We integrate these peaks, on a time basis to determine the Heat of Fusion (melting) and Heat of crystallization

DSC Analysis of Polylactic Acid (PLA)



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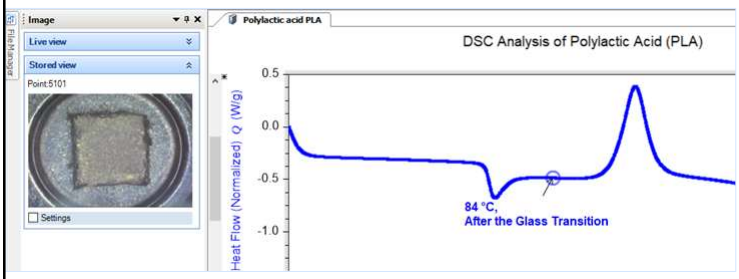
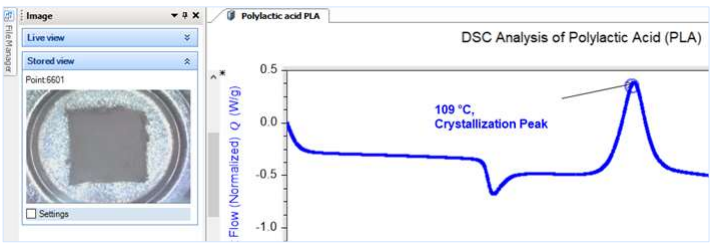
Discovery DSC Microscope Accessory





Provides imaging and video capabilities during a DSC measurement on the Discovery DSC 2500, 250 and 25

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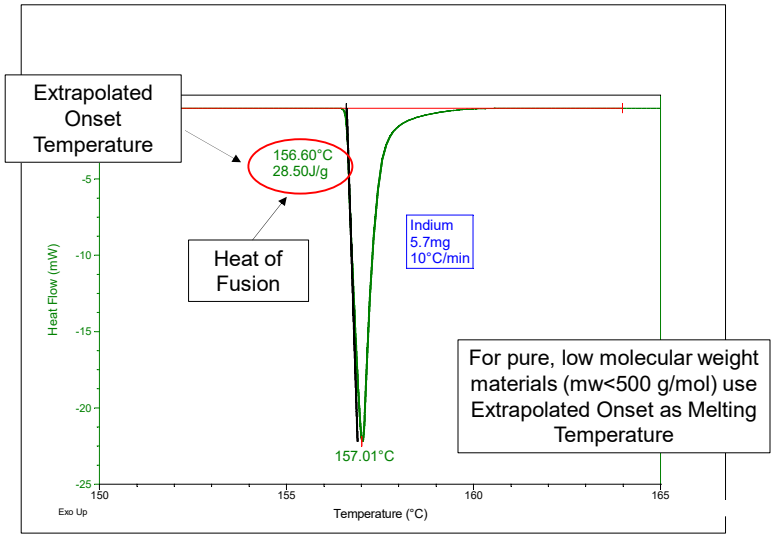
Melting of Indium – (Low molecular weight)



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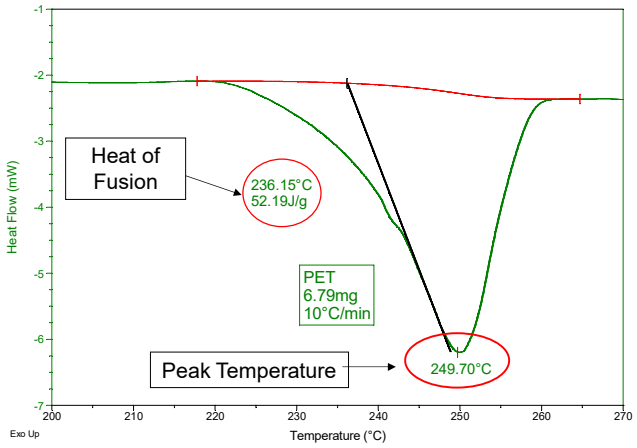
Melting of PET – (High molecular weight)

For polymers, use Peak as Melting Temperature



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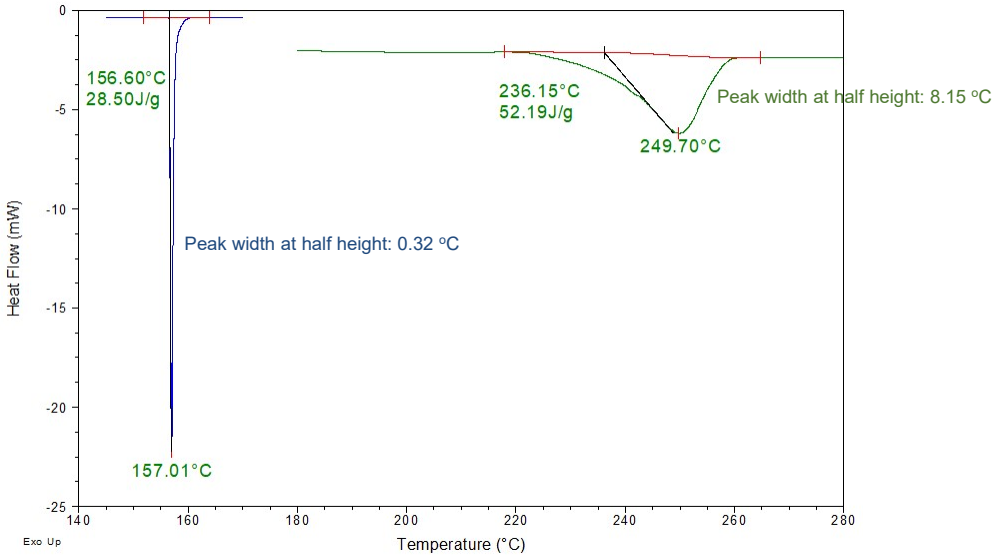
Comparison of Melting of Polymer and Metal



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Definitions of some terms commonly used in crystallinity analysis

- Thermodynamic Melting Temperature – The temperature where a crystal would melt if it had a perfect structure (crystal with no defects)
- Metastable Crystals – Crystals that melt at lower temperature due to small size (high surface area) and poor quality (large number of defects)
- Crystal Perfection – The process of less perfect crystals (metastable) melting at a temperature below their thermodynamic melting point and then (re) crystallizing into more perfect crystals that will melt again at a higher temperature.

