NETZSCH

Starting a Measurement and Data Evaluation of a Cp Measurement

www.netzsch-thermal-analysis.com

Carrying Out Measurements

Values from three different measurements are required for the calculation of the specific heat:

- baseline
- standard
- sample

Within these series of measurements, the following test parameters must be identical:

- atmosphere (purge gas)
- flow rate
- initial temperature
- heating rate and scanning rate (sampling interval)
- mass of crucible and lid
- crucible position on the sensor

General Comments

✓ If possible, carry out **all three measurements** of a series in immediate succession **on the same day**.

✓ **Several samples** with the same test parameters can also be **combined** with the same pair of measurements for baseline and standard.

✓ **Identical initial conditions** must be guaranteed for the measurements of a series. The entire measuring system must be at a **uniform, stable temperature level**.

✓ Start with the **controlled heating** not at the present room temperature, but rather **at a significantly higher temperature**, e.g. 40°C (only for DSC 404 C).

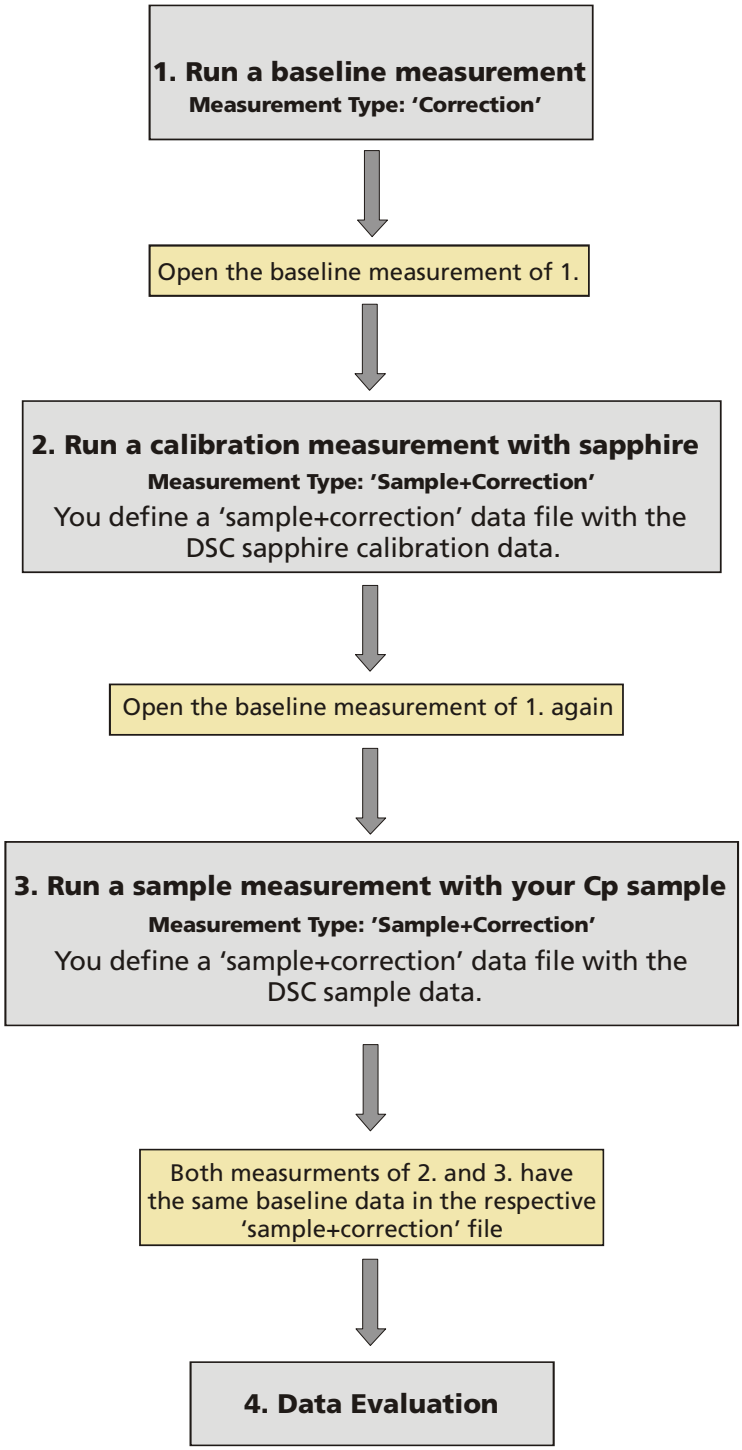
✓ To equalize the temperature in the system, **define a constant segment** prior to starting the heating phase (approximately 15 minutes).

