How To
Measure modulated C$_P$
with Chip-DSC
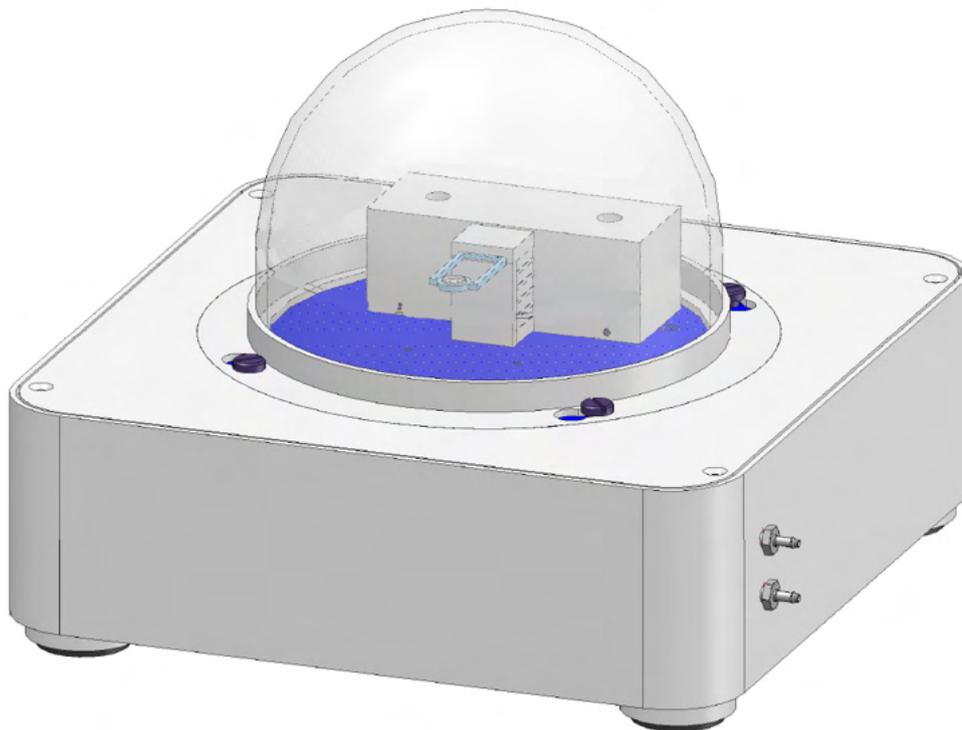
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1. Prepare temperature modulation function

1.1 Administration

- o   Make sure you have a Dongle for Chip DSC and it is connected to your PC

- o   If the temperature modulation function is not unlocked, please contact Linseis Support

Tips and Tricks:

- o   *The more lightweight the crucible (if needed at all), the better your results get*
- o   *The sample is needed to be placed in the mid of the crucible and the crucible must be in the mid of the sensor*
- o   *Doing two or more runs give the option to choose the best result*
- o   *The sample and the bottom of the crucible must be very flat and have good contact to the sensor*
- o   *If possible run modulated measurements with same conditions and settings so you do not need to run a correction curve all the time*
- o   *5 – 10 K/min heating rate | 1 K Amplitude | 60 s Period are recommended settings*
- o   *If the temperature range is too small or the heating rate to high you should lower amplitude and period to get enough measured periods to receive good results*
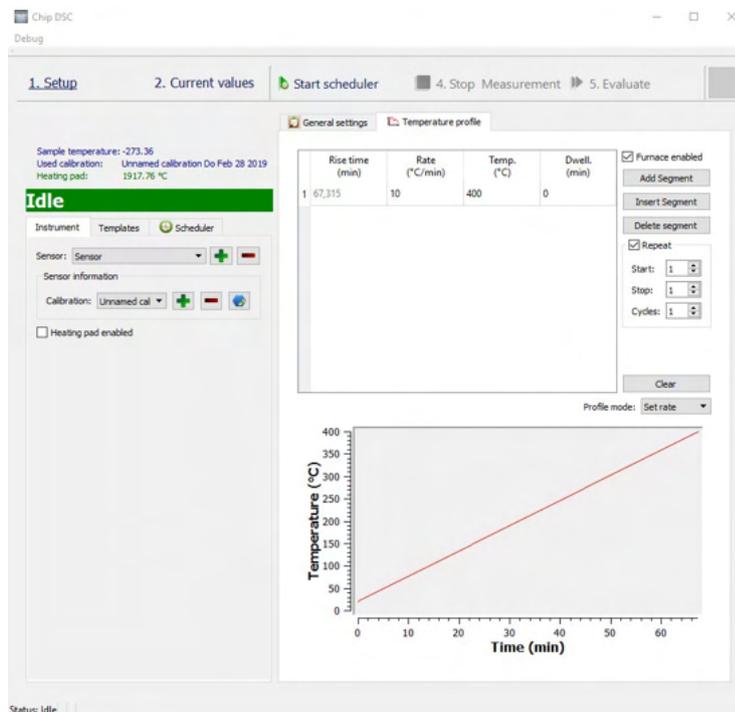- o   *Use the preheat function to get the most reproducible results*