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Crystal Structure Analysis

- Crystal structure is typically broken down during the process of melting
- The formation of crystalline structure or crystallization can occur during heating or cooling
  - Cold crystallization occurs on heating when a solid amorphous material becomes ordered
  - Crystallization on cooling occurs when the liquid amorphous material solidifies into an ordered solid
- Typically, the same amount of energy required to create the structure during crystallization is also required to break down the crystal structure during melting

Selecting Experimental Conditions for Crystallinity Studies

- During first heat the maximum temperature must be higher than the end of melting peak; eventually an isothermal period must be introduced
  - Too high temperature/time:
    - decomposition could occur
  - Too low temperature/time:
    - possibly subsequent memory effect because of the fact that crystalline order is not completely destroyed
- For non-crystallizable (amorphous) polymers the maximum temperature should be above *Tg* (removal of relaxation effects, avoid decomposition)

- MDSC Test conditions:
- Use “MDSC Heat-only” template
- Period: 60 seconds
- (template calculates amplitude for you)
- Heating rate: 2-3°C/min

Heat capacity baseline and Integration Limits

- True Heat Capacity Baseline – Often called the thermodynamic baseline, it is the measured baseline (usually in heat flow rate units of mW) with all crystallization and melting removed.
  - Assumes no interference from other latent heat such as polymerization, cure, evaporation etc. over the crystallization/melting range.
- It is often difficult to select limits for integrating melting peaks
  - Integration should occur between two points on the heat capacity baseline
  - Heat capacity baselines for difficult samples can usually be determined by MDSC® or by comparing experiments performed at different heating rates

Calculation of Initial Crystallinity

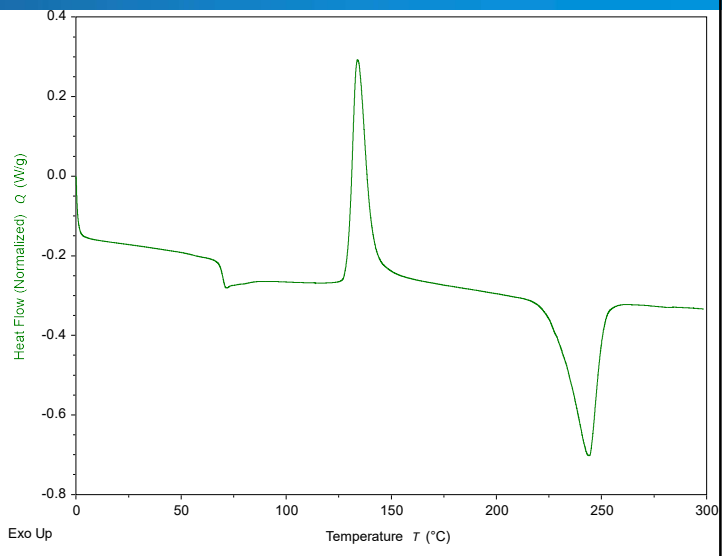
- Because crystalline structure can form as the sample is heated, the actual crystallinity of the sample is usually less than measured
- Sample must be pure material, not copolymer or filled
- Must know enthalpy of melting for 100% crystalline material (ΔHlit)
- You can use a standard ΔHlit for relative crystallinity

For standard samples:

$$\% \text{ crystallinity} = 100 * \Delta H_m / \Delta H_{lit}$$

For samples with cold crystallization:

$$\% \text{ crystallinity} = 100 * (\Delta H_m - \Delta H_c) / \Delta H_{lit}$$



PET Data from ATHAS

Poly(ethylene terephthalate) (PET)

Crystalline Calculated Data

```
*********************************************************************
*          Advanced   THermal   AnalysiS   Laboratory              *
*                  1993  Recommended Data  of                      *
*       Thermodynamic   Properties   of   Macromolecules           *
*********************************************************************

Name :              Poly(ethylene terephthalate)

File Name :         PET

Structure :     O       O
                ||      ||
               (O-C-C6H4-C-O-CH2-CH2-)
```

Calculate g/mole from molecular structure which equals 192 g/mole for PET

< Crystalline >

T	index	Cp	H - HO[C]	S	HO[C] - G
(K)	*	(J/K.mol)	(J/mol)	(J/K.mol)	(J/mol)
0.10	4	0.000	0.00	0.000	0.00
0.20	4	0.000	0.00	0.000	0.00

ATHAS Summary Page for PET

ΔH_f in kJ/mol

Poly(ethylene terephthalate) (PET)

Summary

	Tg	dCp	Tm	dHf	SHG	So	Theta1	Theta3	Ns	Cp
(c)	-	-	553	26.9	X	0	586	54	15	1.0-10
(a)	342	77.8(4+1)	-	-	X	22	586	44	15	1.0-590
PET	8	8	10,43	10	8,57	33*	30	30	30	8,29

- Explanations

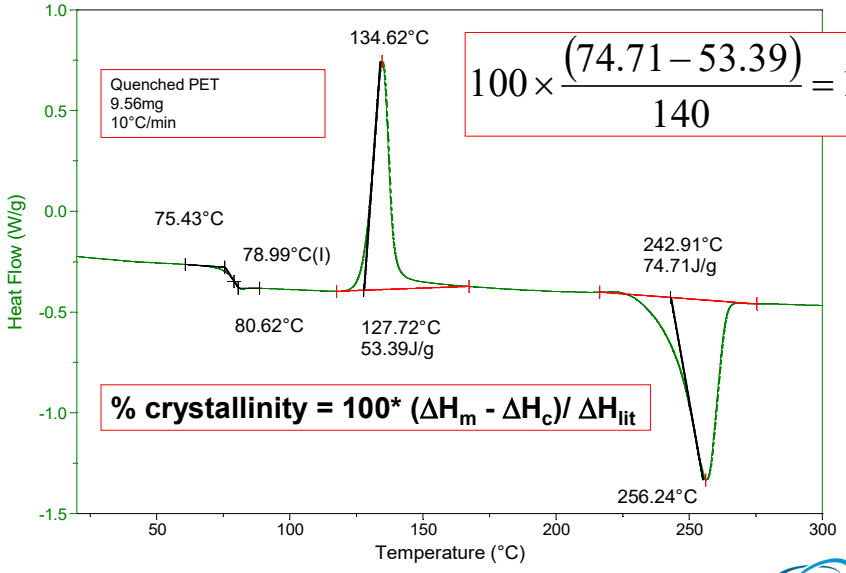
The data are separated into

- Cp Experimental and Calculated -Crystalline
- Cp Experimental and Calculated -Amorphous
- Cp, H,S,G -Crystalline
- Cp, H,S,G -Amorphous
- Cp Figure, H,S,G Figure These are picture files and may need some time to load.
- References

Last revision May 6, 1997 by Marek Pyda
URL : http://funnelweb.utcc.utk.edu/~athas/.../phenylen/pet/pet.html

$$\frac{26.9 kJ/mol}{192 g/mol} \times 1000 = 140 J/g$$

Quench-cooled PET – calculation of initial crystallinity



$$100 \times \frac{(74.71 - 53.39)}{140} = 15\%$$

???

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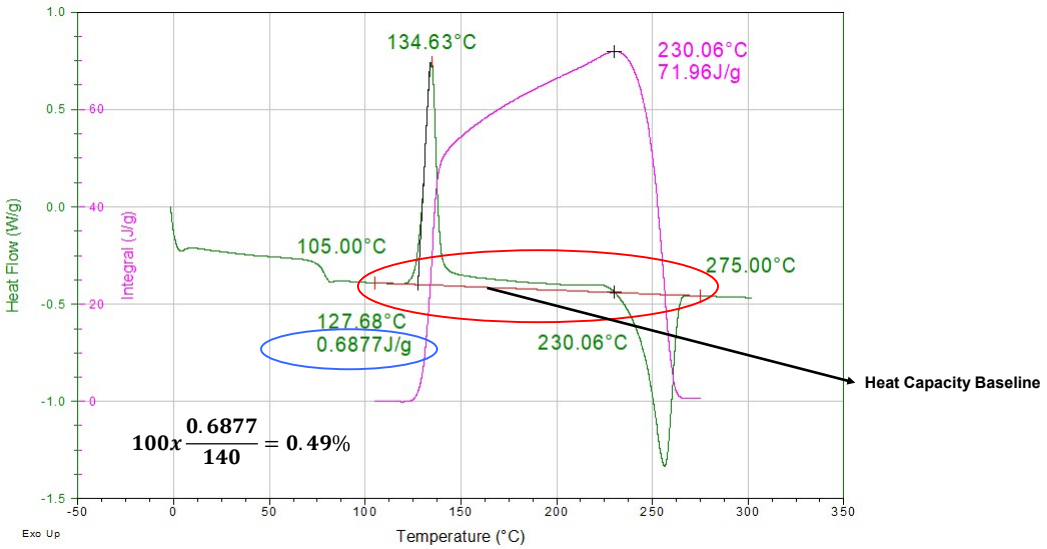
Change in Crystallinity While Heating