✓ If possible, **use the same crucible and lid** for all three individual measurements.

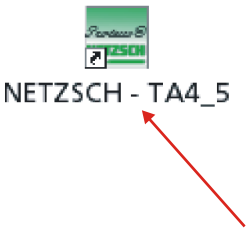
✓ **Heat** the crucibles and lids prior to the measurement.

✓ When selecting the table of standard values (C_p Analysis), **be sure** that the calibration measurement and the table are based on the **same calibration material**.

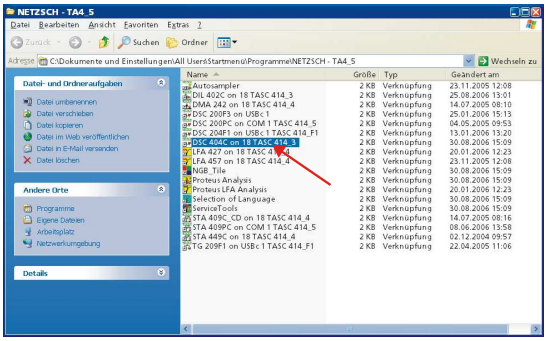
Starting a Measurement



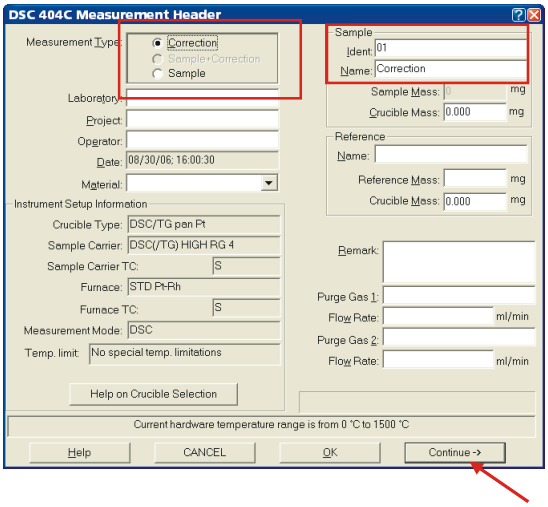
1. Run a Baseline Measurement



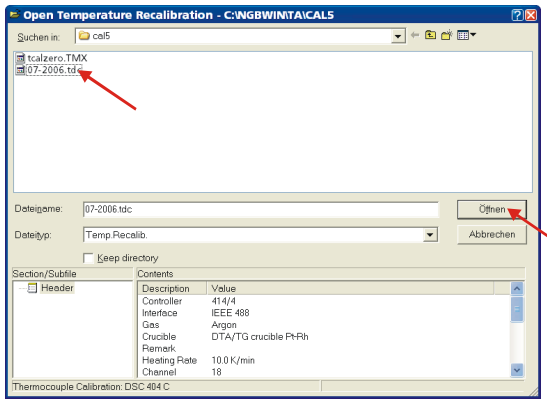
- Open the NETZSCH TA4_5 group.



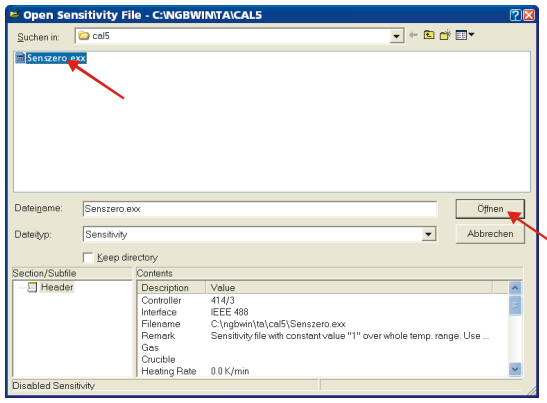
- Select your respective instrument (e.g. DSC 404 C).



- Select Measurement Type **Correction**.
- Define **Ident** and **Name**.
- Click **Continue**.



