Temperature and Sensitivity Calibration Protocol

NETZSCH

Software Manual

DSC Instruments

Temperature and Sensitivity Calibration

www.netzsch-thermal-analysis.com

To be explained:

- ***Measurement of the calibration substances***
- ***Evaluation of the measurements***
- ***Creation of a temperature/sensitivity calibration file***

Please note the following additional information for the calibration procedure:

- The measurement conditions (e.g. heating rate, gases, type of crucible) for the calibration measurement and the subsequent sample measurement must be the same.
- Use an empty crucible for the reference position.
- Note all information for using the calibration substances in the provided documentation.

Melting point standards

Can be used for Al2O3 crucibles and Pt crucibles with Al2O3 liners; C10H16, In, Sn, Bi, Zn also for aluminum crucibles.

Adamantane (C10H16) → for the low temperature range
Indium (In) → most accurate value
Tin (Sn)
Bismut (Bi)
Zinc (Zn)
Aluminum (Al)
Silver (Ag) → melting point depends on oxygen partial pressure
Gold (Au) → very accurate value

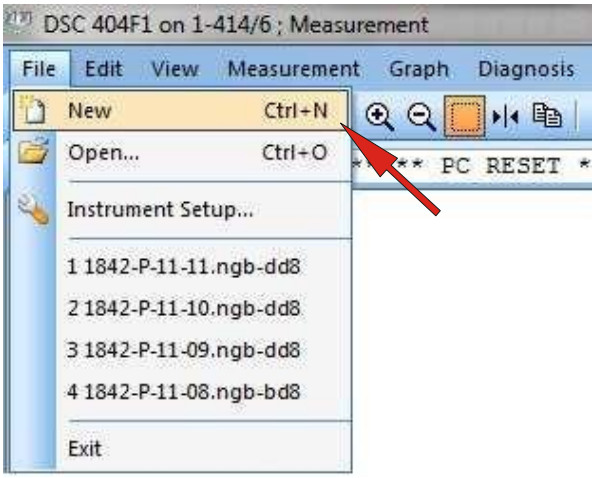
For the low temperature range use C10H16, In, Sn, Bi, Zn.
Al, Ag and Au are not applicable to low temperature furnaces.

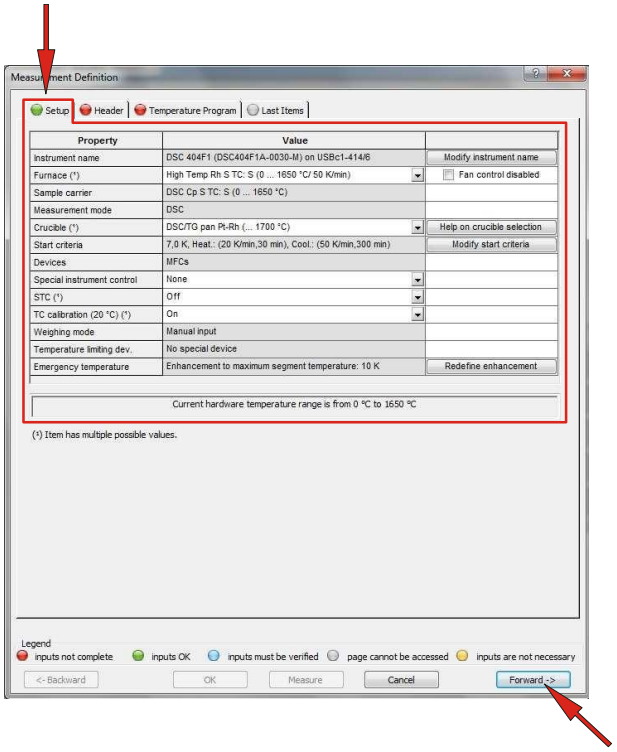
Standards showing polymorphic transitions

Can be used for Pt crucibles; RbNO3, KNO3, KClO4, Ag2SO4 and CsCl also for aluminum crucibles.

Rubidium nitrate (RbNO3) → only for temperature calibration and one heating step (not useable for 2nd and 3rd heating)
Potassium nitrate (KNO3) → only for one heating step (not useable for 2nd and 3rd heating)
Potassium perchlorate (KClO4) → only for one heating step (not useable for 2nd and 3rd heating)
Silver sulphate (Ag2SO4) → only for one heating step (not useable for 2nd and 3rd heating)
Cesium chloride (CsCl)
Potassium chromate (K2CrO4) → not applicable to low temperature furnaces
Barium carbonate (BaCO3) → not applicable to low temperature furnaces
Strontium carbonate (SrCO3) → not applicable to low temperature furnaces