$$100x\frac{0.6877}{140} = 0.49\%$$

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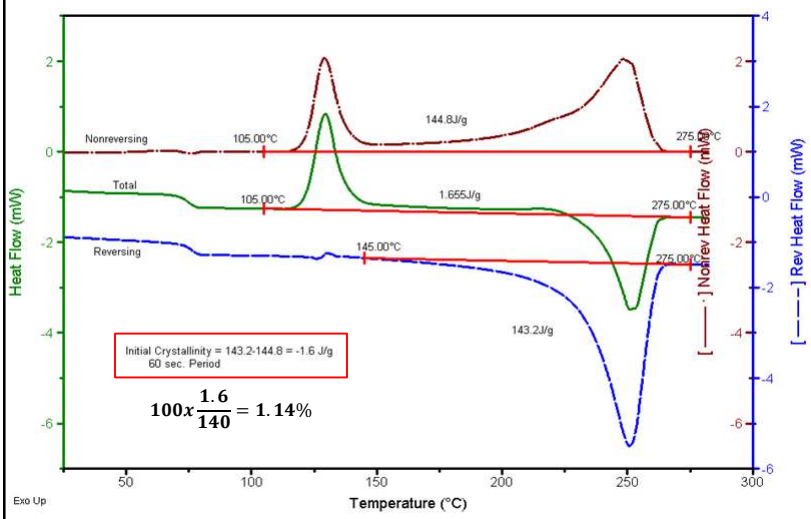
MDSC for measuring Initial Crystallinity



- MDSC "Initial Crystallinity" is always calculated from the sum of all crystallization exotherms and melting endotherms
  - Integrate the Nonreversing signal from the start of crystallization to the end of melting
  - Integrate the Reversing signal from the start of melting to the end of melting

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Impact of crystal perfection on the melting peak

- The shape of the melting peak is also affected by crystal perfection processes that occur over the same temperature range as bulk melting.
  - This often gives the appearance of two melting peaks rather than what actually is an exothermic crystallization peak superimposed on an endothermic melting peak.

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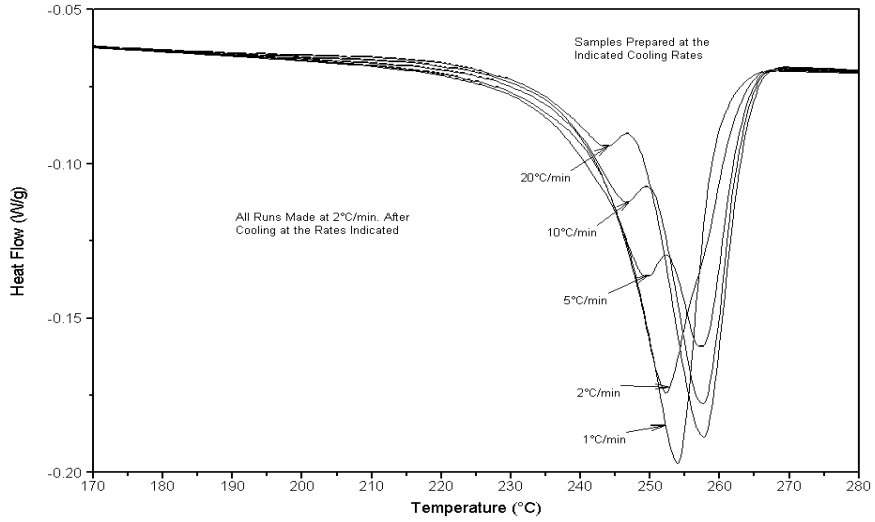
PET Melting after Cooling at Different Rates



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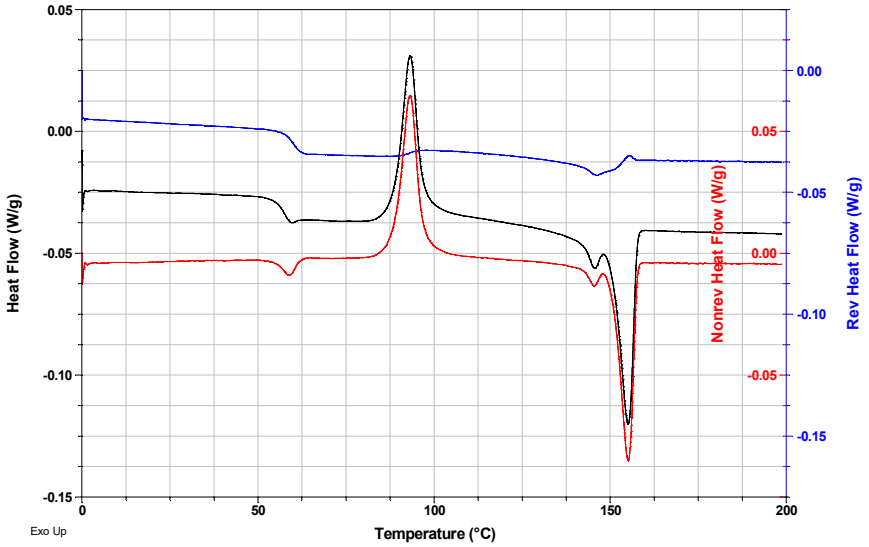
MDSC: Thermal Transitions in PLA



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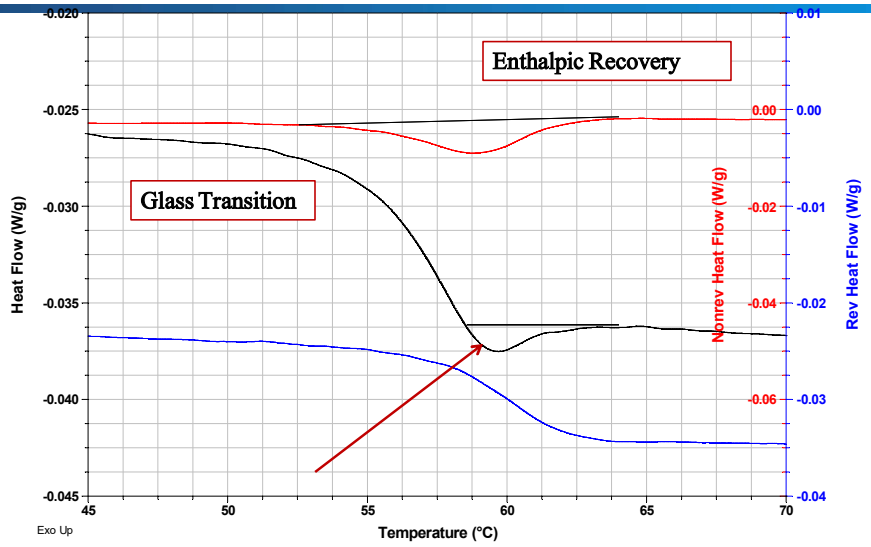
MDSC Applications: Thermal Transitions in PLA