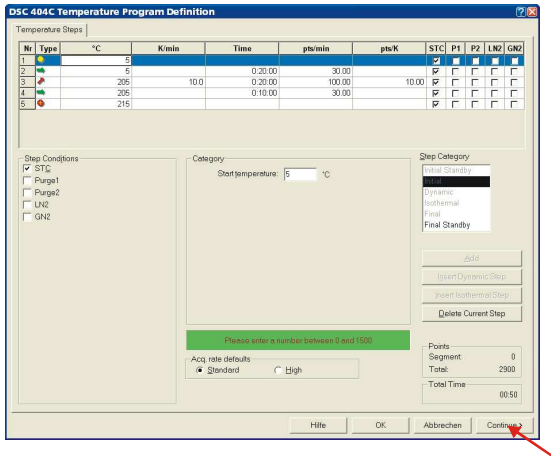
- Select the **temperature calibration** file.
- Continue with **Open**.



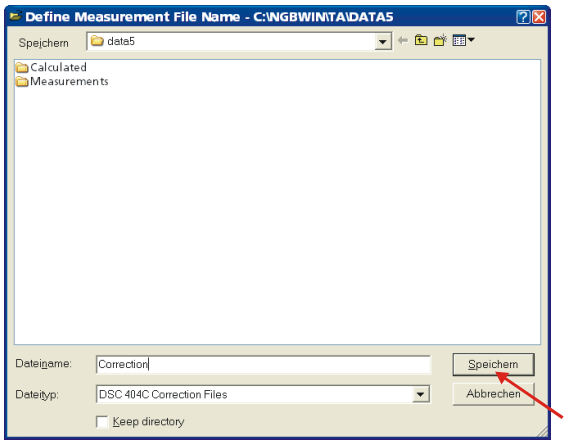
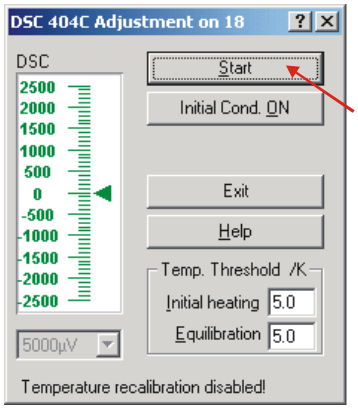
- Select the **sensitivity calibration** file **Senszero.exx**.
- Continue with **Open**.

NOTE

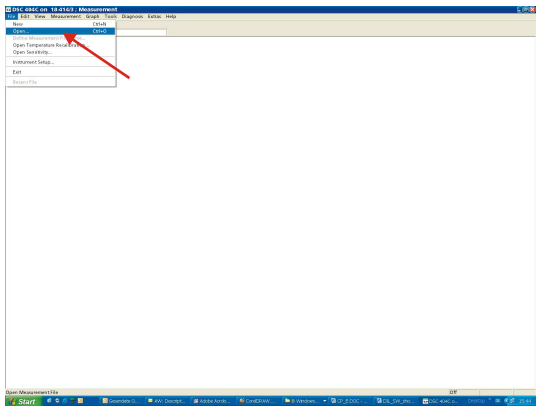
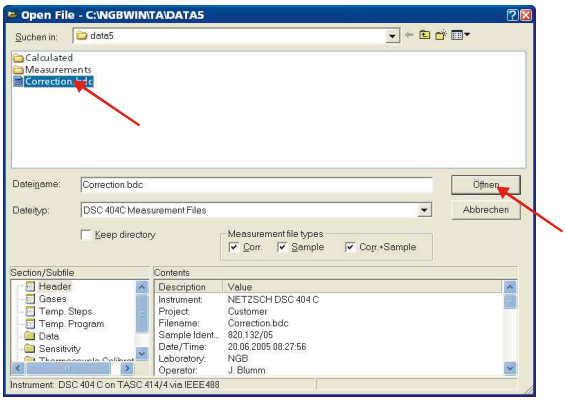
For pure Cp evaluation, no sensitivity calibration is necessary.