1. Measurement of the Calibration Substances

	1. Open the NETZSCH-Proteus group.
	2. Select the instrument (e.g. DSC 404 F1).
	3. Select **New** in the **File** menu.



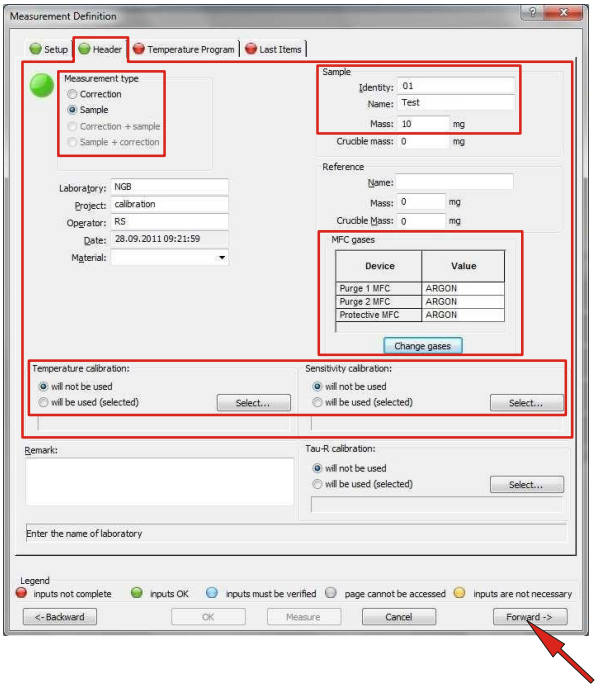
4. **Measurement Definition**

Setup

- Check the instrument setup.

  The values of the different properties must be checked and changed (if necessary).

- Click **Forward**.



5. **Measurement Definition Header**

- Select Measurement Type **Sample**.

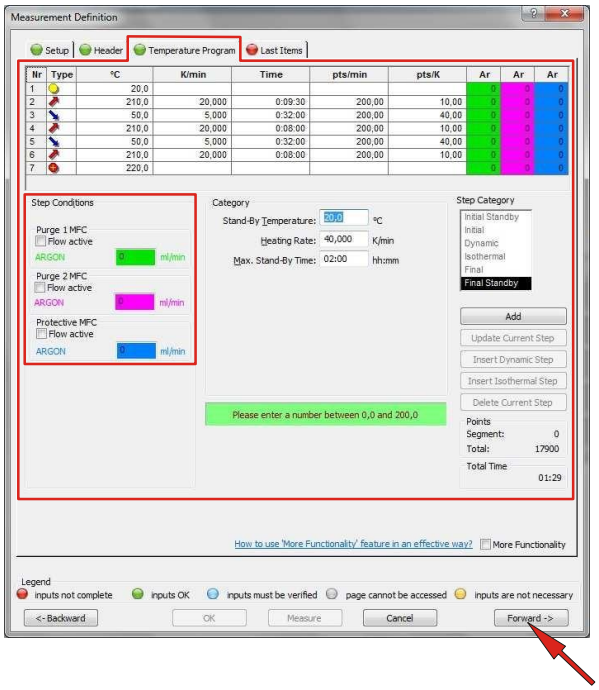
  Optionally:
  Define **Laboratory**, **Project**, **Operator**, **Date** and **Material**.

  Define **Identity**, **Name** and enter the **sample mass**.

- Set the checkmark **will not be used** for temperature calibration and also for sensitivity calibration.

- Define the gas for **Purge 1**, **Purge 2** and **Protective**.

- Click **Forward**.



Heating rate: depending on the application (e.g. 20 K/min)

Acquisition rate: 100 Points/min

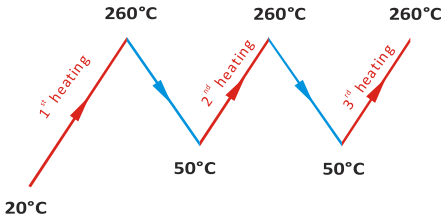
Cooling rate: select a practicable cooling rate (depending on the instrument)

6. **Measurement Definition Temperature Program**

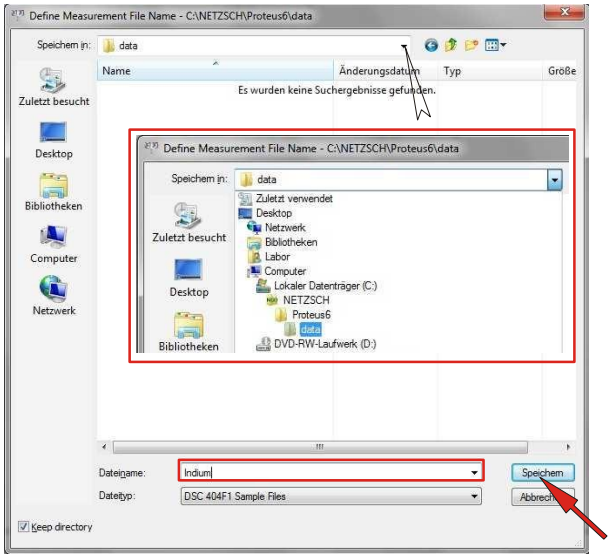
- Enter the temperature program for the first calibration substance.

