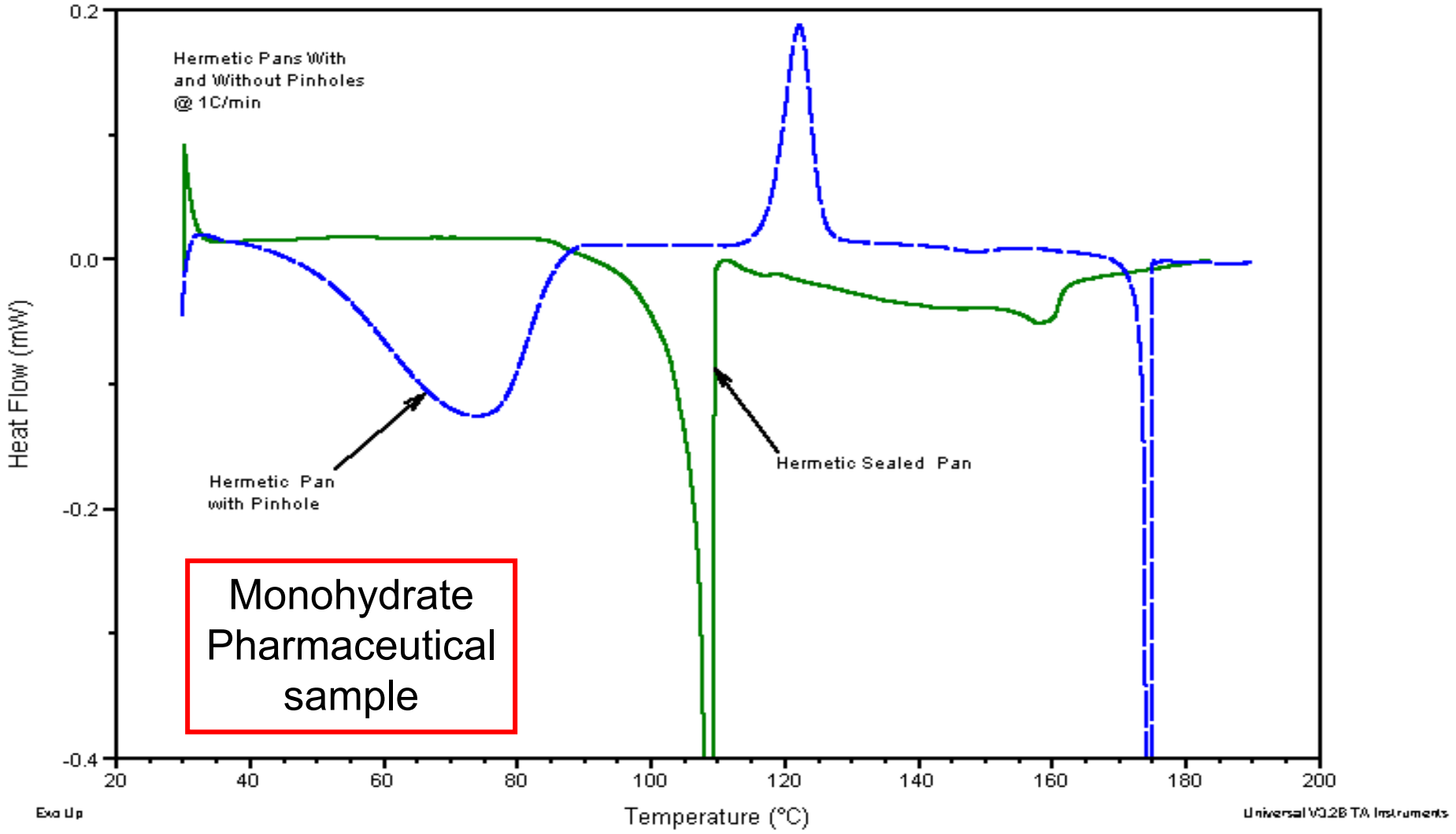
BASIC INFO FOR TRAINING. DSC Q2000.