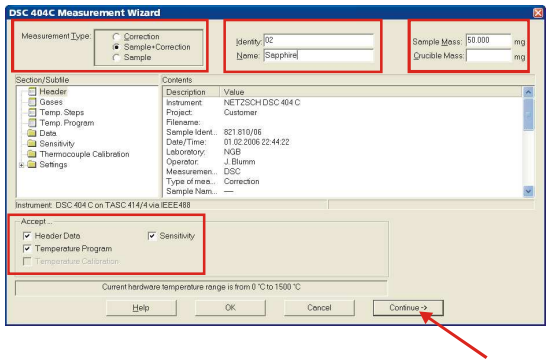


- Define the temperature program for the measurement (an example of a temperature program is shown in the figure).
- Click **Continue**.

	- **Define** a **file name** and **save** the **measurement parameters**.
	- **Start** the **measurement**.

2. Run a Calibration Measurement with Sapphire

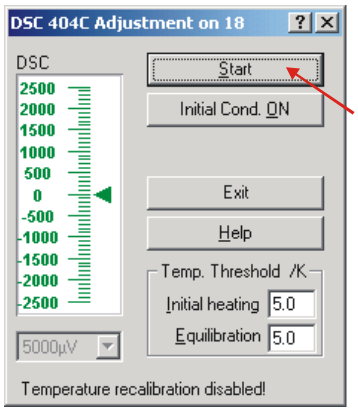
	- After the baseline measurement is finished, select **Open** from the **File** menu.
	- Select the baseline measurement (carried out in section 1). - Continue with **Open**.



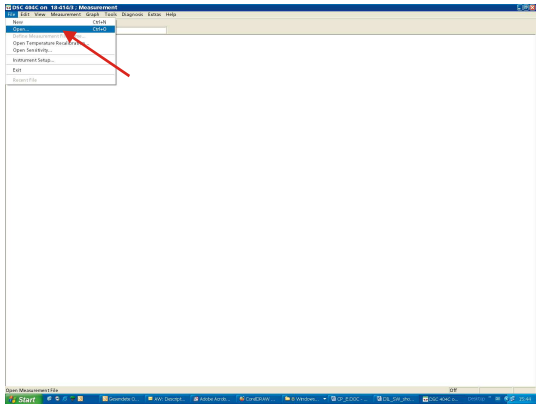
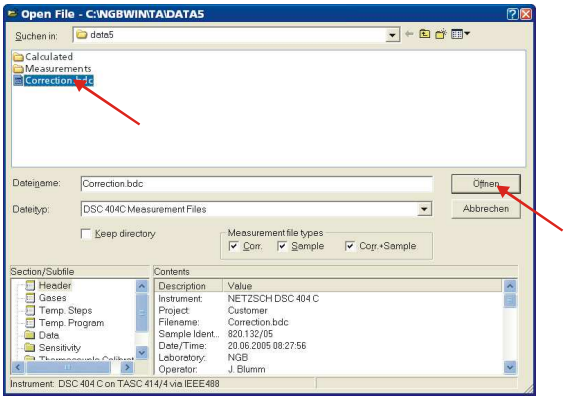
- Select **Sample + Correction** (the sample measurement is thus corrected with the baseline measurement).
- Define **Ident** and **Name.**
- Enter the **sapphire sample mass** (mass of the sapphire disc). Crucible mass is optional, reference crucible is empty.
- In order to define the same settings as for the baseline measurement (e.g. temperature program), set the check marks for Header Data, Temperature Program and Sensitivity.
- If no check mark is set, you can define a new temperature program.
- Please note that the temperature program settings must be identical to the temperature program settings of the baseline measurement.
- Click **Continue**.

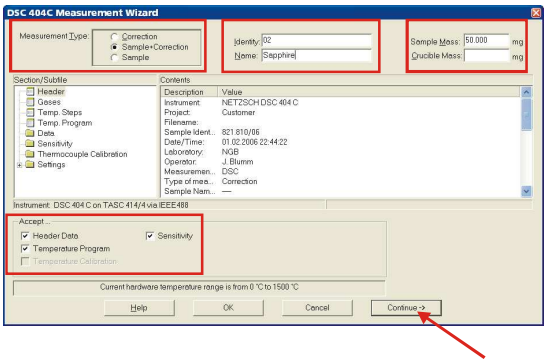
- Select the **temperature calibration** file (the same file as for the baseline measurement).
- Continue with **Open**.

- **Define** a **file name** and **save** the **sapphire measurement parameters**.

