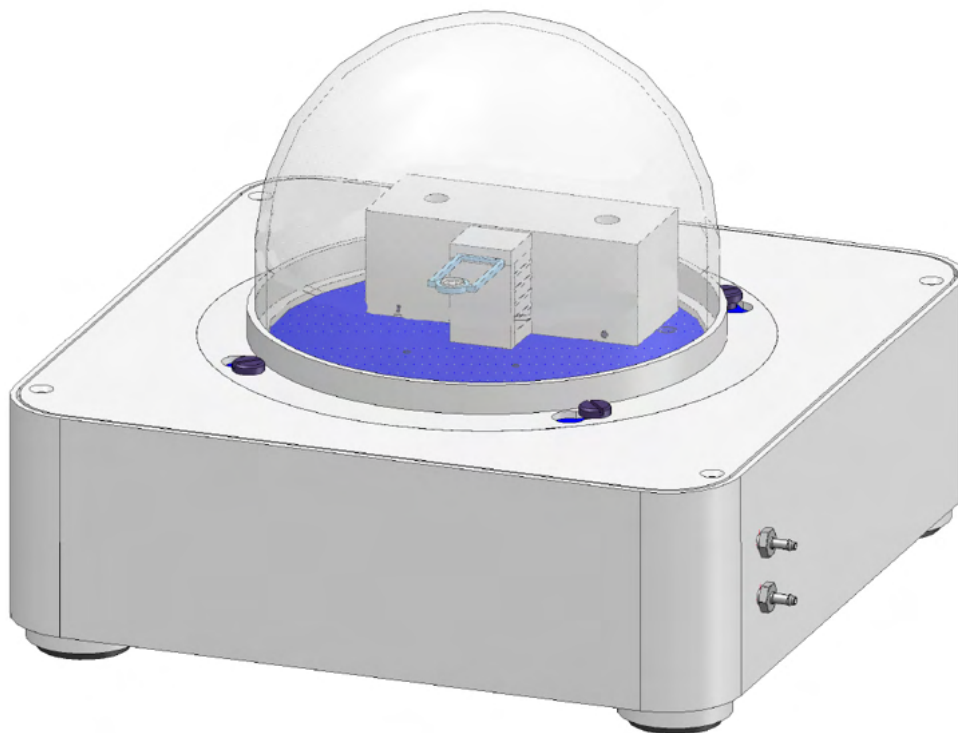


How To calibrate with Chip-DSC



Linseis Messgeräte GmbH
Gerlach
Date: 18.12.2023

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How To calibrate with Chip DSC

1. General information

This manual is a short description for doing calibrations with the Chip-DSC. The presented features are the most common, that are usually used. However, there are much more features that cannot all be displayed here. For more Information, read the other available instructions about software or specific manuals for the Chip DSC.

2. Prepare the device and software

2.1 Requirements

- Make sure your device is connected and complete
- Make sure LINSEIS Platinum software is installed and additional features (e.g. cryo mode) are unlocked by dongle
- Make sure you have all calibration materials you need for the desired temperature range
- Make sure all accessories for e.g. low temperature are available

2.2 Setup your device and PC

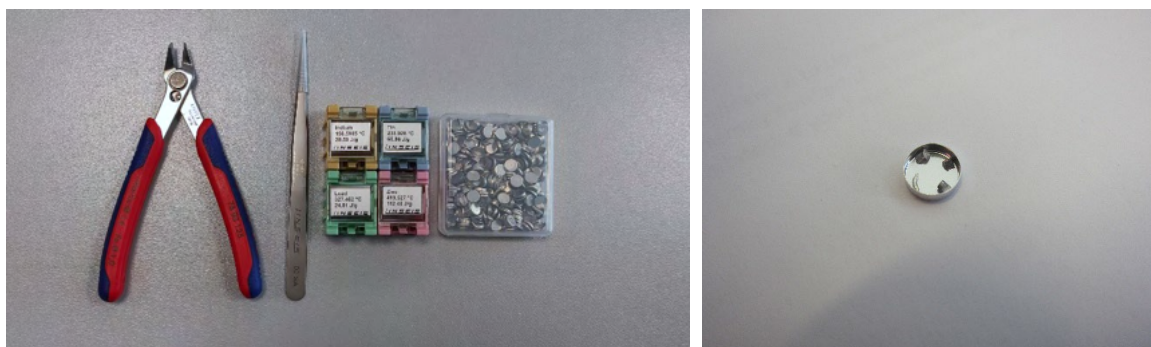
- Turn on your PC and connect the Chip-DSC device
- Turn on your Chip-DSC and start the measurement software
- Select a new Sensor if needed or choose the right one from the dropdown menu