2. Measuring

2.1 Measuring Software

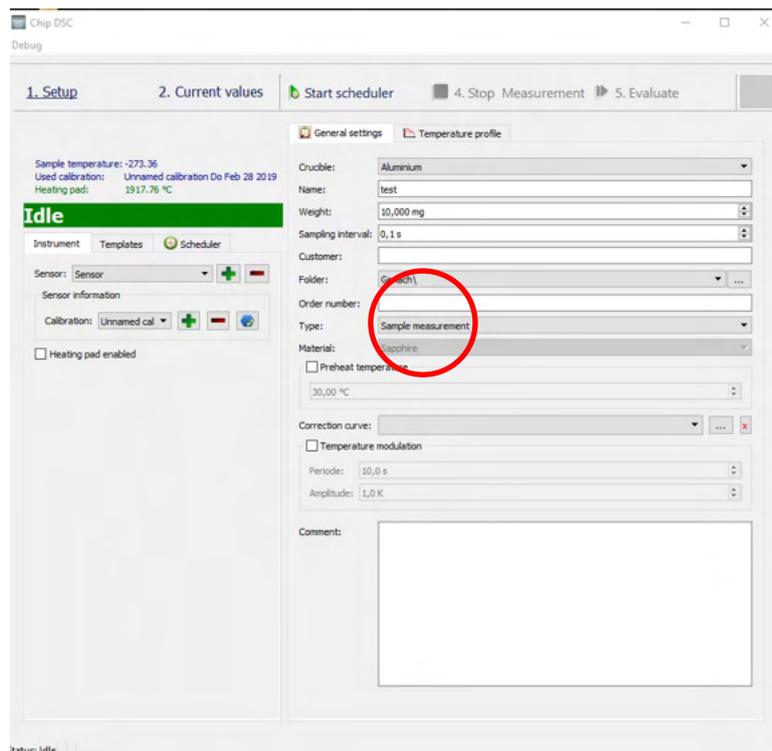o Open the Chip DSC measurement software and enable the temperature modulation
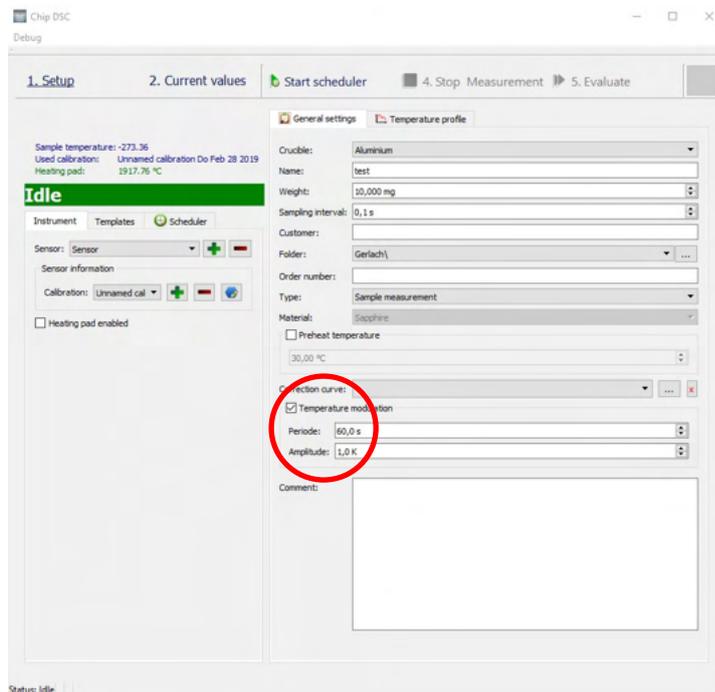
o For Period 60s and Amplitude 1K are recommended (depends on measurement conditions)