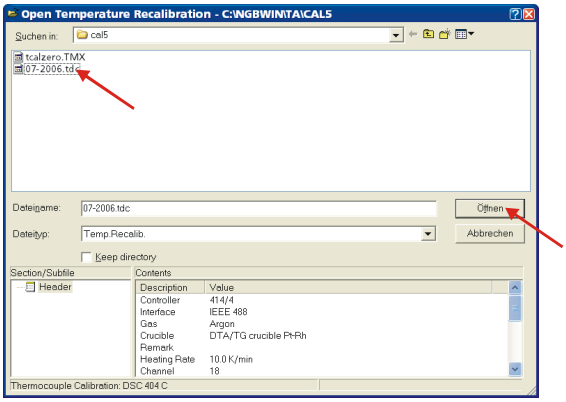
	- **Start** the **measurement**.

3. Run a Sample Measurement with your Cp Sample

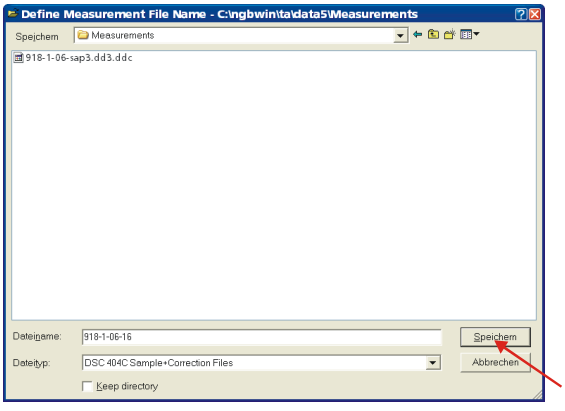
	- After the sapphire measurement is finished, select **Open** from the **File** menu.
	- Select the baseline measurement (carried out in Section1). - Continue with **Open**.



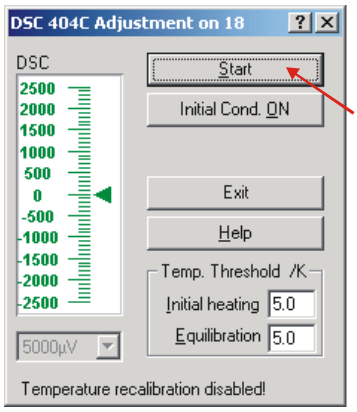
- Select **Sample + Correction** (the sample measurement is thus corrected with the baseline measurement)
- Define **Ident** and **Name.**
- Enter the **sample mass** of the **Cp sample**. Crucible mass is optional, reference crucible is empty.
- In order to define the same settings as for the baseline measurement (e.g. temperature program), set the check marks for Header Data, Temperature Program and Sensitivity.
- If no check mark is set, you can define a new temperature program.
- Please note that the temperature program settings must be identical to the temperature program settings of the baseline measurement.
- Click **Continue**.



- Select the **temperature calibration** file (the same file as for the baseline measurement).
- Continue with **Open**.

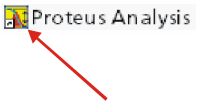
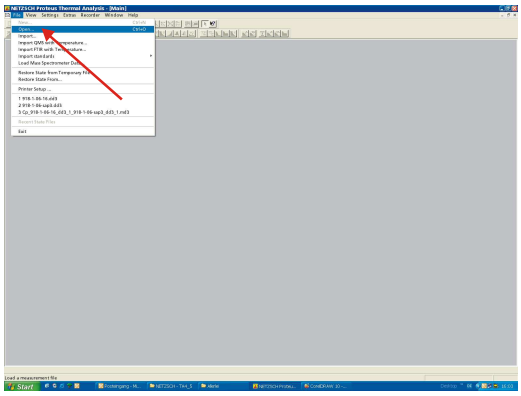
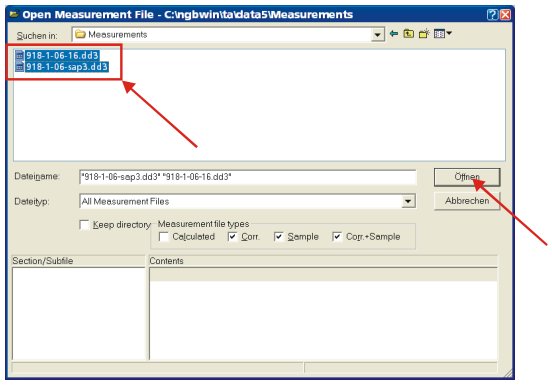
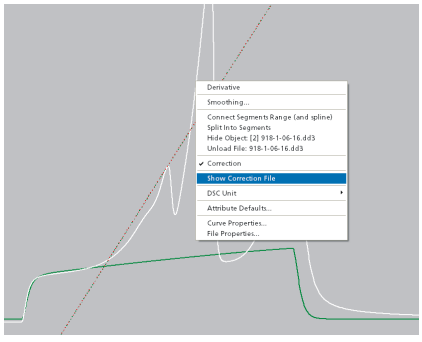