  e.g. Indium (melting point 156.6°C)

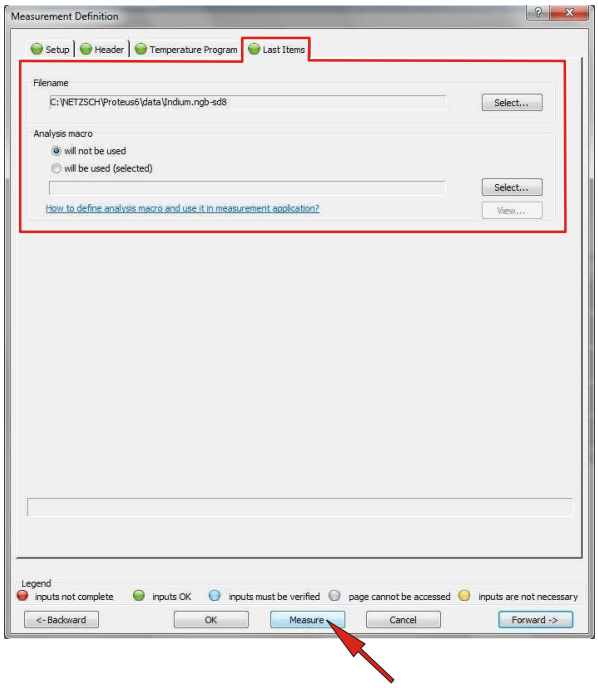
  Carry out a measurement with 3 sequential heating segments up to 260°C and 2 cooling segments down to 50°C (see figure below). For the evaluation afterwards use only the peak from the 2nd and 3rd heating. See also all additional information on page 6 for a detailed description of the temperature program for each standard!



- Define the gas flow for purge and protective (e.g. purge 50 ml/min, protective 20 ml/min).
- Click **Forward**.



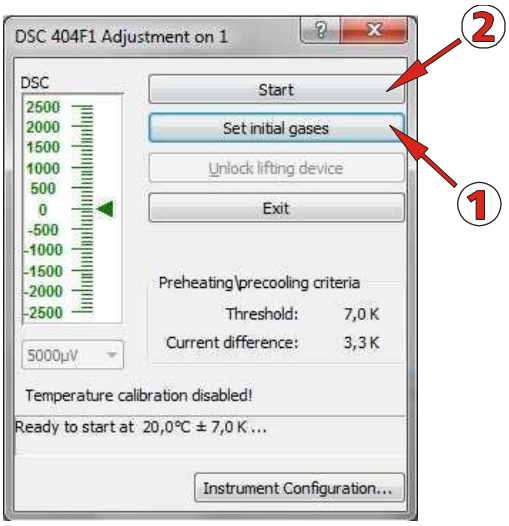
7. Define folder and file name and save the measurement.



8. **Measurement Definition**

Last Items

- The previously selected folder and filename is shown in the window (to change folder and filename once again click the **Select** button).
- Click **Measure**.



9. **Start** the **Measurement**

- Push **Set initial gases**
  (the gas switches are set to the INITIAL start conditions (carried out automatically during preheating/ precooling)).
- Click **Start**.

  If necessary, check the **Instrument Configuration**.

Additional Information for the Temperature Program

- *Carry out measurements for all standard materials with 3 heating and 2 cooling segments so that the melting peak is measured three times.*
- *Depending on the instrument configuration (e.g. furnace type, sample carrier) modify the end temperature of the heating segment (shown as an example in the table below) in such a way that the complete melting peak can be evaluated. Cool down to about 100°C below the melting point.*
- *For the evaluation use only the values from the 2nd and 3rd heating (both for temperature and sensitivity calibration).*