3. Run a high temperature calibration

3.1 prepare a sample

- Select the right crucible for your measurement
- Select the right calibration standards for the desired temperature range
- Cut the samples, weigh them and place them in your crucible

3.2 insert your

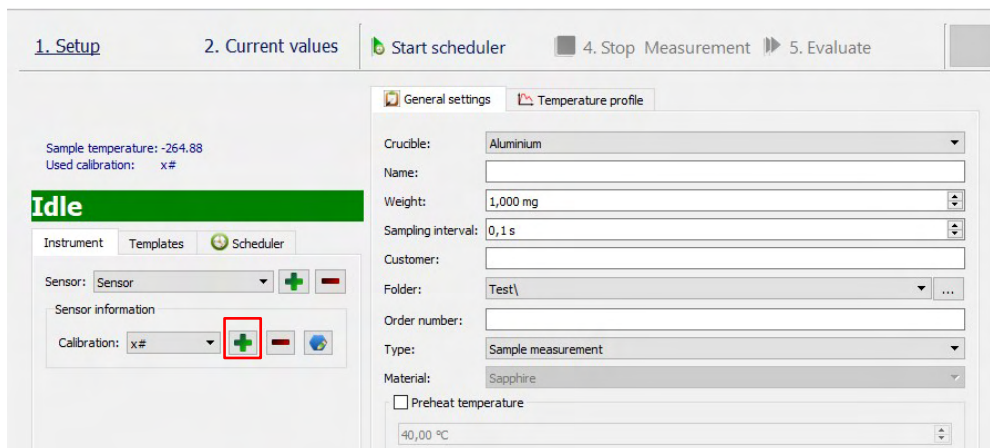


sample

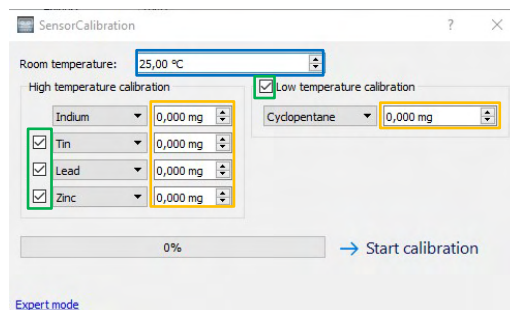
- Place the crucible in the middle of your sensor and make sure the samples do not touch each other. Use tweezers to separate the calibration standards if necessary so they do not touch.
- Close the glass dome of your Chip-DSC

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3.3 setup your calibration in easy mode



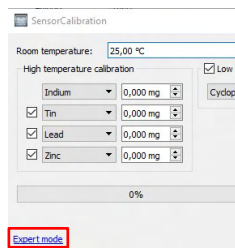
- Click on the “+” in the Acquisition software to create a new calibration □