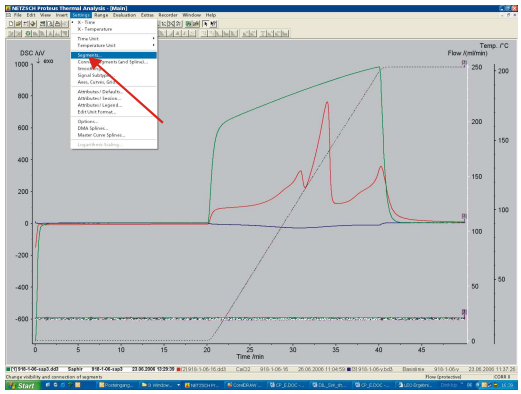
- **Define** a **file name** and **save** the **sample measurement parameters**.

	- **Start** the **measurement**.

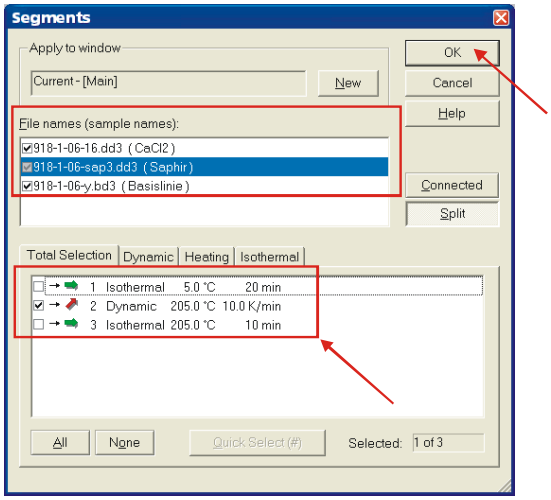
4. Data Evaluation

Determine the specific heat as follows:

	- **Open** the **Proteus Analysis** program.
	- Select **Open** from the **File** menu.
	- **Open** the 'sample+correction' data files for the **calibration measurement** with sapphire and **sample measurement** which were carried out in sections 2 and 3 (to do so **select** the **directory** under which the measurement files are saved). - The measured baseline is already received in both of the 'sample+correction' files. To show the baseline, click with the right mouse button on a measurement curve and select **Show Correction File**. - 

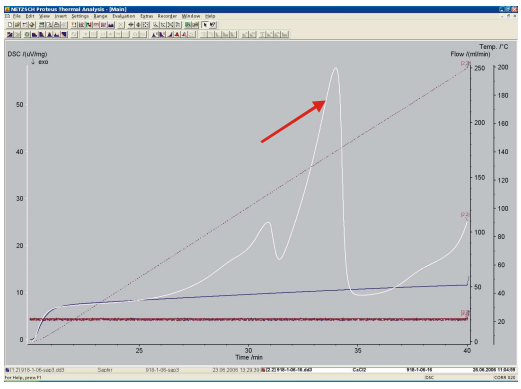


- Select **Segments** from the **Settings** menu.

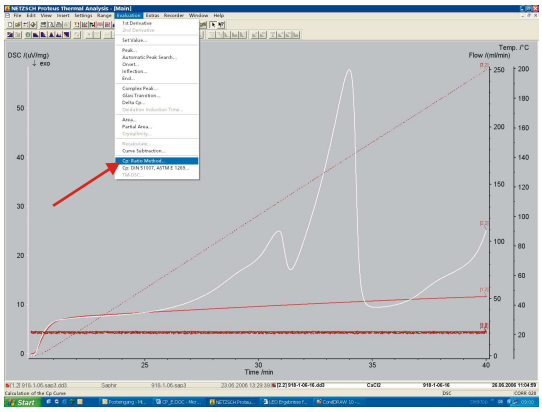


- For **all three measurements** (sample, calibration and baseline), select the **dynamic segment** of interest (the analysis of Cp-measurements is only possible for dynamic segments). The x-axis must be temperature-scaled.