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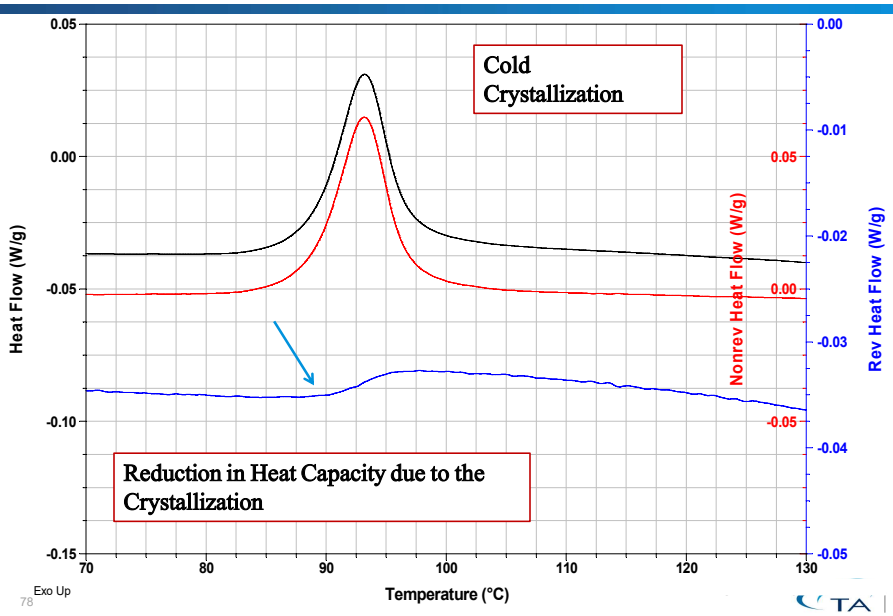
MDSC Applications: Thermal Transitions in PLA



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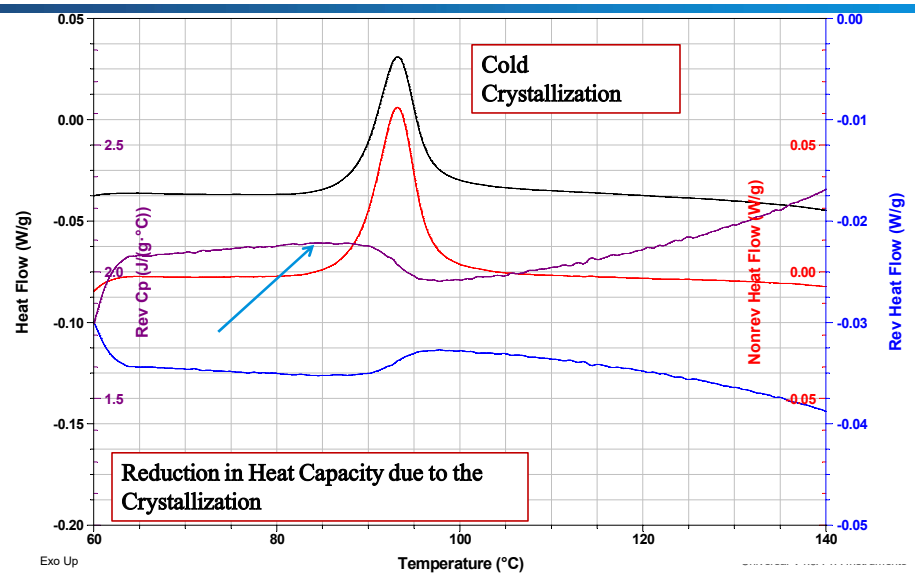
MDSC Applications: Thermal Transitions in PLA



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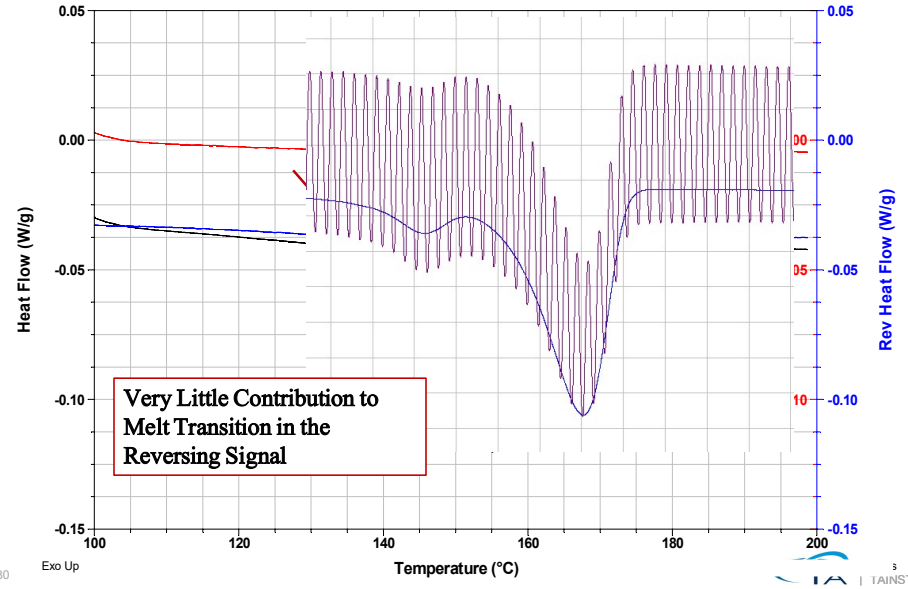
MDSC Applications: Thermal Transitions in PLA



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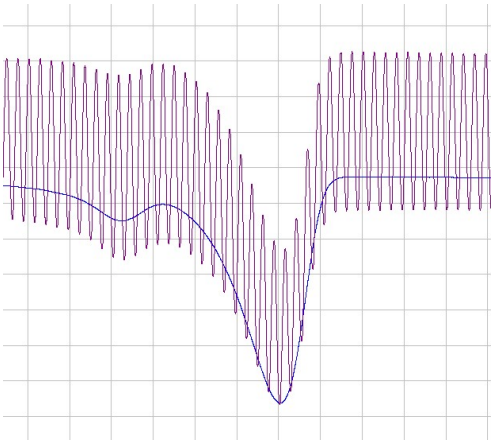
MDSC Applications: Polymer Transitions; PLA

- Based on the number of modulations through the transition, it is reasonable to conclude that our experiment is valid.
- Sometimes we see this from highly crystalline materials.



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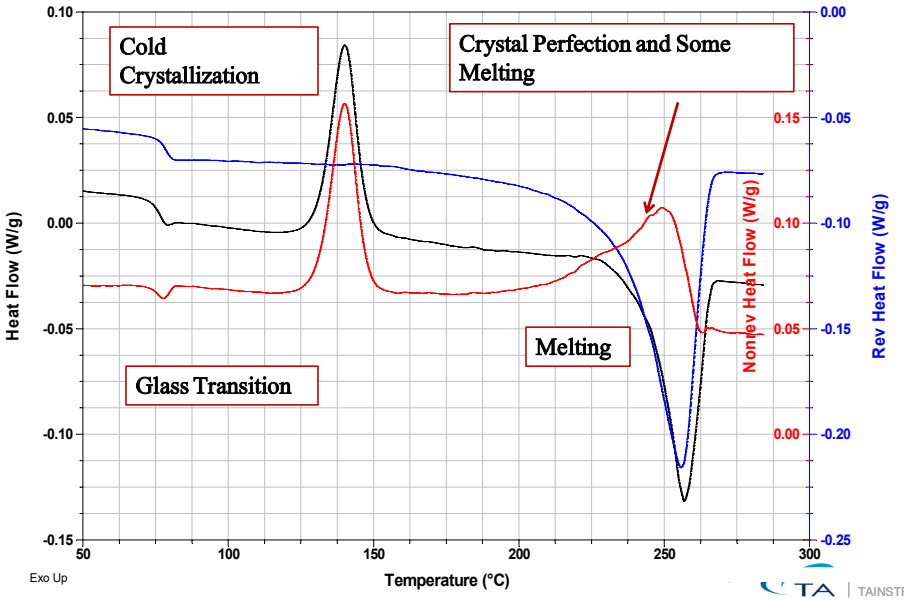
MDSC Applications: Polymer Transitions; PET



