Tips and Hints for Improved Cp Measurements

1. Sample Form

Optimum form is a disk with at least one plan surface to ensure a good contact to the crucible bottom.
Diameter should be slightly smaller than the inner diameter of the crucible, this means

Al_2O_3 crucible:	5mm
Pt with Al_2O_3 liner, graphite:	5.5 mm
Pt	6 mm

Thickness: depending on the material and the density of the material, but as general rule a thickness of about 1mm is most often best.

If the sample is in form of a powder, you might press it to a disk with the above dimensions if possible. Otherwise ensure a good thermal contact to the crucible bottom. (For example by slightly pressing the powder by the NETZSCH forming tool into the crucible).

2. Atmosphere

The sensitivity of the DSC sample holder is dependent on the gas atmosphere. Ar delivers the highest sensitivity, followed by air, nitrogen and oxygen. The lowest sensitivity is received by using He as purge gas. This means, if possible use Ar as gas atmosphere. This ensures the highest signal gain and therefore also higher precision.

For pure inert gas atmosphere: The instrument should be vacuum-tight. To ensure oxygen-free gas atmosphere evacuate and backfill 3 times at least. Open the gas outlet only when you have a slight overpressure at the instrument and still purge gas flowing, to avoid backflow from exterior atmosphere. Gas purity should be 4.6N or better. Use oxysorb devices.

3. Crucibles

Also the crucibles influence extremely the sensitivity (signal gain). Highest sensitivity is obtained by using Pt crucibles. If you have to measure metals a good alternative are Pt crucibles with Al_2O_3 liner. They have nearly the same sensitivity. Graphite and Al have also high sensitivity.

The worst choice is alumina. Alumina crucibles have a lower sensitivity and get translucent for the radiation at higher temperature. Because of the latter effect the thermal absorption of reference and sample crucible gets different at higher temperatures and the baseline shifts.

Graphite crucibles are a good choice for Mg alloys and materials containing Si, SiC, SiN_3 etc.

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4. Furnace

The furnace should have a very uniform temperature distribution over the whole required measurement range. Also a very reproducible and stable baseline is necessary. This is given by the NETZSCH noble metal wired furnaces (Pt furnace 1500°C, Rh furnace 1650°C). Also the low temperature furnace (Kanthal wired) has the same advantages.

The SiC furnace is less suited for cp measurements. The error of the results might be in the range of +/-10% or higher.

5. Sample holder

Available DSC sensors for cp measurements are:

- High accuracy cp-DSC sample holder
- cp-DSC sample holder
- "normal" DSC sample holder

All three types are in principle ok for cp measurements, but the special designed cp-type sample holders are more accurate and should be chosen. The high accuracy cp sample holder is the best choice, of course.

Especially at high temperature cp work the type S sensor (Pt/PtRh10% thermocouple) is the best choice. When working below RT or up to 600°C a type E (NiCr-Konstantan thermocouple) can/must be used. Please note there are only "normal" DSC type sample holders available. Because of the high signal gain (type E) and the heat transport is mainly by convection (radiative transport is very small) this sample holder construction is fine for excellent cp results.

6. Temperature program

The temperature program should look like following:

Start temp (i.e. RT)
Heating to an isothermal segment (i.e. 5°C higher) with 5 K/min
Isothermal for 10 min
Heating to end temperature
Isothermal at that temperature for 10 min

With this temperature program you can use the evaluation by the ratio method and the ASTM E 1269 method.

Note: It is also possible to calculate the cp during cooling. This means a cooling curve can be added and used for evaluation, too.

Important: switch off the STC option for all segments

7. Heating rate

Normally heating rates of 20 and 10 K/min were used. 5 K/min is also possible. The lower the heating rate, the lower the signal gain per time. This means also the signal to noise ratio increases with very small heating rates. This can affect a lower accuracy of the cp results (remember you need three stable curves for the calculation: baseline, calibration, sample). Best choice: 20 K/min

8. Cleanness, Avoiding Sticking of Pt crucibles

Pt crucibles tend to stick on type S DSC sensors at higher temperature. There are several reasons:

a) not conditioned sample holder and/or crucibles (heat them separately to 1500°C)
b) the crucible was sitting on a bench with some dirt/dust (inorganic particles which melt)
c) metallurgical effect (diffusion because of different alloys of crucible and sample holder)

Avoiding or minimizing by:
Lift the crucibles after every measurement. Don't forget to lift also the reference crucible.

Use sapphire spacers on the sensor plates. For example the sapphire standards from NETZSCH with 0.25 mm thickness. This is especially a good idea when you exceed an end temperature above 1400°C.

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