Understanding the differential scanning calorimetry of PCMs

Frequently asked questions about the operation of and preparation for the DSC of phase change materials.

By Heather Watson, Ph.D.

Q: What is DSC used to measure?

A: Differential scanning calorimetry (DSC) measures heat flow. A typical DSC curve plots heat flow versus temperature. The sample's melting and freezing temperatures as well as latent heats of fusion and freezing can be calculated from this measurement.

Q: How is heat flow measured?

A: DSC measures heat flow rate differences between a sample and an inert reference. These differences may be caused by several factors, including:

Endothermic (heat flow into the sample)	**Exothermic (heat flow out of the sample)**
Heat capacity (heating)	Heat capacity (cooling)
Melting	Crystallization
Evaporation	Curing
Glass transitions	Oxidation
Other endothermic processes	Other exothermic processes

DSC heat flow differences are dependent on heating rate, sample heat capacity (which is sample mass dependent) and heat flow at an absolute temperature. A typical DSC curve plots heat flow versus temperature. The latent heats of fusion and freezing can then be calculated by integrating the areas under the curves for the respective transitions.

Q: How are samples prepared?

151 Cheshire Lane N., Suite 400, Plymouth, MN 55441 USA
P: +952-941-0306 | F: +952-944-6893 | www.puretemp.com

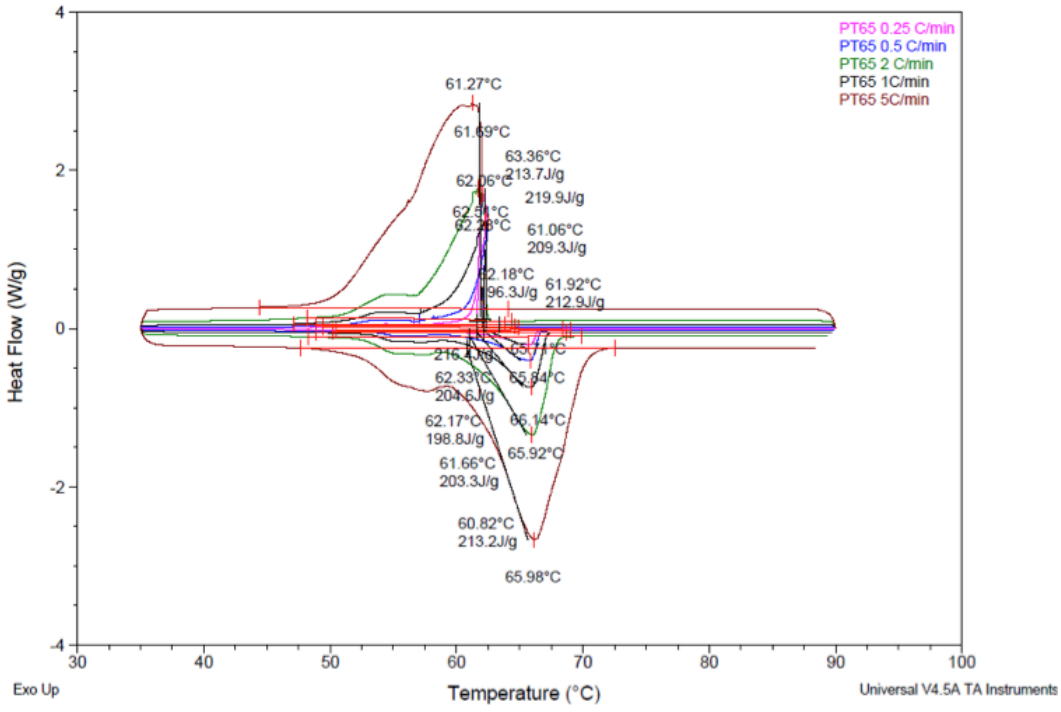
A: A disposable Pasteur pipette is used to place 3 to 6 mg of representative sample in the center of an aluminum pan. Care is taken not to get any sample around the rim of the pan so that the entire mass is contributing to the heat flow measured across the bottom of the pan. Tweezers are used to avoid touching the pan or lid with fingers.

Q: Why is a 3-6 mg sample range used?

A: A sample mass of 3-6 mg allows for the bottom of the pan to be completely covered with a layer of sample that is thin enough to allow for even heating and cooling throughout the sample during a DSC run. The goal is to achieve 0.1 – 10 mW heat flow over the transitions and increase consistency between samples.

Q: Why are hermetically sealed aluminum pans used?

A: Thermal resistance of the pan is considered in calculations. Pan mass, material, shape and thermal resistance is all factored in to measurement and calculations. A lid is used to improve thermal contact and prevent evaporation through gas stripping. Both the sample pan and reference pan masses are included in the calculation. Heat capacity of pans and heating rate as well as differences between the sample and reference pan are considered. This results in sharper taller peaks and thus better resolution and sensitivity. Care should be taken to keep the pans as flat as possible when preparing samples.



151 Cheshire Lane N., Suite 400, Plymouth, MN 55441 USA
P: +952-941-0306 | F: +952-944-6893 | www.puretemp.com

Q: Why is a 1 °C/min scan rate used?

A: DSC measures heat flow as a function of sample mass and scan rate. Slower heating and cooling rates more evenly affect the sample and lead to better resolution.

Q: What temperature range is chosen?

A: The anticipated melting temperature + 20 °C and - 30 °C is used. This ensures that the baseline is stable as transitions are approached and that unanticipated artifacts are not missed.

Q: What does a typical DSC method consist of?

A: Below is an example of a typical DSC method. A heat-cool-heat cycle is used. It is important to ensure that the baseline is stable as transitions are approached.

1. Ramp 1.00 °C/minute to 20°C above the anticipated melting point (slowly heat sample to erase possible thermal history).
2. Isothermal for 2.00 minutes (ensures entire sample has thermally equilibrated).
3. Mark cycle end 0 (marks end of heating/ beginning of cooling cycle).
4. Ramp 1.00 °C/minute to 30°C below the anticipated melting point (slowly cool sample to determine its freezing point and heat of freezing).
5. Isothermal for 2.00 minutes (ensures entire sample has thermally equilibrated).
6. Mark cycle end 0 (marks end of cooling/ beginning of heating cycle).
7. Ramp 1.00 °C/minute back up to the original high temperature. (slowly heat sample to determine its melting point and heat of fusion).
8. Mark cycle end 0 (marks the end of heating cycle and DSC method).

Q: Why is high purity N_2 at 50 mL/minute flow rate used?

A: Thermal conductivity of the purge gas is important. High purity nitrogen at a flow rate of 50 mL/minute is used for all measurements, calibrations and verifications. Nitrogen flow should be high enough that humidity is not allowed to build up in the cell and low enough that no excess noise is imparted to the baseline.

About the author

Dr. Heather M. Watson earned her Ph.D. in chemistry at the University of Alabama in 2011. As manager for analytical services, she developed the lab

151 Cheshire Lane N., Suite 400, Plymouth, MN 55441 USA
P: +952-941-0306 | F: +952-944-6893 | www.puretemp.com

methods and procedures associated with Entropy Solutions' ISO 9001 quality management system and oversees key analytical laboratory testing of all manufactured lots. She has expertise in a number of advanced analytical techniques including differential scanning calorimetry and mass spectrometry.

151 Cheshire Lane N., Suite 400, Plymouth, MN 55441 USA
P: +952-941-0306 | F: +952-944-6893 | www.puretemp.com