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Thermosets

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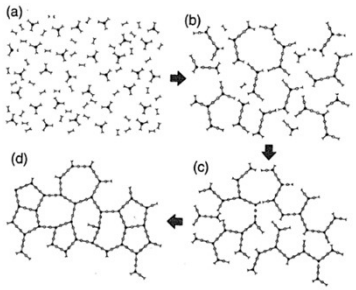
Thermosetting Polymers

A “thermoset” is a cross-linked polymer formed by an irreversible exothermic chemical reaction

Thermosetting polymers react (cross-link) irreversibly. A+B will give out heat (exothermic) when they cross-link (cure).

A + B → C



-R. Bruce Prime, *Thermosets*, Thermal Characterization of Polymeric materials

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Typical properties of crosslinking reactions

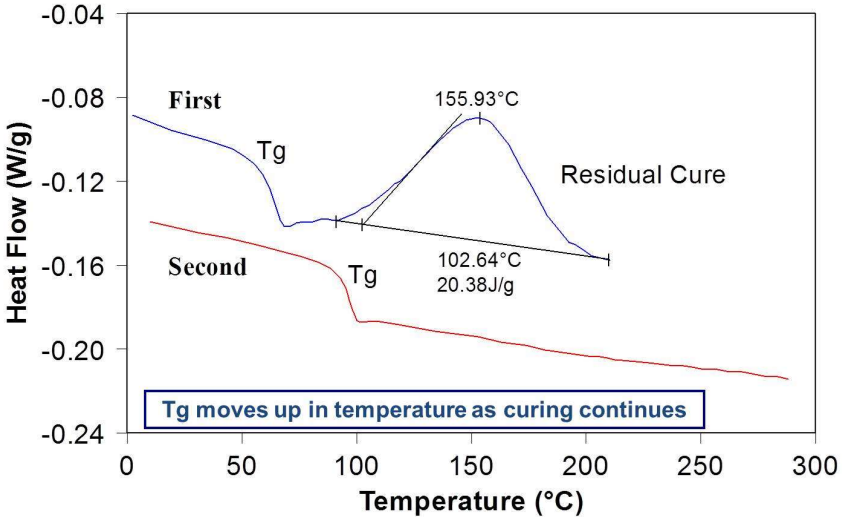
- Crosslinking reactions are generally exothermic. The reactions can be easily monitored using a DSC.
  - **Heat of reaction**
  - **Residual cure**
  - **Glass transition**
  - **Heat capacity**
- Crosslinking reactions are generally accompanied by a sharp change in the material's mechanical properties.
- Increase in modulus that may be accompanied by shrinkage.
- The reactions can thus be monitored using a Thermo-mechanical Analyzer (TMA)/Dynamic Mechanical Analyzer (DMA)/Rheometer.
  - **Viscosity**
  - **Modulus**
  - **Glass transition**
  - **Dimension change (shrinkage)**

These techniques give useful information about the impact of the polymerization conditions on the end product's thermo-mechanical properties.

DSC: General considerations for selecting optimum experimental conditions

- Sample weight: 10–15 mg
- Pan types:
  - Solids – Standard aluminum pan/lid
  - Liquids – Hermetic aluminum pan/lid
- General protocol for studying thermosets:
  - **<u>Determine decomposition temperature using TGA</u>**
  - Heat-Cool-reheat at 10°C/min
  - First Heat is used to measure Tg of starting material, heat of reaction and presence of any reactive functional groups.
  - Second Heat is used to measure the Tg of the fully cured sample and any residual cure from the first heat.

Comparison of First and Second Heats



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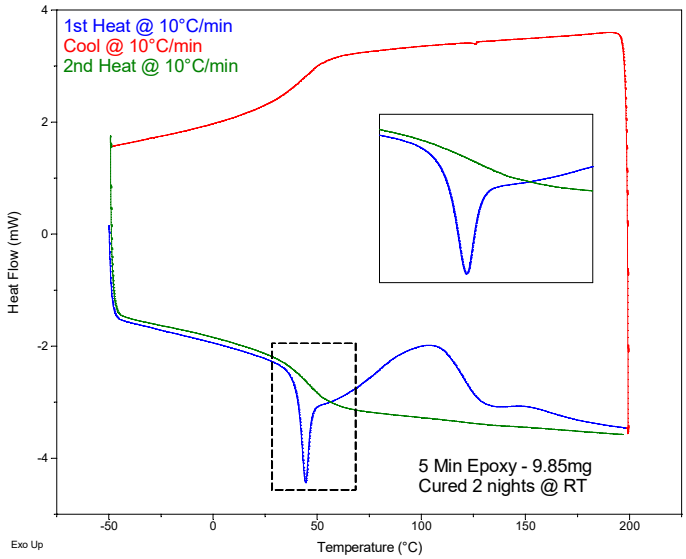
Epoxy Cured 48 Hours: Heat Cool Heat



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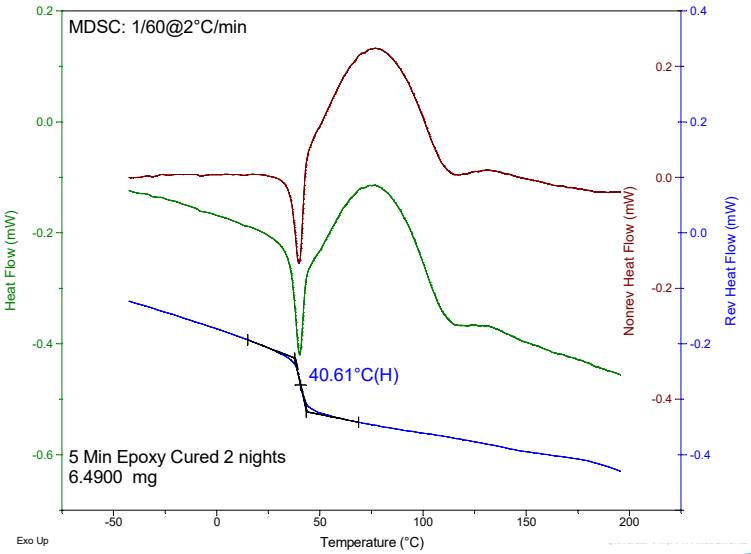
Rev-Heat Flow Easily Shows Tg



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Percent Cure Calculation by DSC

- Need Heat of Reaction (Enthalpy) of unreacted material curing
  - Typically run uncured material in DSC
- Run cured or partially cured sample in DSC