o Set the Temperature profile you want to measure

o For heating rate 5 – 10 K/min are recommended

o Now run the measurements

- o At first decide if you want to use a crucible or not (you should use the same crucible for every needed measurement)
- o Setting a Preheat temperature is recommended
- o Select Correction (Type) and run the measurement without any sample on Sensor/in crucible (insert any Weight it is not evaluated in the correction)
- o Select Calibration (Type) and run the measurement with the material you want to use for the calibration (insert the weight as precise as possible)
- o A typical material used for calibration is a sapphire disk. Note different thickness disks can offer the ability to best match the thermal mass of your Cp investigation material.
- o Select Sample (Type) and run the measurement with the sample material you want to measure (insert the weight as precise as possible)
- o For every measurement: make sure all samples and the crucibles have good contact to the bottom and lay in the centre of the sensor / crucible!

3. Evaluate

3.1 Evaluation Software

- o If all measurements are done start the evaluation software and load the Calibration and sample curve (RAW – signal over time)
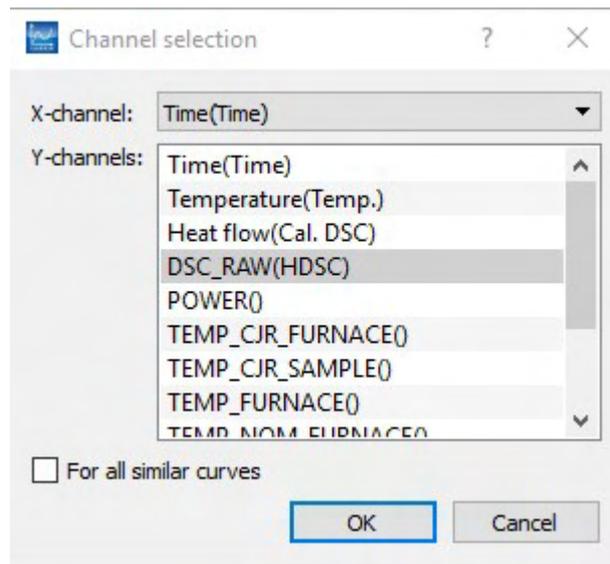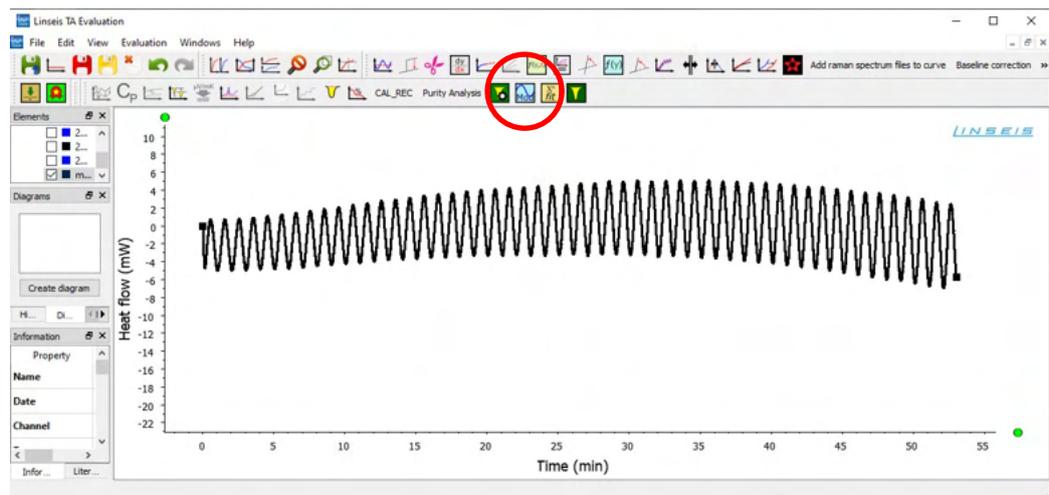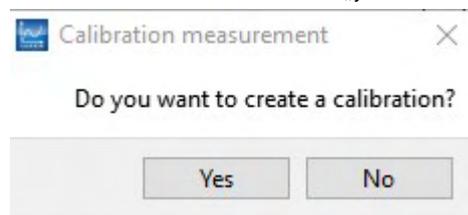
Note you will be asked if you want the zero correction at this time...Say No and click past this request. Another opportunity will present itself later in the process.

