

# Starting a Measurement and Data Evaluation of a Cp Measurement

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### **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.
- $\checkmark$  When selecting the table of standard values (C<sub>p</sub> Analysis), **be sure** that the calibration measurement and the table are based on the **same calibration material**.









# 1. Run a Baseline Measurement

NETZSCH - TA4_5	- Open the NETZSCH TA4_5 group.
Artic 25 CH - YA - 5       Control         Catility Berdbetten       Section         Catity Berdb	- Select your respective instrument (e.g. DSC 404 C).
DSC 404C Measurement Header       Image: Correction image: Cor	<ul> <li>Select Measurement Type Correction.</li> <li>Define Ident and Name.</li> <li>Click Continue.</li> </ul>



Open Temperature Recalibration - C:NGBWINTAICAL5     Suchen in      col5     cols     co	<ul> <li>Select the temperature calibration file.</li> <li>Continue with Open.</li> </ul>
Open Sensitivity File - C:WGBWINYTALCAL5     Suchain     cols     suchain     cols     Suchain     Construct     Sensterio exx     Contents     Leep directory     Section/Subfile     Contents	<ul> <li>Select the sensitivity calibration file Senszero.exx.</li> <li>Continue with Open.</li> <li>Continue with Open.</li> <li>For pure Cp evaluation, no sensitivity calibration is necessary.</li> </ul>
DSC 404C temperature Program Definition         Temperature Steps         Image: Control on the steps </td <td><ul> <li>Define the temperature program for the measurement (an example of a temperature program is shown in the figure).</li> <li>Click <b>Continue</b>.</li> </ul></td>	<ul> <li>Define the temperature program for the measurement (an example of a temperature program is shown in the figure).</li> <li>Click <b>Continue</b>.</li> </ul>



Define Measurement File Name - C:\NGBWIN\TA\DATAS     Speichem     deta5	- <b>Define</b> a <b>file name</b> and <b>save</b> the <b>measurement parameters</b> .
DSC       404C Adjustment on 18       ? ×         DSC       Start       Initial Cond. ON         1500       Initial Cond. ON       Exit         500       Exit       Help         1500       Temperature recalibration disabled!       5.0	- <b>Start</b> the <b>measurement</b> .



#### -After the baseline measurement is finished, select **Open** from the **File** menu. Open File - C:\NGBWIN\TA\DATA5 Select the baseline measurement -😂 da (carried out in section 1). -Continue with **Open**. Correction.bd Öffnen DSC 404C Measu • Abb Keep directory Measurement file types Value NETZSCH DSC 404 C • Customer Correction.bdc 820.132/05 20.06.2005.08:27:56 v NGB J. Blumm ×

# 2. Run a Calibration Measurement with Sapphire



#### Software Manual Cp Measurements

DSC 4942 Measurement Wizard     Image: Concelon       Measurement Type     Concelon       Sector/Solide     Sector/Solide       Sector/Solide     Concelon       Sector/Solide     Sector/Solide       Sector/Solide     Concelon       Sector/Solide     Sector/Solide       Sector/Solide     Concelon       Sector/Solide     Sector/Solide       Secto	<ul> <li>Select Sample + Correction (the sample measurement is thus corrected with the baseline measurement).</li> <li>Define Ident and Name.</li> <li>Enter the sapphire sample mass (mass of the sapphire disc). Crucible mass is optional, reference crucible is empty.</li> <li>In order to define the same settings as for the baseline</li> </ul>
	measurement (e.g. temperature program), set the check marks for Header Data, Temperature Program and Sensitivity.
	<ul> <li>If no check mark is set, you can define a new temperature program.</li> </ul>
	<ul> <li>Please note that the temperature program settings must be identical to the temperature program settings of the baseline measurement.</li> </ul>
	- Click <b>Continue</b> .
Open Temperature Recalibration - C:\NGBWINTAICALS     Suchen in     Cals     talzero.TMX     T.2005.ttl2	- Select the <b>temperature</b> <b>calibration</b> file (the same file as for the baseline measurement).
Deteigame: 07-2006.tdc Öffnen	- Continue with <b>Open</b> .
Leep directory     Leep directory     Leep directory     Leep directory     Contents     Co	
Define Measurement File Name - C:\ngbwin\ta\data5\Weasurements     Spejchem     Messurements     T     +      +     +     +     +	<ul> <li>Define a file name and save the sapphire measurement parameters.</li> </ul>
Døtelgeme: 3181-106-sap3.dd3 Speichem Døteltyp: DSC 404C Sample-Correction Files Abbrecht Keep directory	







# 3. Run a Sample Measurement with your Cp Sample

	<ul> <li>After the sapphire measurement is finished, select <b>Open</b> from the <b>File</b> menu.</li> </ul>
Copen File - C:\NGBWINTALDATAS         Suchenin <ul> <li>datas</li> <lidatas< li=""></lidatas<></ul>	<ul> <li>Select the baseline measurement (carried out in Section1).</li> <li>Continue with <b>Open</b>.</li> </ul>