% Uncured = (ΔH Residual Cure / ΔH Full Cure) * 100

% Cure = 1 - (ΔH Residual Cure / ΔH Full Cure) * 100

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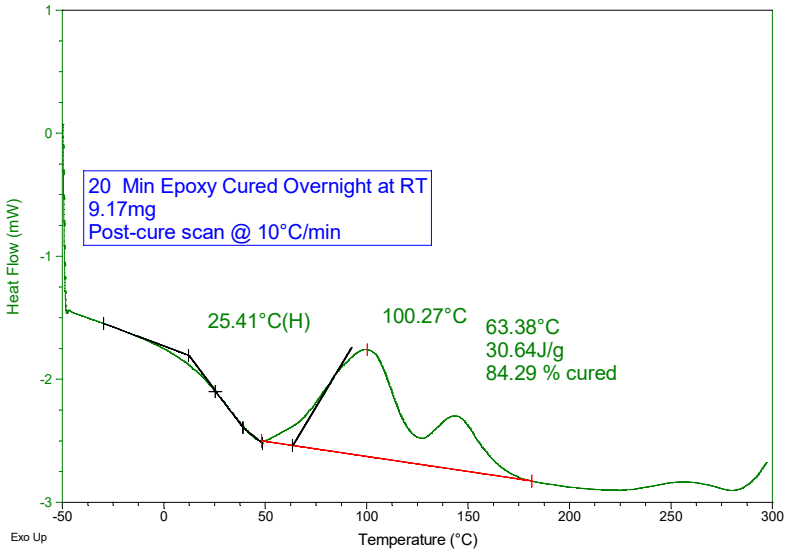
Epoxy Cured Overnight at Room Temp – Standard DSC



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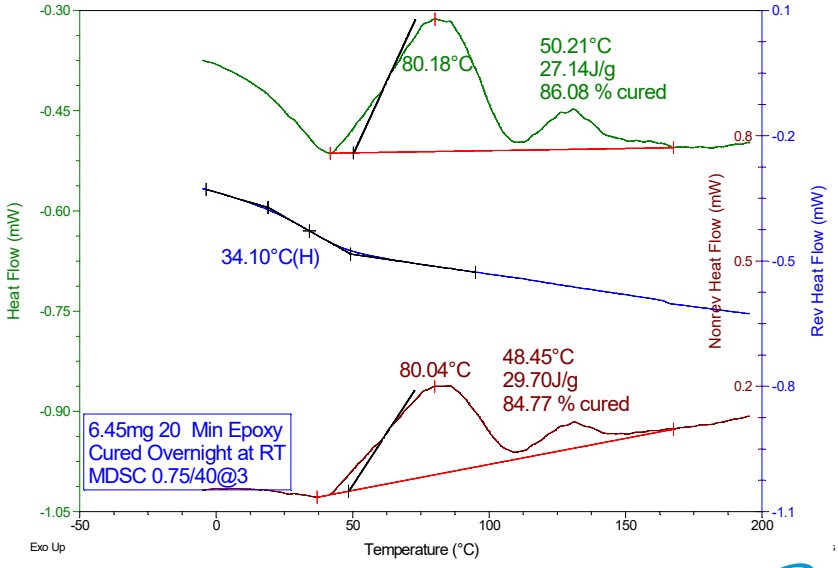
Epoxy Cured Overnight at Room Temp - MDSC



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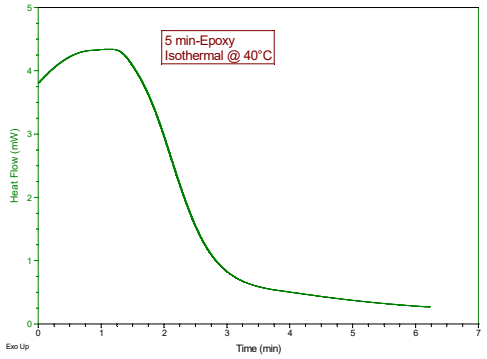
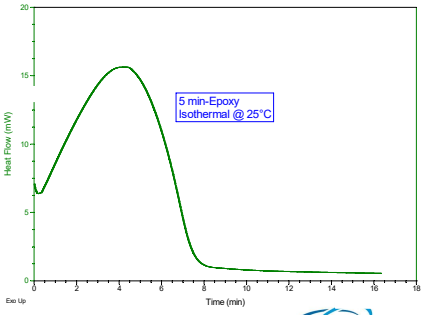
Isothermal curing of a Thermosetting Material



Temperature is too high
Missed most of the reaction



Temperature is better

Need to either go even lower in temperature or work faster

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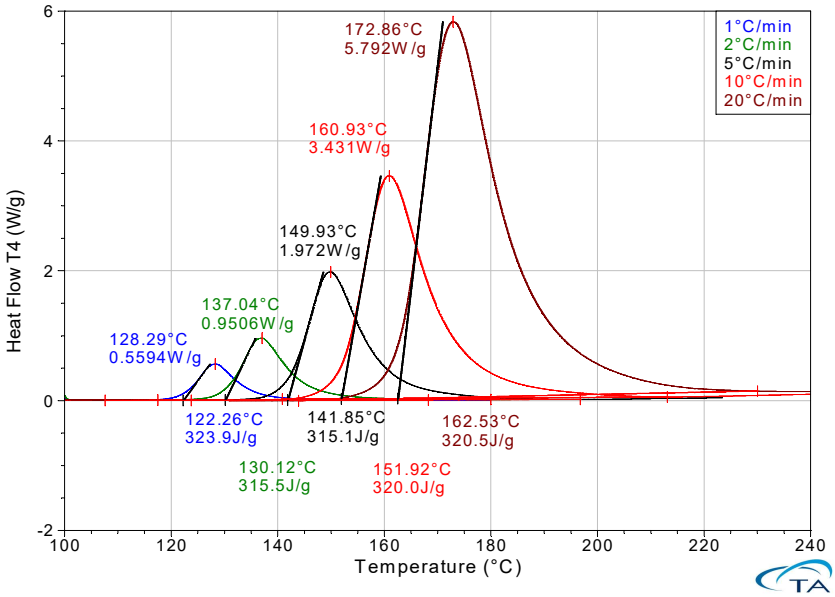
Curing reactions are kinetic in nature



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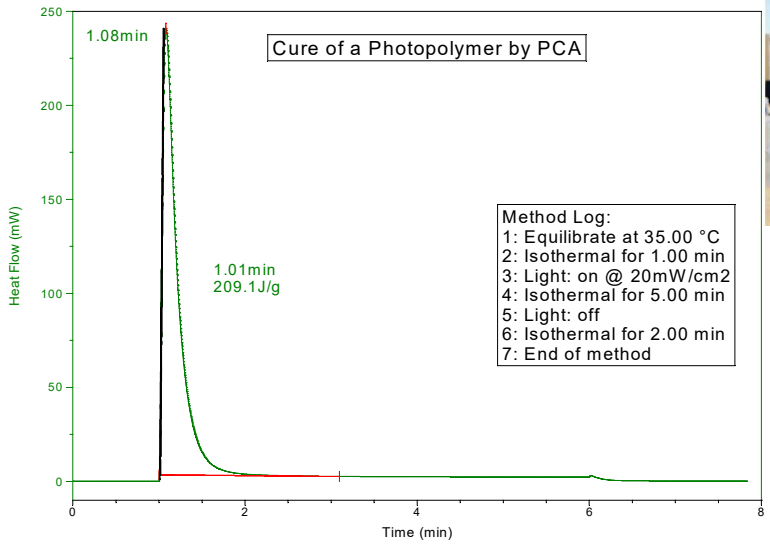
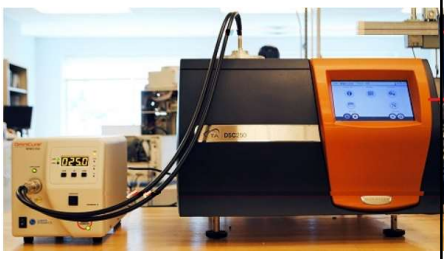
Photopolymer Cure by PCA