Extracted from the ppt TA DSCQSeminar

It Does Matter What Pan you use

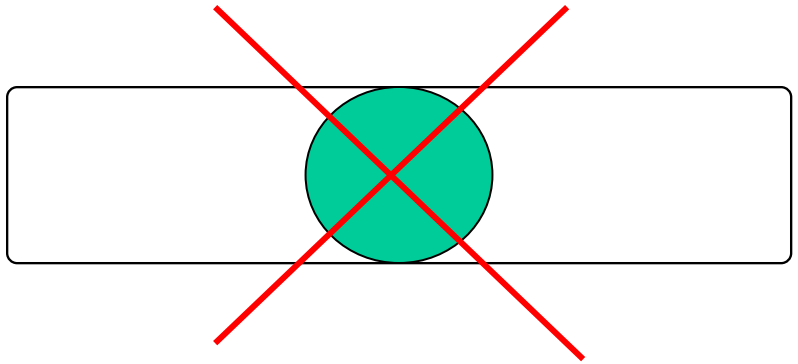


Sample Shape

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

Sample Shape

- Cut sample to make thin, don't crush
- If pellet, cut cross section



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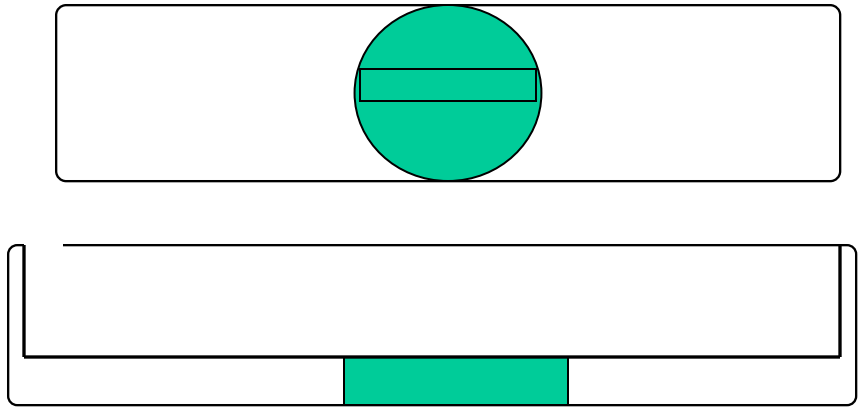
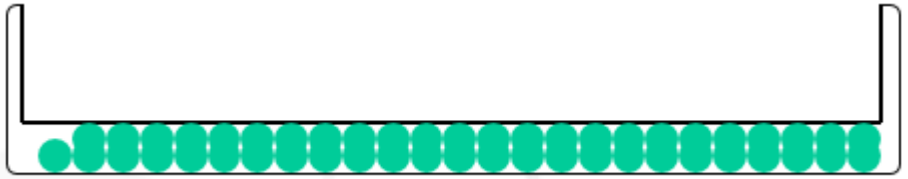