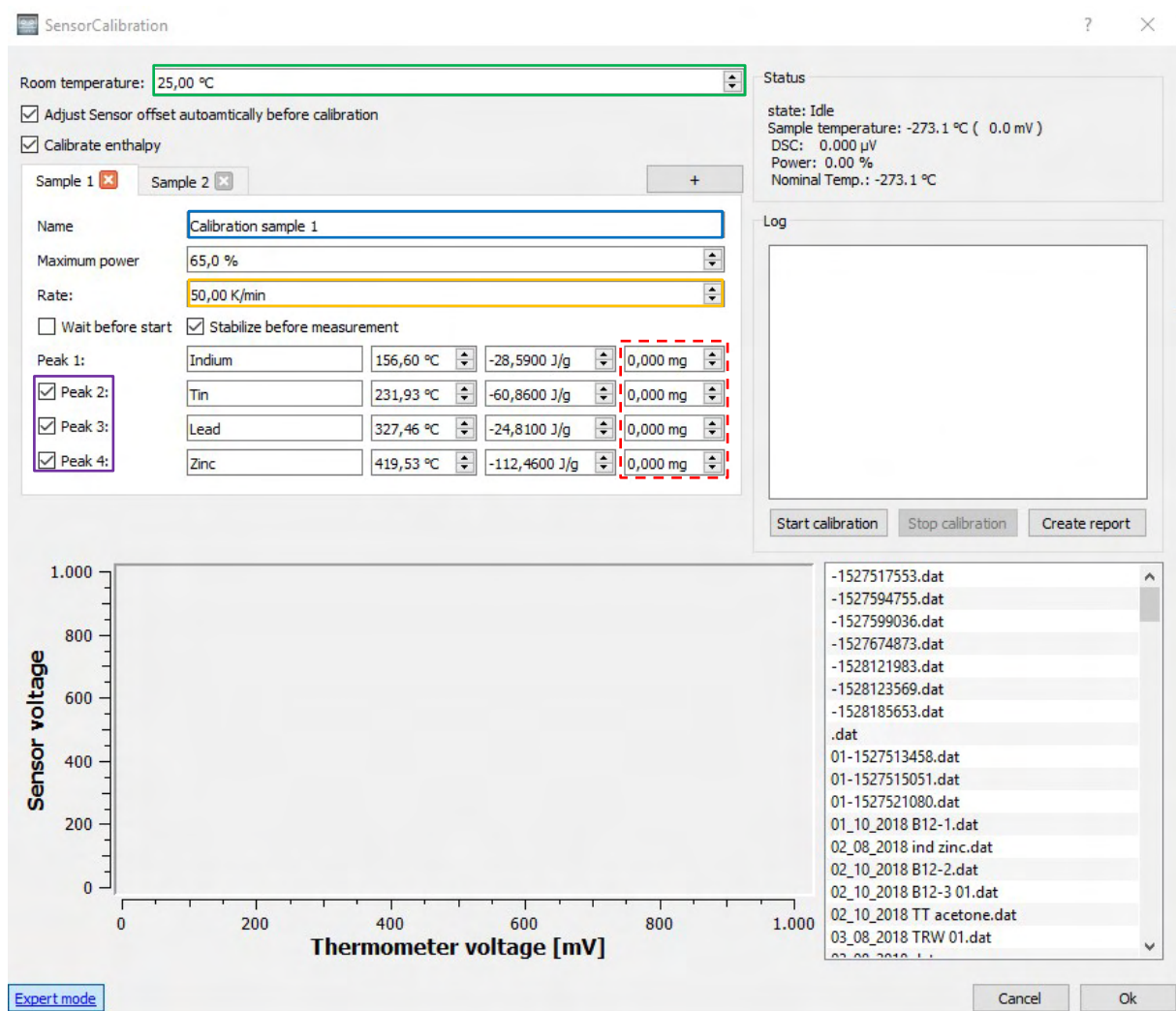
- Select the calibration standards you want to use for the calibration. □
- Measure the room temperature using a secondary laboratory thermometer at the DSC-Sensor and enter the temperature value in the software field. □
- Enter the correct weight for your samples in the software fields. □

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3.4 setup your calibration in expert mode