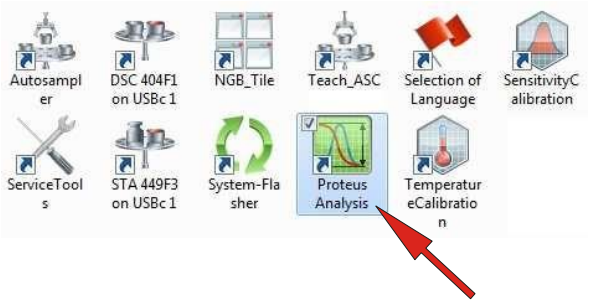
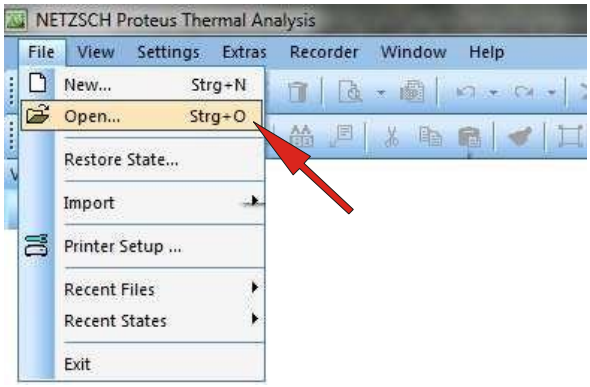
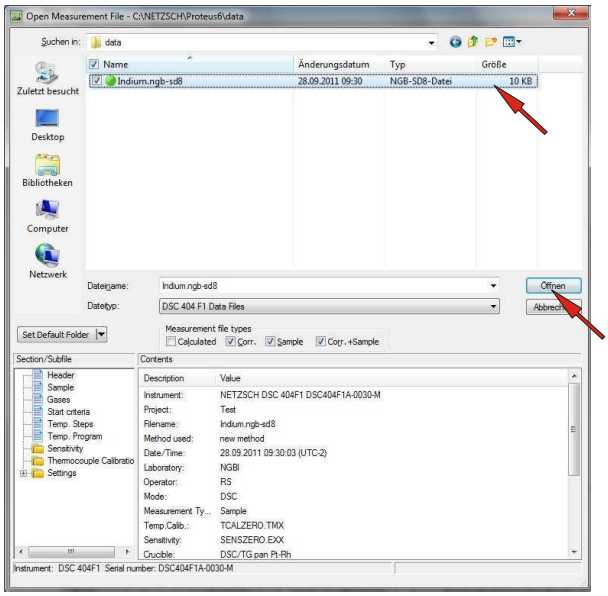
calibration substance	melting point in °C	temperature program (example)
Adamantane (C10H16) (for low temperature range)	-64.5	-120°C to -20°C (heating segment) *-20°C to -120°C (cooling segment)* -120°C to -20°C (heating segment) *-20°C to -120°C (cooling segment)* -120°C to 25°C (heating segment) (so that you can open the furnace)
Indium (In)	156.6	20°C to 260°C (heating segment) *260°C to 50°C (cooling segment)* 50°C to 260°C (heating segment) *260°C to 50°C (cooling segment)* 50°C to 260°C (heating segment)
Tin (Sn)	231.9	20°C to 340°C (heating segment) *340°C to 130°C (cooling segment)* 130°C to 340°C (heating segment) *340°C to 130°C (cooling segment)* 130°C to 340°C (heating segment)
Bismuth (Bi)	271.4	20°C to 380°C (heating segment) *380°C to 170°C (cooling segment)* 170°C to 380°C (heating segment) *380°C to 170°C (cooling segment)* 170°C to 380°C (heating segment)
Zinc (Zn)	419.6	20°C to 480°C (heating segment) *480°C to 320°C (cooling segment)* 320°C to 480°C (heating segment) *480°C to 320°C (cooling segment)* 320°C to 480°C (heating segment)
Aluminium (Al)	660.6	20°C to 760°C (heating segment) *760°C to 560°C (cooling segment)* 560°C to 760°C (heating segment) *760°C to 560°C (cooling segment)* 560°C to 760°C (heating segment)
Gold (Au)	1064.4	20°C to 1150°C (heating segment) *1150°C to 950°C (cooling segment)* 950°C to 1150°C (heating segment) *1150°C to 950°C (cooling segment)* 1150°C to 950°C (heating segment)

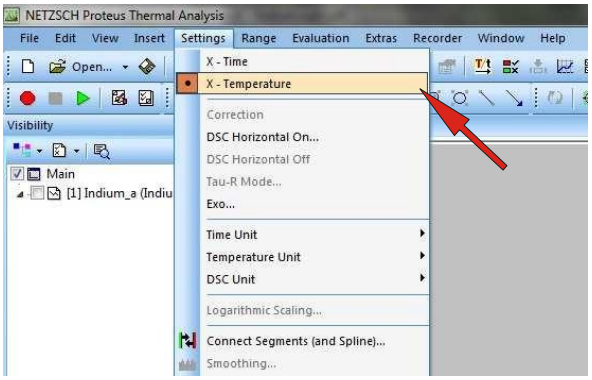
Heating rate: depending on the application (e.g. 20 K/min)

Acquisition rate: 100 Points/min

Cooling rate: select a practicable cooling rate (depending on the instrument)

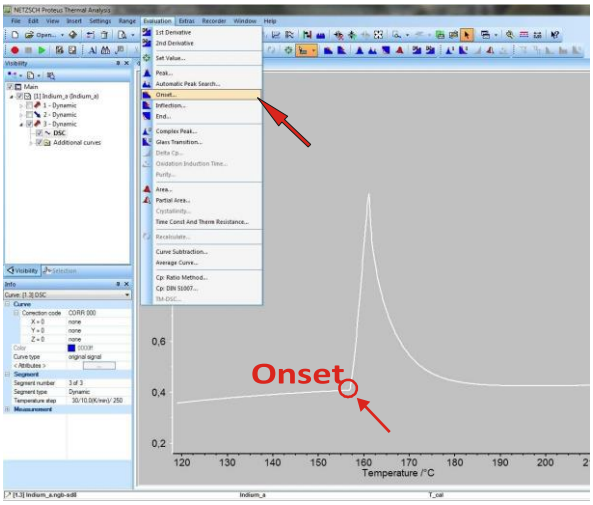
2. Evaluation of the Measurements

	10. Open the **Proteus Analysis**.
	11. Select **Open** in the **File** menu.
	12. Open your respective measurement (the measurement file which you have saved under item 7). Continue with **Open**.