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Pharmaceuticals

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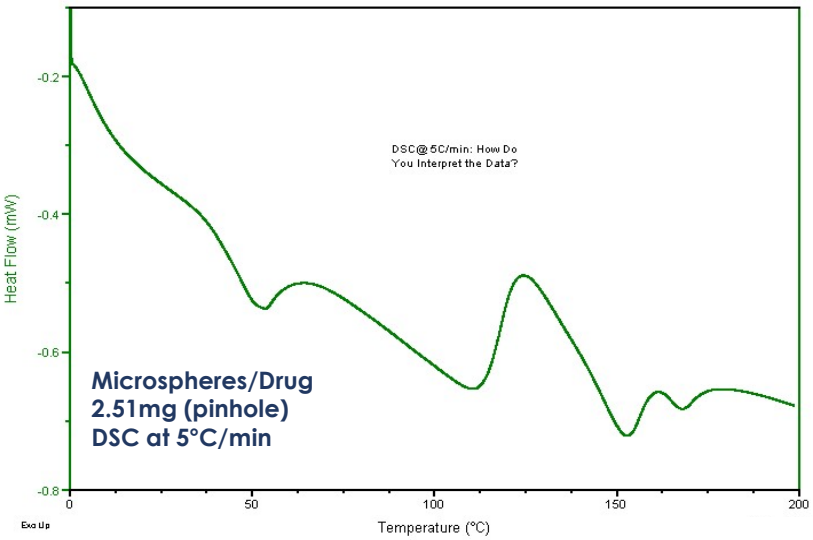
Thermal Events in Pharmaceuticals

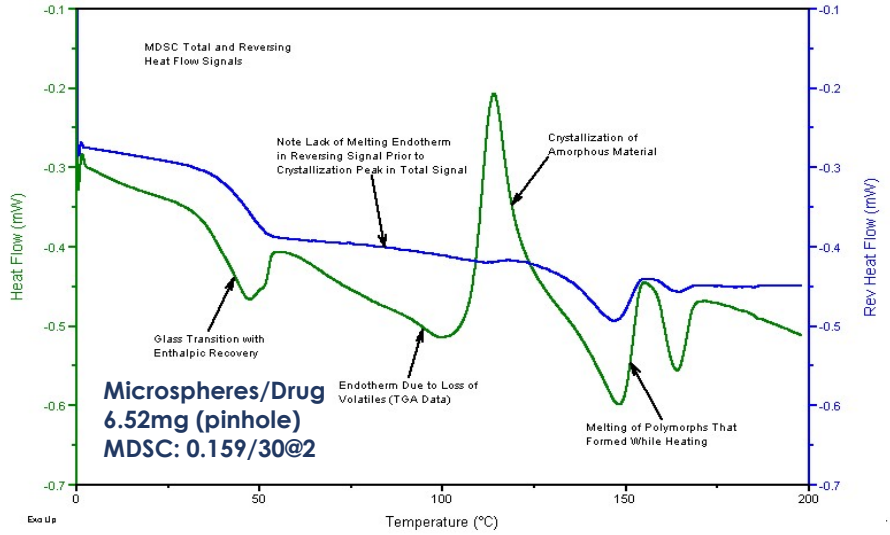
- Tg
- Melting
  - Purity
- Polymorphs

- General Recommendations
  - Use TGA to determine pan type
  - Use 1-5 mg samples (use 1mg for purity)
  - Initial H-C-H @ 10°C/min (1°C/min for purity)
  - If polymorphs present, heat faster to inhibit polymorphic transformations

DSC at 5°C/min for Drug Microspheres



MDSC for Drug Microspheres



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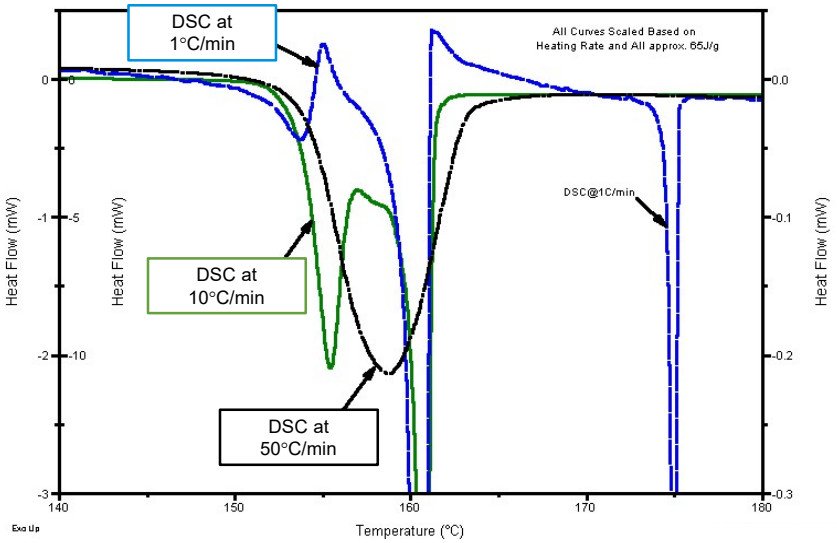
Effect of Heating Rate on Polymorph



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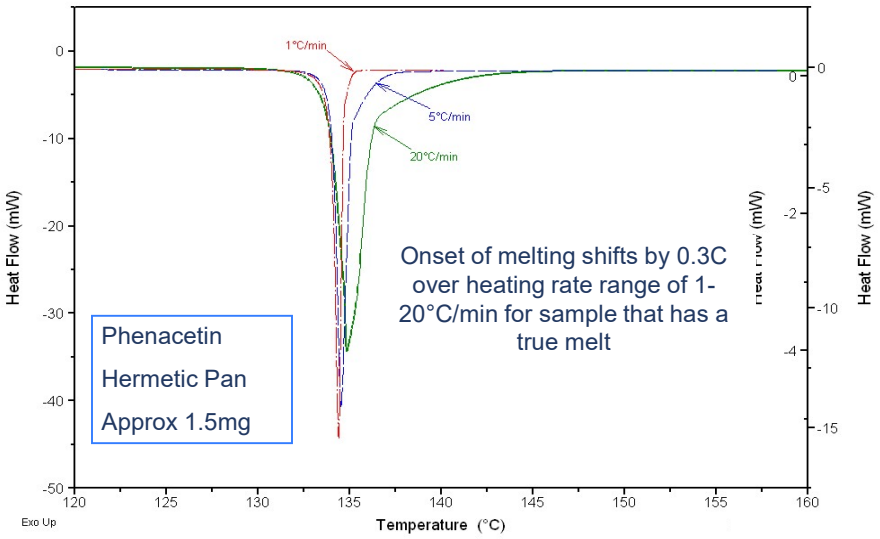
Thermodynamic Melting (Not Heating Rate Dependent)