- In easy calibration mode click on “expert mode” in the lower left corner

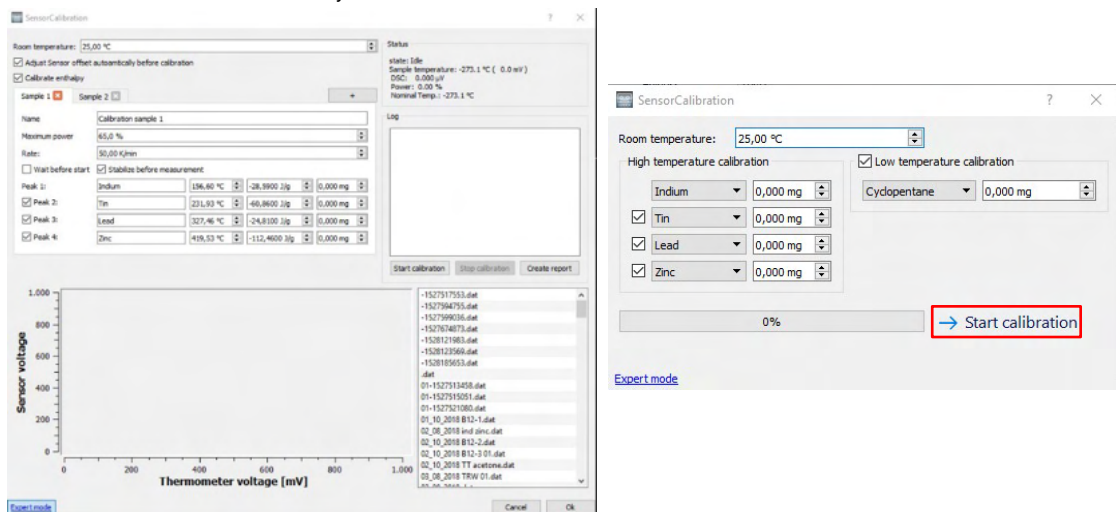


- Measure room temperature at the DSC-sensor and insert the right value in the field.
- Give your calibration run a name
- Set the heating rate you want to use for the calibration. Note the same rate has to be used for the Sample run.
- Select the calibration standards you want to use
- Insert the right weight for your samples
- Adjust other Settings if necessary

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3.5 Run and finish your high temperature calibration

- Click on “Start calibration” in your calibration window



- Wait until the calibration finished



- In the first message popup insert the name for your calibration curve

| | Sample | Real temperature | Measured voltage | Calibration factor |
|-------------------------------------|----------|------------------|------------------|--------------------|
| <input checked="" type="checkbox"/> | Sample 1 | 156.6 °C | 125.573 mV | 563.246 mW/(mV*s) |
| <input checked="" type="checkbox"/> | Sample 1 | 231.9 °C | 147.371 mV | 575.513 mW/(mV*s) |
| <input checked="" type="checkbox"/> | Sample 1 | 327.46 °C | 175.674 mV | 756.961 mW/(mV*s) |
| <input checked="" type="checkbox"/> | Sample 1 | 419.53 °C | 199.015 mV | 704.278 mW/(mV*s) |

- Check the values of your calibration to see if everything worked well. If not try again without touching the calibration standards (the shape of your samples is better after first run since the sample makes better contact with the crucible.)



- Click “Ok”

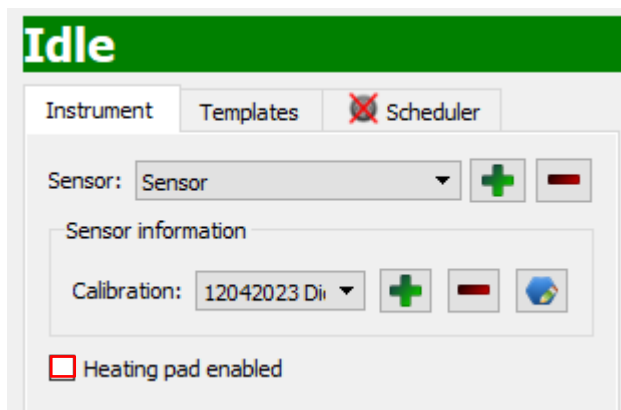


- Insert the name for the saved calibration

4. Run a low temperature calibration

4.1 prepare a sample

- Select the right crucible for your measurement
- Select the right calibration standard for the wanted temperature range
- Fill in your sample with syringe (only if you use crimpable crucible, otherwise wait until 4.4 finished) and close crucible
- Turn on Heating Pad in General Settings

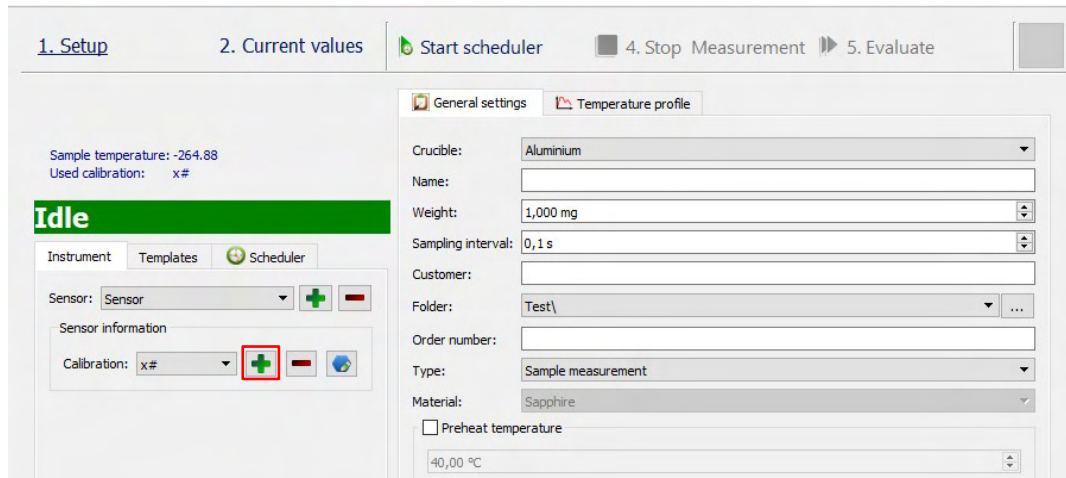


Tips and Tricks:

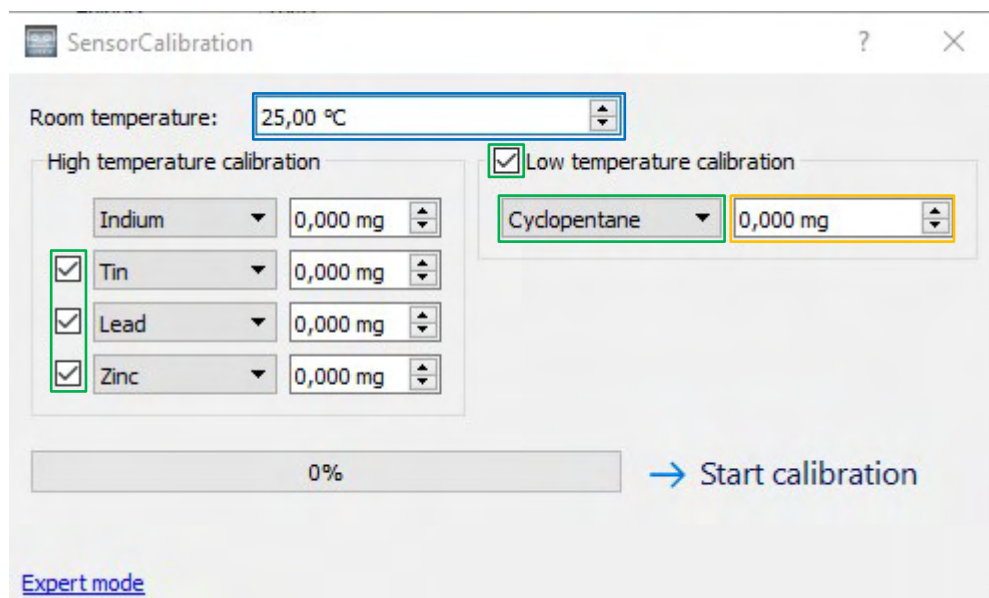
- *The first sample is always active*
- *For enthalpy calibration minimum two melting points are necessary*
- *To calibrate samples one by one in separated crucibles you can repeat calibration runs or add samples by clicking "+" in expert menu*
- *In case you want to use the Linseis Calibration Disc check the specific HowTo*

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4.2 setup your device in easy mode



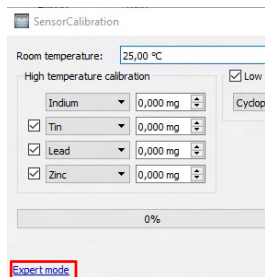
- Click on the “+” to create a new calibration



- Select the calibration standard you want to use for the calibration.
- Measure the room temperature at the DSC-Sensor and insert the right value
- Insert the right weight for your sample

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4.3 setup your device in expert mode



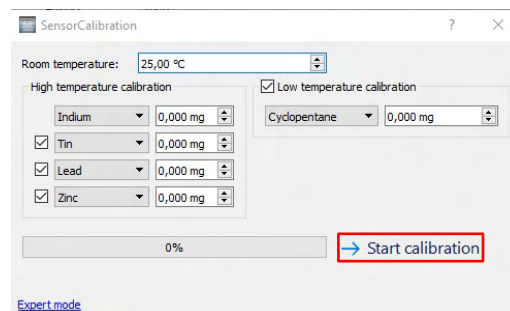
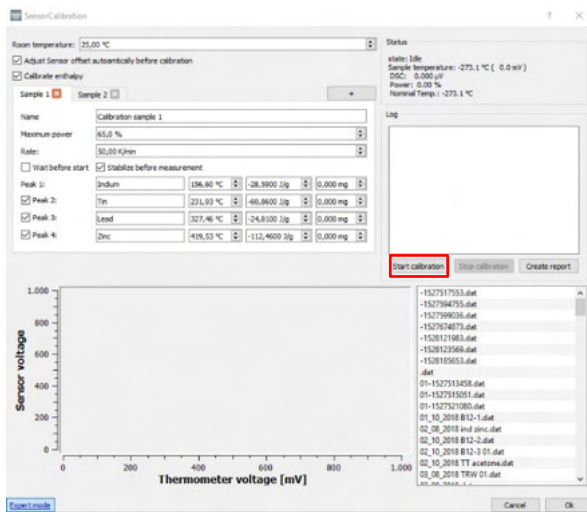
- In easy calibration mode click on “expert mode” in the lower left corner