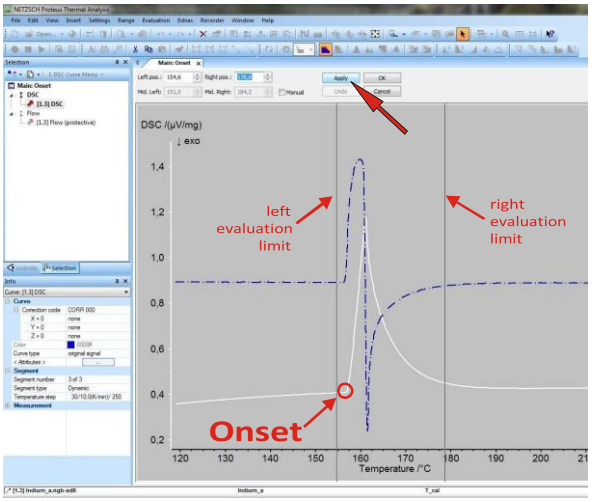


13. Select **X-Temperature** in the **Settings** menu. The temperature is shown on the X axis.

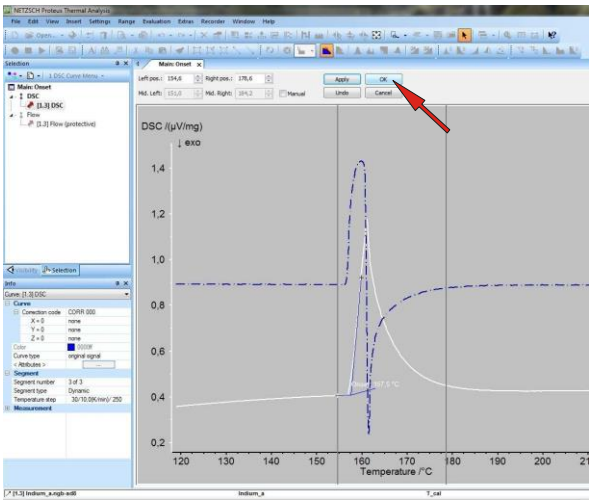
Temperature Calibration



14. Click on the DSC curve for selection. Select **Onset** in the **Evaluation** menu to determine the extrapolated onset.



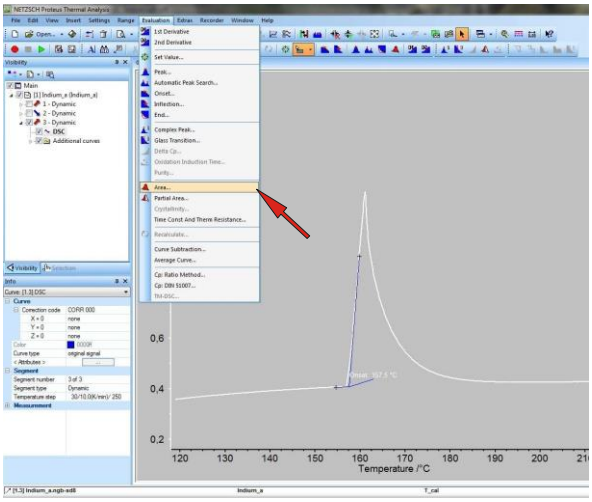
15. To evaluate the extrapolated onset, define the right and left evaluation limit as shown in the figure and click **Apply**.



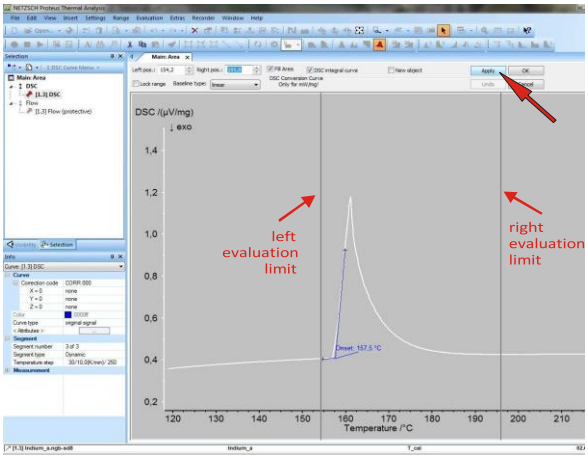
16. If the extrapolated onset was determined correctly, click **OK**. If not, it is still possible to change the right and left evaluation limit. To evaluate click **Apply**. If the extrapolated onset was determined correctly, click **OK**.