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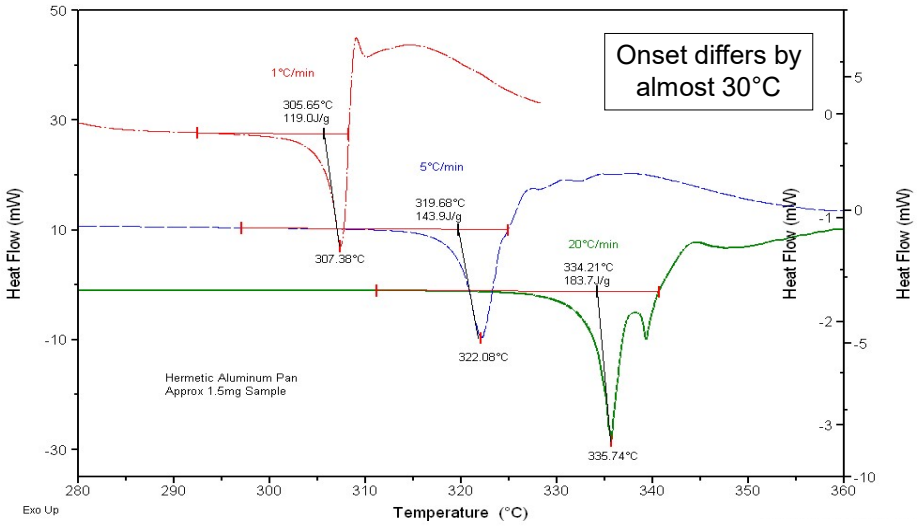
Apparent Melting (Heating Rate Dependent)

Ciprofloxacin Hydrochloride Decomposes



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Evaluating Material Compatibility Using DSC

- **Active components** of drugs are mixed with other materials called **excipients** that act as bulking agents, improve long-term stability, or as processing lubricants
- Understand the Interaction of excipients with the active pharmaceutical ingredient (API) is critical when developing a formulation
- The Discovery X3 DSC is a multi sample DSC that allows running 3 samples simultaneously
- Increase in productivity, ability to run MDSC
- Compatibility studies of API and excipients can be done In one test under the same conditions

Discovery X3 DSC

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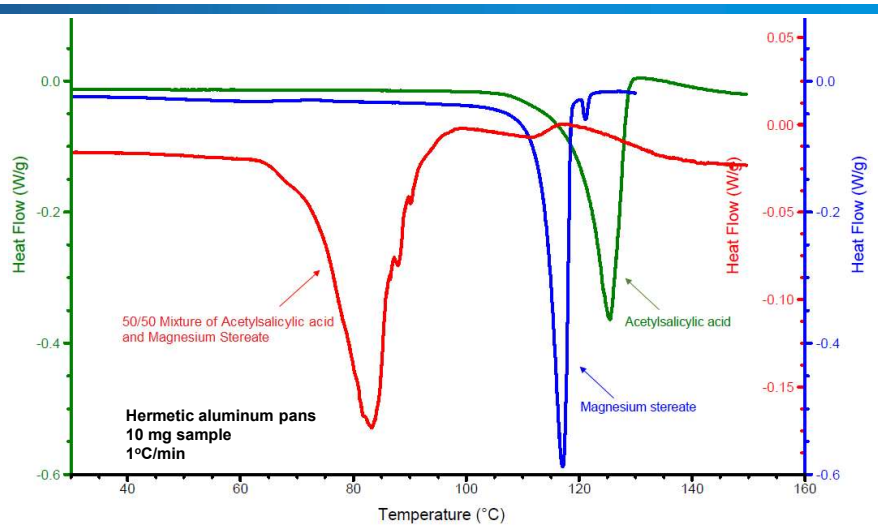
Detection of Apparent Melting by Chemical Interaction in the X3 DSC

Chemical interaction between these materials causes a loss of crystalline structure at a much lower temperature



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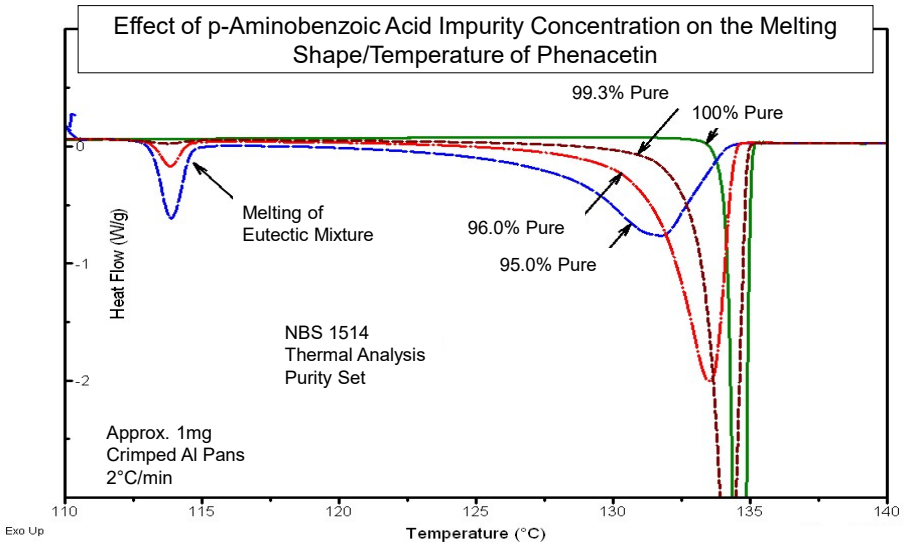
Effect of Impurities on Melting



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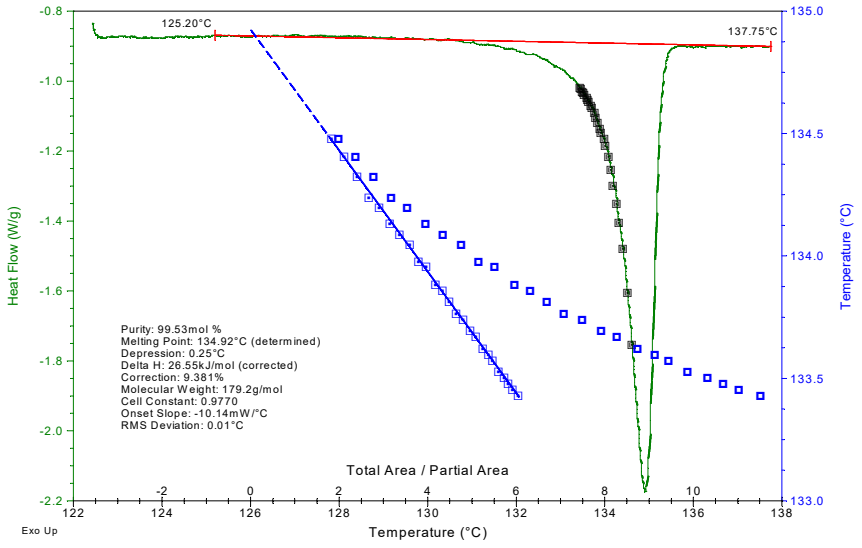
Van't Hoff Purity Calculation



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Remainder and Final Notes

- MDSC® is a slow technique. Always start the analysis of a new material with standard DSC and only use MDSC if you need:
  - Improved sensitivity
  - Better resolution
  - Separation of overlapping transitions
  - Most accurate measurement of polymer crystallinity
- DSC General Methods Recommendations:
  - Run a Heat-Cool-Heat at 10-20 °C/min
  - Use specific segments as needed, i.e. gas switch, abort, etc.
  - Ensure that the starting temperature of the experiment is chosen to encompass the entire transition (2 minutes of baseline)

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- For additional questions:
- Email thermalsupport@tainstruments.com
- Please put Online Training Questions in the subject line
- To download this presentation: https://www.tainstruments.com/online-training-course-downloads/

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