3.2 Calibration

    o   Select the calibration curve
    o   Click on the modulation – button

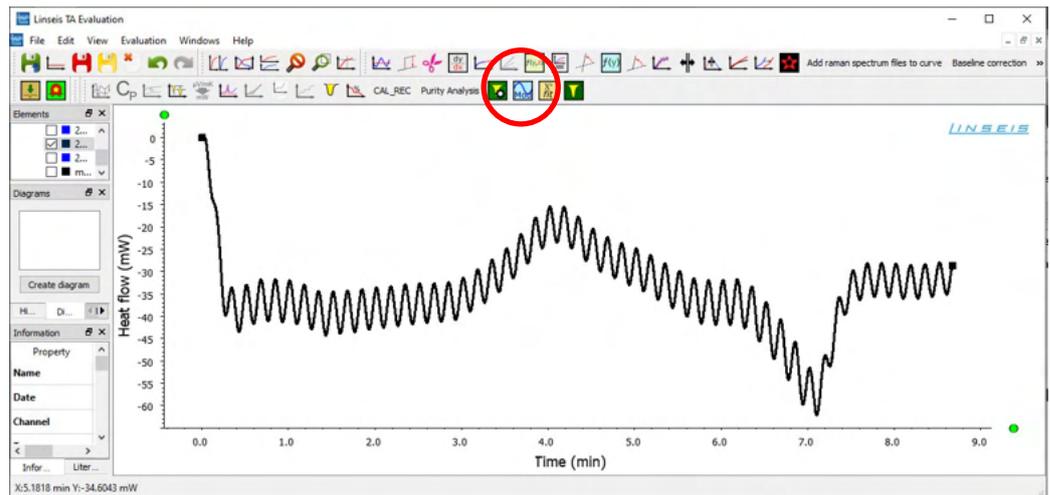   o   In the selection window click „yes"

    o   Choose the right correction curve
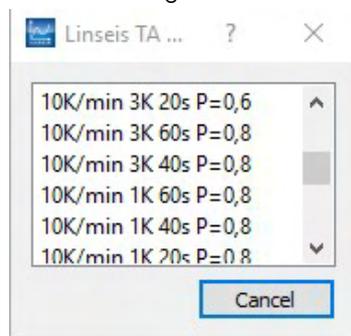   o   Give your calibration a name

3.3 Sample evaluation

    o   Select your sample curve
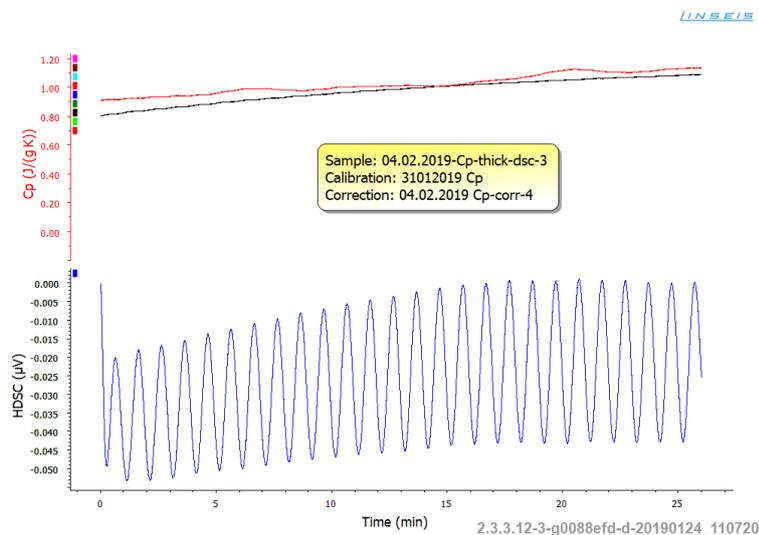   o   Click on the modulation – button

- o Choose the right correction curve (the same from 3.2)
- o Choose the right C$_P$ – calibration and double click on it
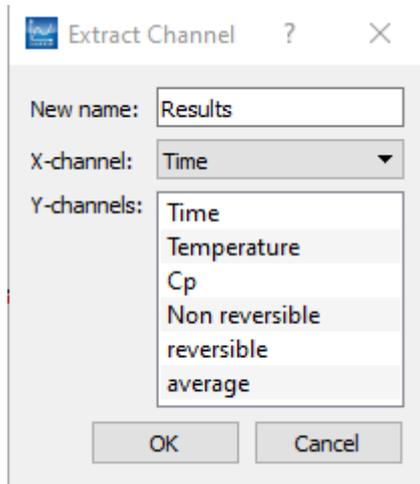