- Measure room temperature at the DSC-sensor and insert the right value
- Give your calibration curve a name
- Set the heating rate you want to calibrate with
- Select the calibration standard you want to use and insert the right values
- Insert the right weight for your sample (if you want to calibrate enthalpy)
- Select “Wait before start”
- Unselect “Stabilize before measurement” and “Calibrate Enthalpy” (depends on your needs)
- Limit “maximum power” to 1% - 2%

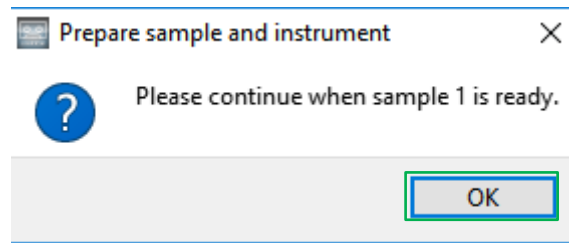
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4.4 Run the calibration and go on with sample

- Click on “Start calibration”



- Wait until room temperature measurement and sensor adjust is done



- When window “continue when sample *samplename* is ready” appears start cooling
- If the device is cold enough insert the sample in your crucible and put the crucible on the sensor.
- Make sure sample is frozen
- If Temperature is 20 K – 30 K below onset temperature of your sample wait until LN2 is gone (if you use quenching option)
- In the “Continue when sample *samplename* is ready” window click Ok

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4.5 finish your low temperature calibration

- Wait until the calibration finished



- In the first message popup insert the name for your calibration curve
- Check the values of your calibration if everything worked well. If not try again
- Click "Ok"



- Insert the name for the saved calibration

Tips and Tricks:

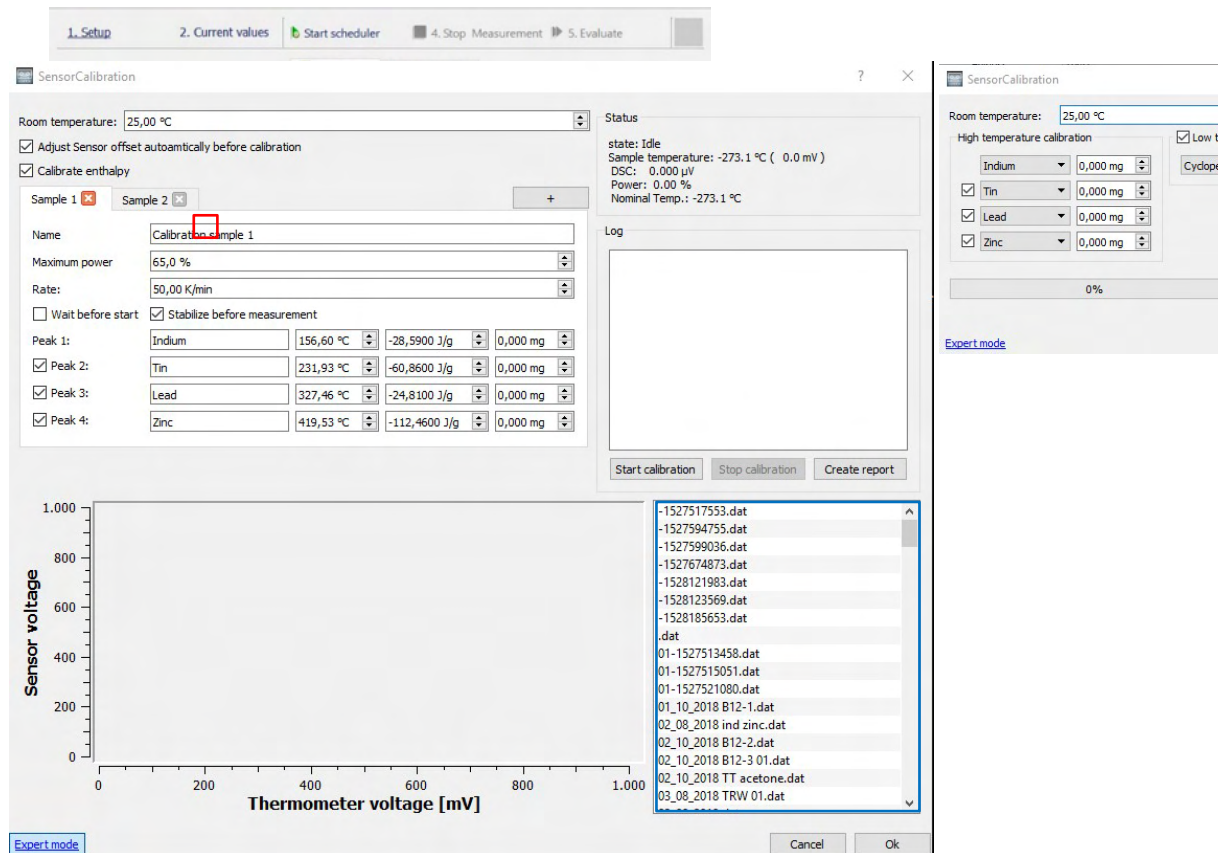
- *Low temperature calibration starts always at room temperature without cooling*
- *In case of not hermetically sealed crucible enthalpy calibration can be turned off due to inaccuracies caused by sample evaporation*
- *For liquids its just possible to calibrate materials one by one*
- *For enthalpy calibration minimum two melting points are necessary*
- *During cooling it is highly recommended to enable Heating Pad to avoid condensation inside the device*