- If powder, spread evenly over the bottom of the pan



Sample Size

- Larger samples will increase sensitivity

  but……

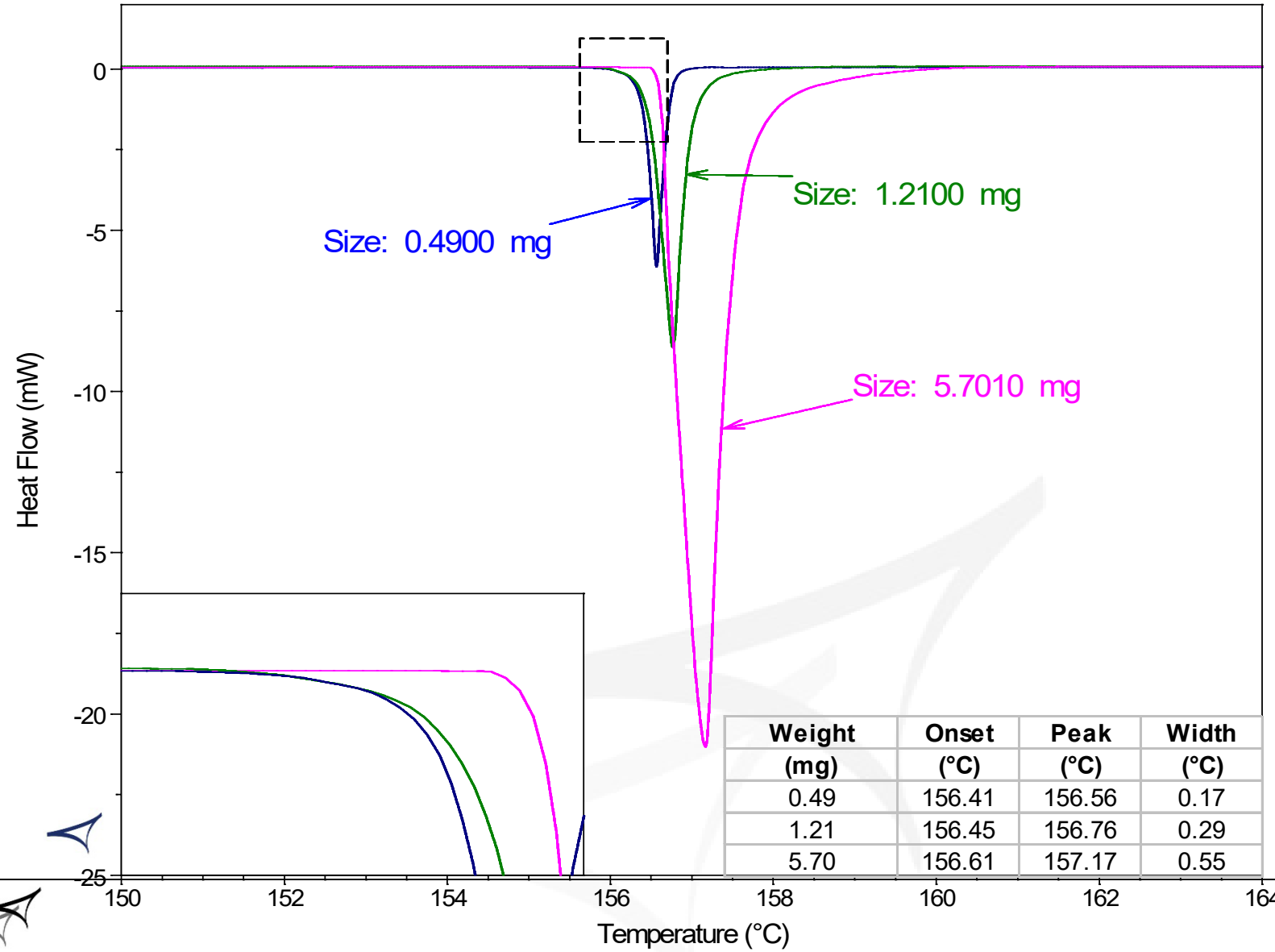
- Larger samples will decrease resolution

- Goal is to have heat flow of 0.1-10mW going through a transition

Sample Size

- Sample size depends on what you are measuring
  - If running an extremely reactive sample (like an explosive) run very small samples (<1mg)
  - Pure organic materials, pharmaceuticals (1-5mg)
  - Polymers - ~10mg
  - Composites – 15-20mg

Effect of Sample Size on Indium Melt



Weight (mg)	Onset (°C)	Peak (°C)	Width (°C)
0.49	156.41	156.56	0.17
1.21	156.45	156.76	0.29
5.70	156.61	157.17	0.55

Agenda

- Keeping your DSC cell clean
- Calibration
- Sample Preparation
- Thermal Method