#### Software Manual Cp Measurements

Volume       Social Calculation       Social Calculation <th><ul> <li>Select Sample + Correction (the sample measurement is thus corrected with the baseline measurement)</li> <li>Define Ident and Name.</li> <li>Enter the sample mass of the Cp sample. Crucible mass is optional, reference crucible is empty.</li> <li>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.</li> <li>If no check mark is set, you can define a new temperature program.</li> <li>Please note that the temperature program settings must be identical to the temperature program settings of the baseline measurement.</li> <li>Click Continue.</li> </ul></th>	<ul> <li>Select Sample + Correction (the sample measurement is thus corrected with the baseline measurement)</li> <li>Define Ident and Name.</li> <li>Enter the sample mass of the Cp sample. Crucible mass is optional, reference crucible is empty.</li> <li>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.</li> <li>If no check mark is set, you can define a new temperature program.</li> <li>Please note that the temperature program settings must be identical to the temperature program settings of the baseline measurement.</li> <li>Click Continue.</li> </ul>
Open Temperature Recalibration - C:NGBWINYTAYCALS      Suchen in:      Cal5     Jeter TMX     Jorden in:      Cal5     Jeter TMX     Jorden in:      Detergene:      Dr2006.tdc     Detergene:      Dr2006.tdc     Detergene:      Deterg	<ul> <li>Select the temperature calibration file (the same file as for the baseline measurement).</li> <li>Continue with Open.</li> </ul>
Define Measurement File Name - C'ingbwinitaidata5Weasurements     Speichem Measurements     P + & + + + + + + + + + + + + + + +	- Define a file name and save the sample measurement parameters.



#### Software Manual Cp Measurements

DSC 404C Adjustment on 18	- Start the measurement.
DSC 2500 2000 1500 1000	
500 0 -500 -1000 -1500 Temp. Threshold /K →	
2500	



# 4. Data Evaluation

# Determine the specific heat as follows:

Roteus Analysis	- <b>Open</b> the <b>Proteus Analysis</b> program.	
	- Select <b>Open</b> from the <b>File</b> menu.	
Open Measurement File - Cingbwinita/da/s5/Measurements         Suchenin       Measurements         Suchenin       Measurements         Suchenin       Measurements         Suchenin       Status         Suchenin       Measurement file         Deleigeme:       *918-106-sep3.dd3**318-106-16.dd3*         Deleigeme:       *918-106-16.dd3**         Section/Suble       Contents	<ul> <li>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).</li> <li>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.</li> </ul>	







	- Select <b>Cp Ratio Method</b> from the <b>Evaluation</b> menu.
Open Cp Standard File - C/WGBWINTAICALS     Image: Constraint of the constra	<ul> <li>Open the Cp standard table and select your standard material (e.g. sapphire).</li> </ul>
Select DSC Curves for Cp Calculation         Filename       Segm. Heat.         Bits 1:05-sig 3: 63       2         IP       P         Select DSC Curves for Cp Calculation         OK         Bits 1:05-sig 4:3         2       P         P         200: 200         Concell         Heip         Options         IP Promptito gave as file         Use separate Cp window	<ul> <li>A new table is opened showing all measured DSC curves and the Cp standard table.</li> <li>The white marked curve (sample measurement curve) was already automatically set into the dialog boxes (check marks in the <b>Baseline</b> and <b>Sample</b> dialog boxes) when the table was opened.</li> </ul>
Select DSC Curves for Cp Calculation       Image: Constraint of the constraint o	- The DSC measurement curve with the standard must be selected by a check mark in the <b>Standard</b> dialog box. A further check mark in the <b>Baseline</b> dialog box is set automatically.



Save C	Iculated Data As NETZSCH TA File - C:\NGBWINTA\DATA		-	Save the evaluation file.
Test.md				
Datei <u>n</u> ame:	Cp_918-1-06-16_dd3_1_918-1-06-sep3_dd3_1.md3	Speichem		
Dateityp:	DSC 204 F1 Calculated Data Files	Abbrechen		
	☐ Keep directory	h.		
DISTO TO DESCRIPTION	ennalistustyske-johan) 1. person janeta Dadaton Lyna Recorder Window Help 1. julicija wiednik ≤ ⊕ 1.03 Auksbeide Helm (* ¥)	- d +	-	The Cp curve is shown in the diagram.
Cp (U)(g*K))	l a and of Adata Shiek & Idde	Flow Resimin) DSC //(Wing) Terro, /*C		1 5
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B1121018-5-06-sep3 of	30         30           Time finds         2005/00120100000000000000000000000000000	40 318-168-16, 463, 1, 318-168 org2, 463, 1 me3, CoC2, 1918-168-16		
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