    Write down the determined onset value of the 2nd and 3rd measurement.

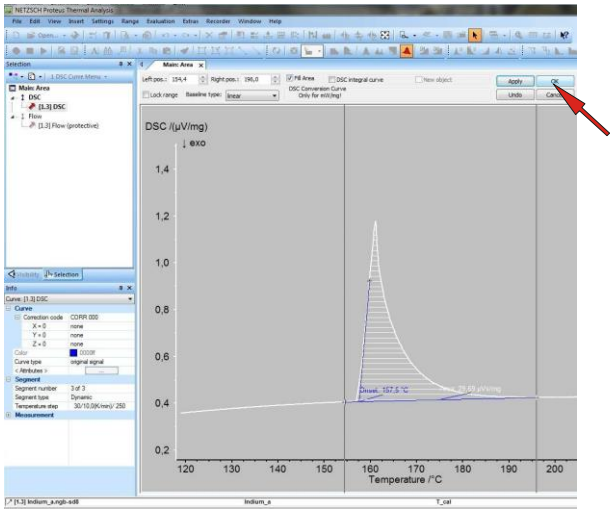
Sensitivity Calibration



17. Select **Area** in the **Evaluation** menu.



18. To evaluate the peak area, define the right and left evaluation limit as shown in the figure and click **Apply**.



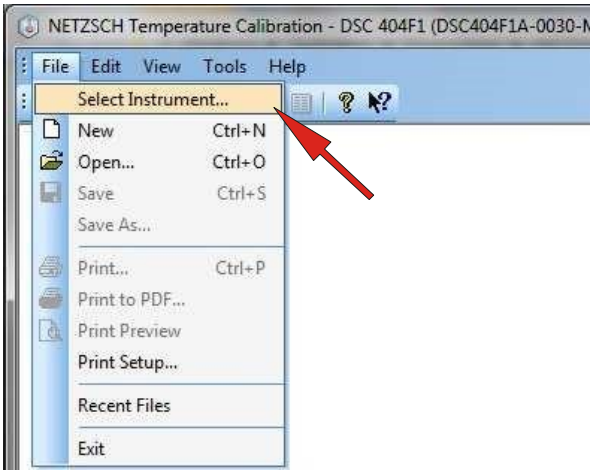
19. If the peak area was determined correctly, click OK. If not, it is still possible to change the right and left evaluation limit. To evaluate click **Apply**. If the peak area was determined correctly, click **OK**.

    Write down the determined peak area of the 2nd and 3rd measurement.

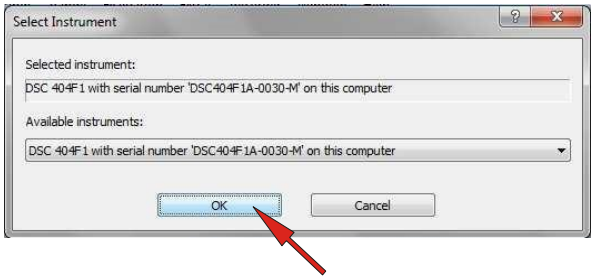
3. Creation of a Temperature/Sensitivity Calibration File

Temperature Calibration

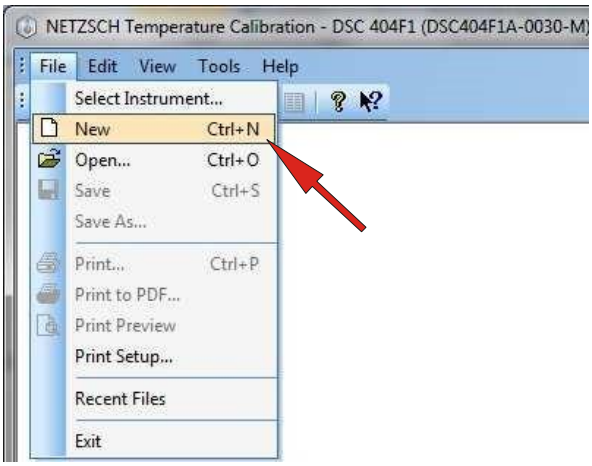
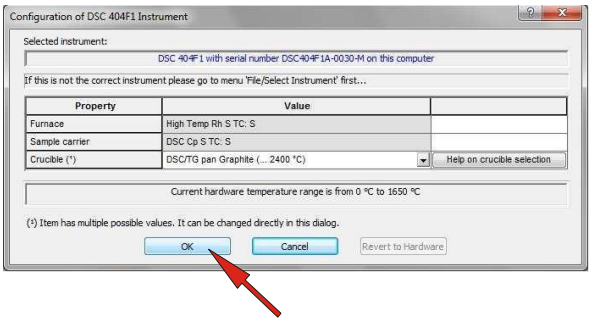
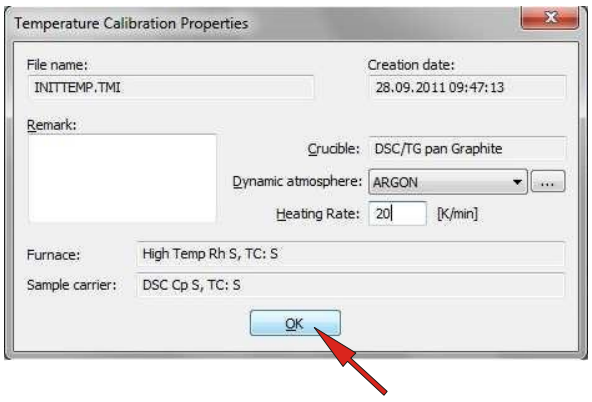
20. Open **Temperature Calibration** in the NETZSCH-Proteus group.