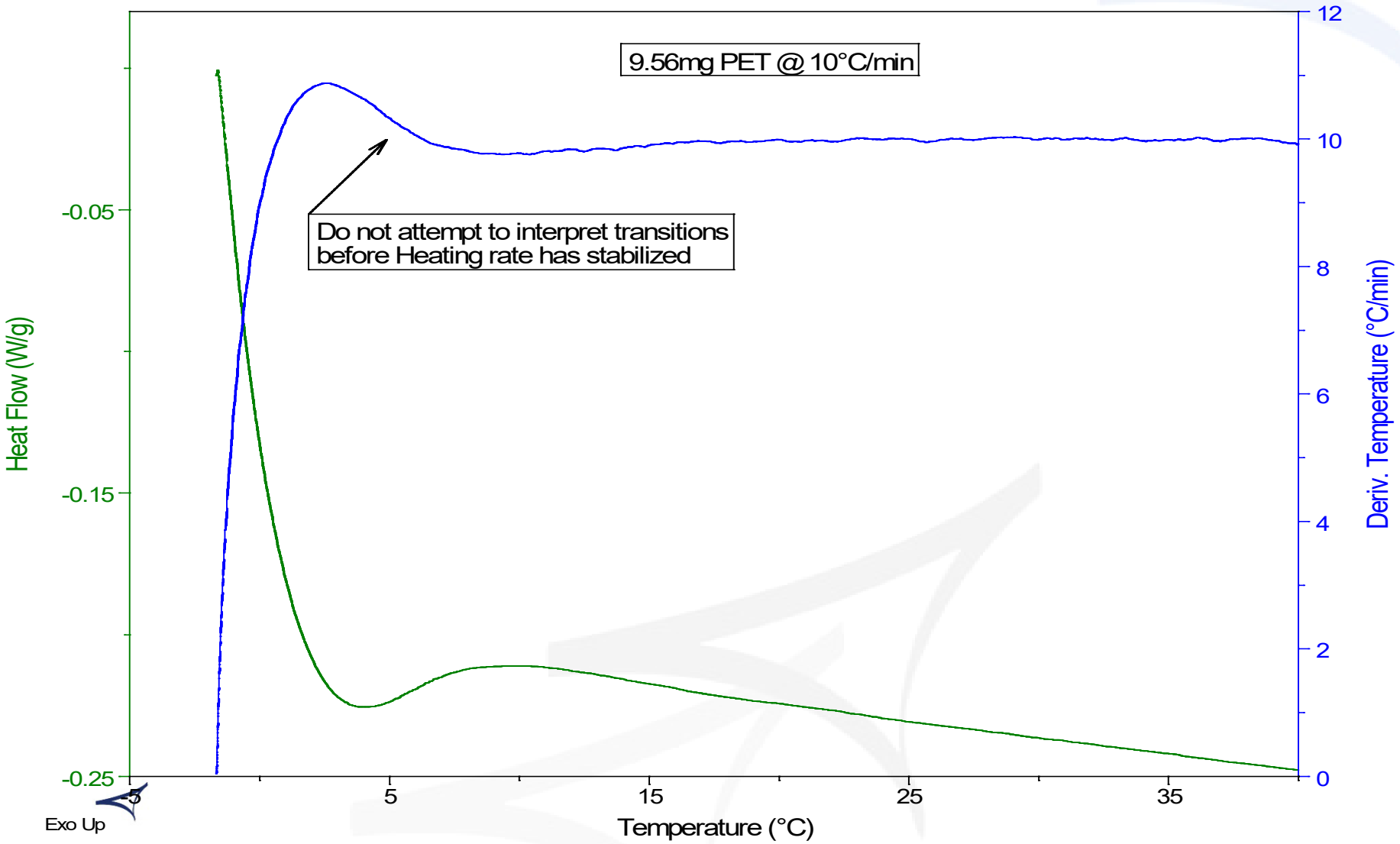
Purge Gas

- Purge gas should always be used during DSC experiments
  - Provides dry,inert atmosphere
  - Ensures even heating
  - Helps sweep away any off gases that might be released
- Nitrogen
  - Most common
  - Increases Sensitivity
  - Typical flow rate of 50ml/min

Sample Temperature Range

- Rule of Thumb
  - Have 2-3 minutes of baseline before and after transitions of interest - if possible
    - DO NOT DECOMPOSE SAMPLES IN DSC CELL
  - Temperature range can affect choice of pans
  - Just because the instrument has a temperature range of –90°C to 550°C (with RCS) doesn't mean you need to heat every sample to 550°!