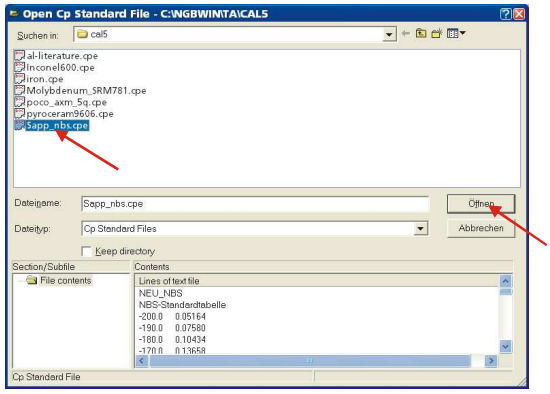
If all segments of the curve are selected to be shown, then the individual segments must be split in order to carry out Cp.



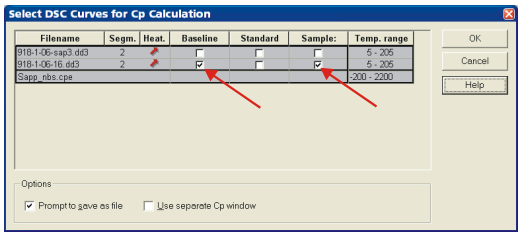
- Click on the Cp sample measurement curve to select it (carried out in section 3).



- Select **Cp Ratio Method...** from the **Evaluation** menu.

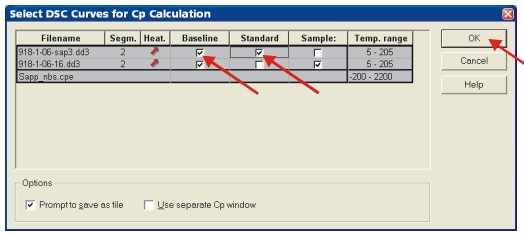


- Open the **Cp standard table** and select your standard material (e.g. sapphire).

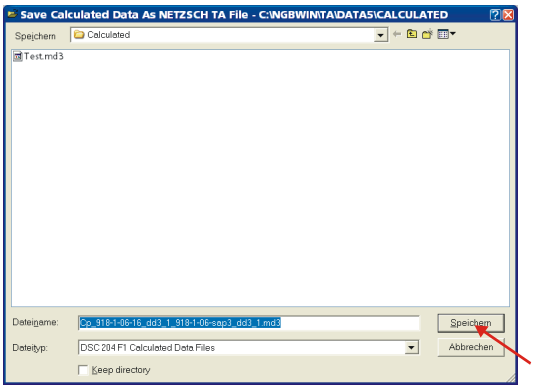
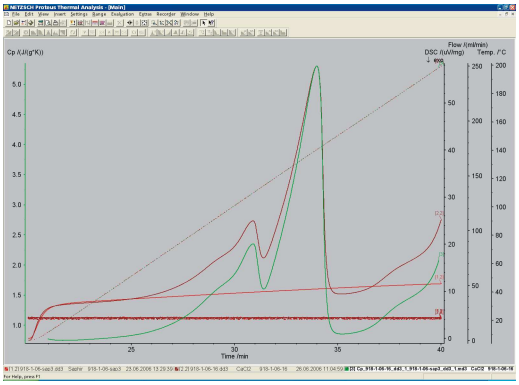


- A new table is opened showing all measured DSC curves and the Cp standard table.

- The white marked curve (sample measurement curve) was already automatically set into the dialog boxes (check marks in the **Baseline** and **Sample** dialog boxes) when the table was opened.



- The DSC measurement curve with the standard must be selected by a check mark in the **Standard** dialog box. A further check mark in the **Baseline** dialog box is set automatically.

	- **Save** the evaluation file.
	- The Cp curve is shown in the diagram.