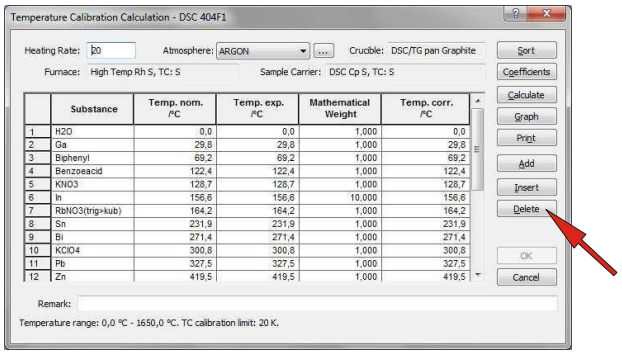


21. Open **Select Instrument** in the **File** menu.

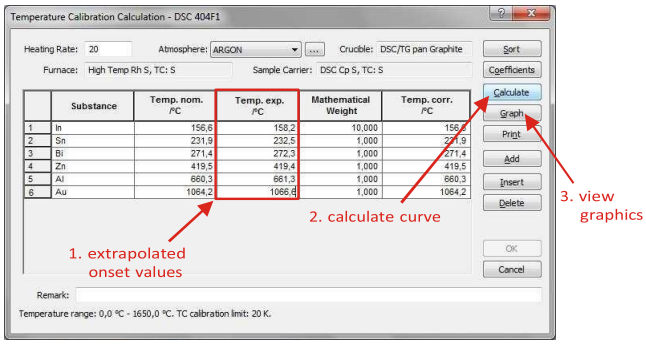


22. Select your instrument and click **OK**.

	23. Select **New** in the **File** menu.
	24. Check the instrument configuration (change if necessary). Continue with **OK**.
	25. Define both the **atmosphere** and the **heating rate** used for the calibration measurements. Continue with **OK**.



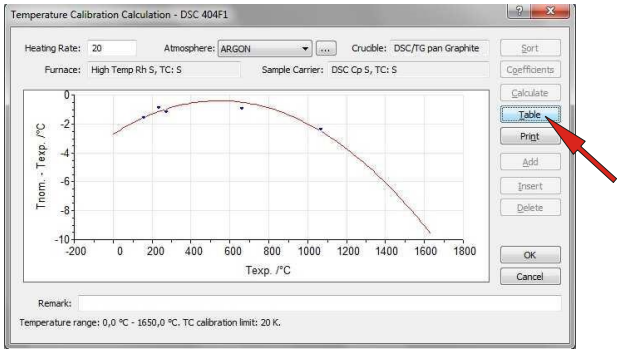
26. Click **Delete** to remove all materials you have not measured.



27. Enter the determined onset values for all calibration materials.

    Click **Calculate** to evaluate the curve.

    Click **Graph** to see the calibration curve.



28. Check the correct form of the curve. The curve is acceptable if:

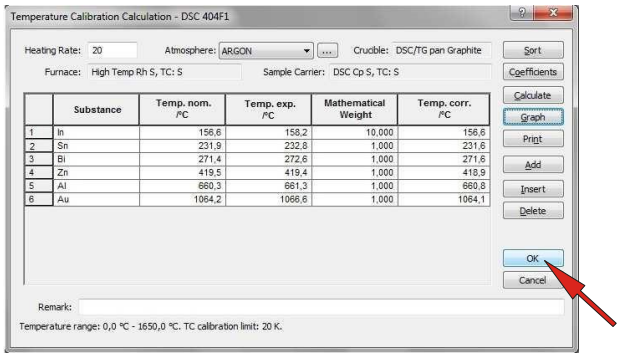
- The fitted curve goes through all points.
- **Temp. nom.** and **Temp. corr.** are nearly the same (depending on your requirements, e.g. **Temp. nom. - Temp. corr. ± 1-3 K).**