Start-up Hook



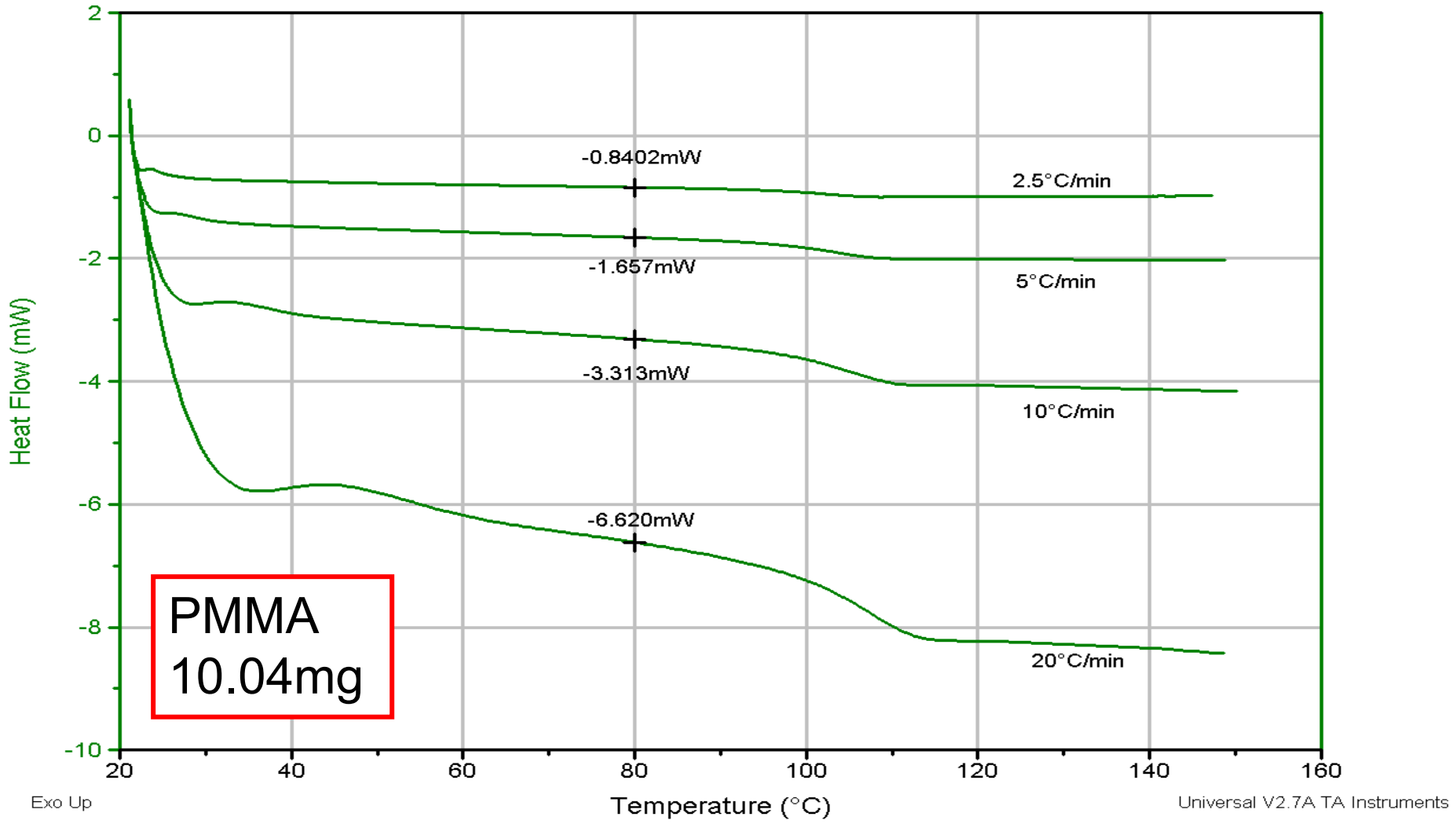
Heating Rate

- Faster heating rates increase sensitivity

  but……

- Faster heating rates decrease resolution
- **Good starting point is 10°C/min**

Effect of Heating Rate



Thermal History

- The thermal history of a sample can and will affect the results
- The cooling rate that the sample undergoes can affect :
  - Crystallinity of semi-crystalline materials
  - Enthalpic recovery at the glass transition
- Run Heat-Cool Heat experiments to see effect of & eliminate thermal history
  - Heat at 10°C/min
  - Cool at 10°C/min
  - Heat at 10°C/min