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5. Find and select your calibrations

5.1 Select your (multiple) calibrations

- Click on “+” in the acquisition software to create a new calibration



- Click on “Expert mode” in the lower left corner
- Select the calibration you want to use in the right window
- If you want to use more than one calibration curve select multiple calibrations by pressing “Ctrl” and select the calibrations you want
- Right click on one of your wanted calibrations and select load

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Room temperature: 26,00 °C

Adjust sensor offset

Adjust Sensor offset automatically before calibration

Status

Calibrate enthalpies

Peak selection

| Sample | Real temperature | Measured voltage | Calibration factor |
|--|------------------|------------------|--------------------|
| <input checked="" type="checkbox"/> Sample 1 | 156.6 °C | 125.573 mV | 563.246 mW/(mV*s) |
| <input checked="" type="checkbox"/> Sample 1 | 231.9 °C | 147.371 mV | 575.513 mW/(mV*s) |
| <input checked="" type="checkbox"/> Sample 1 | 327.46 °C | 175.674 mV | 756.961 mW/(mV*s) |
| <input checked="" type="checkbox"/> Sample 1 | 419.53 °C | 199.015 mV | 704.278 mW/(mV*s) |

OK Cancel

Stop calibration

Sensor voltage

Thermometer voltage [mV]

1529504343.dat
1529505803.dat
1529913037.dat
1530016242.dat
1530019805.dat
1530022847.dat
1530080333.dat
1530081086.dat
29_06_2018.dat
30_07_2018 japan.dat
31_07_2018 02.dat
31_07_2018 03.dat
31_07_2018 06.dat
31_07_2018 07.dat
31_07_2018 B7-6.dat
B7-1.dat
B7-2.dat

Ok Cancel

- Select the onset temperatures you want to use
- Click "OK"
- Select the enthalpies you want to use
- Click "OK"
- Click "OK" in the calibration window

New Sensor ? X

Name:

OK Cancel

- Insert a name for the saved calibration

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5.2 Find your calibration on your PC

- Open your Data explorer (WIN + E)

| | | |
|-----------------|------------------|-------------|
| ChipDSC | 14.01.2020 11:22 | Dateiordner |
| driver | 04.12.2019 10:57 | Dateiordner |
| EDSC | 04.12.2019 10:57 | Dateiordner |
| Firmware | 11.12.2019 16:14 | Dateiordner |
| Log | 04.12.2019 10:57 | Dateiordner |
| manuals | 04.12.2019 10:54 | Dateiordner |
| ReportTemplates | 04.12.2019 10:54 | Dateiordner |
| SetupFiles | 14.01.2020 11:47 | Dateiordner |
| Temp | 15.01.2020 12:08 | Dateiordner |
| ThermalLibrary | 04.12.2019 10:57 | Dateiordner |

- Go to your Linseis data directory (normally C:/Linseis)
- Go to C:/Linseis/EDSC or C:/Linseis/ChipDSC (depends on version)
- Here you find the .dat-files of your calibration curves