3.4 Finish the evaluation

- o Now you have your C$_P$ value of the sample
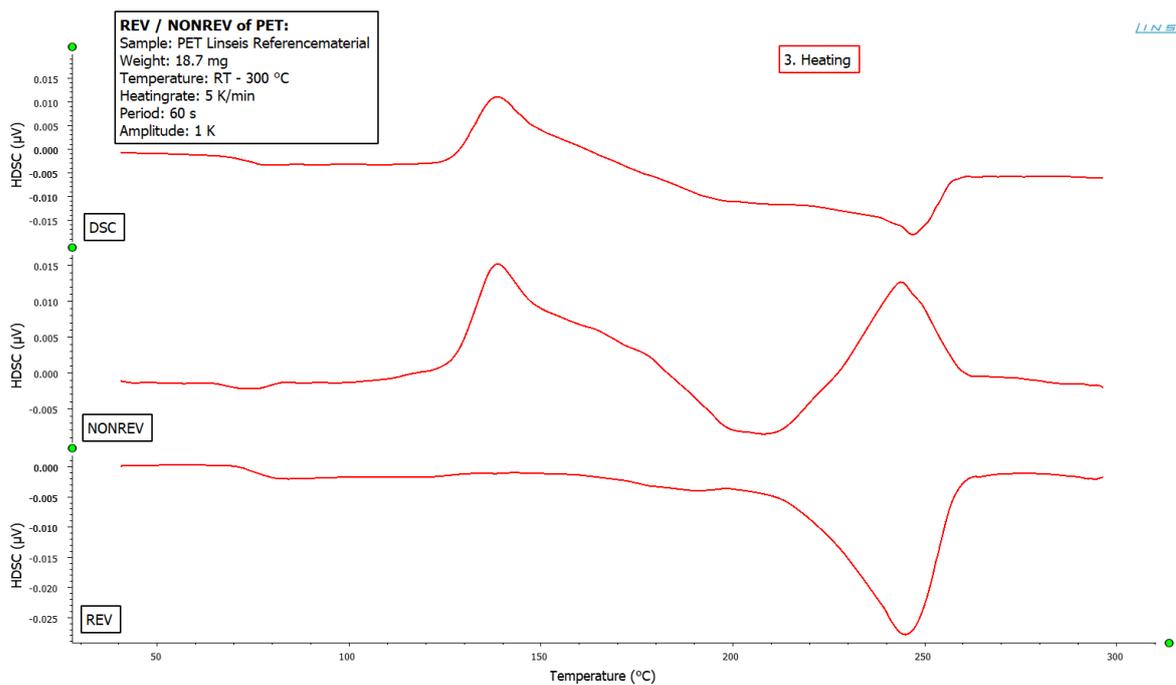- o You can modify your results in the evaluation tool

4. Reversing / Nonreversing parts

o   You can split your signal into reversing and nonreversing parts of the DSC signal
o   Select your evaluated Cp measurement
o   Duplicate your Cp curve (rightclick)
o   Choose the channels you want to see

Extract Channel ? ✕

New name: Results
X-channel: Time ▾
Y-channels:
  Time
  Temperature
  Cp
  Non reversible
  reversible
  average

OK   Cancel

o   Cp   → specific heat capacity of your signal
o   Non reversible   → non reversing transitions
o   Reversible   → reversing transition
o   Average   → DSC-signal without modulation
o   Evaluate the signals to your needs

REV / NONREV of PET:
Sample: PET Linseis Referencematerial
Weight: 18.7 mg
Temperature: RT - 300 °C
Heatingrate: 5 K/min
Period: 60 s
Amplitude: 1 K

3. Heating

DSC

NONREV

REV

Tips:

- o Good Cp data strongly relies on good repeatability of correction, calibration and sample runs.
- o Run all types of measurements at least 5 times to demonstrate repeatability. If the scheduler is used allow for enough time after cooling before starting the next run.
- o Use only the heating data for Cp measurement. If data was recorded during cooling, use the split heat/cooling tab to separate.
- o Drop any outliers and create a mean curve for the correction and calibration using the mean tab.
- o Also run the calibration material as a sample and compare Cp value with literature value that can be added to the plot using the literature value tab. Lastly, lots of data demands good organization...After each evaluation create a new project for each type of data set.