Temp. nom. /°C	Temp. exp. /°C	Mathematical Weight	Temp. corr. /°C
156,6	158,2	10,000	156,6
231,9	232,8	1,000	231,6
271,4	272,6	1,000	271,6
419,5	419,4	1,000	418,9
660,3	661,3	1,000	660,8
1064,2	1066,6	1,000	1064,1

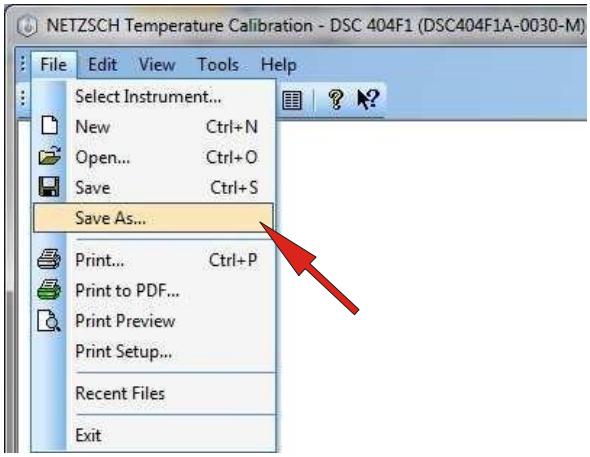
Temp. nom. Temp. corr.

The column **Mathematical Weight** contains the mathematical weighing for the fit. It can be edited for each standard to change the shape of the calibration curve. From all calibration standards Indium can be measured with highest accuracy ⇒ Mathem. Weighing 10.

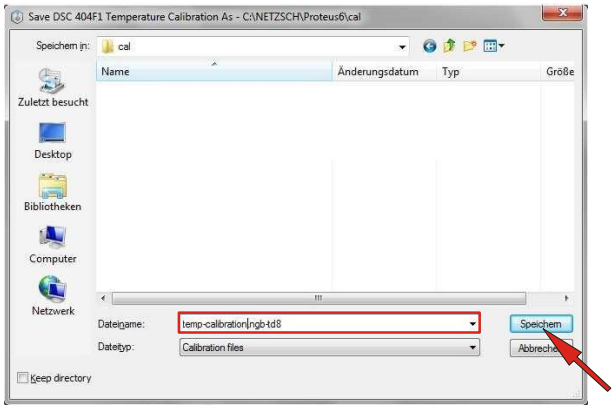
Click **Table** to change the input or accept the settings.



29. Click **OK** to save the calculation.

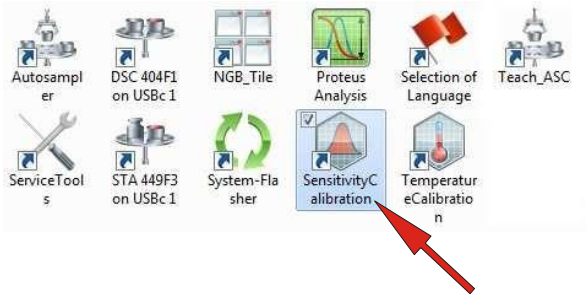


30. Select **Save as** in the **File** menu to save the data.

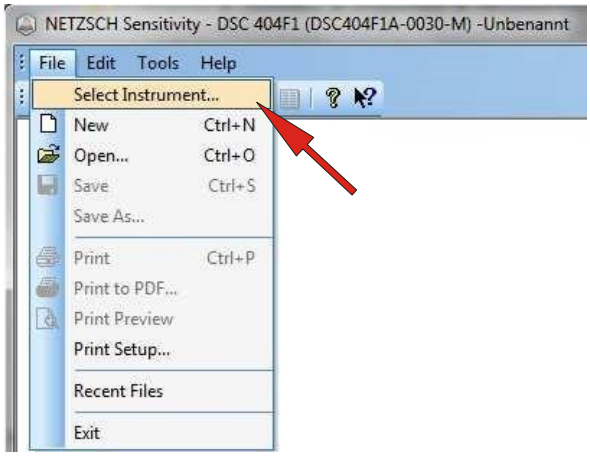


31. Define the file name of the **temperature calibration file** and click **Save**.

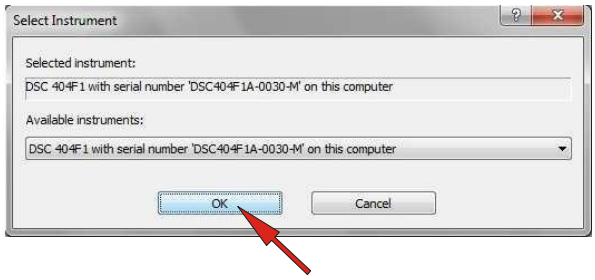
Sensitivity Calibration



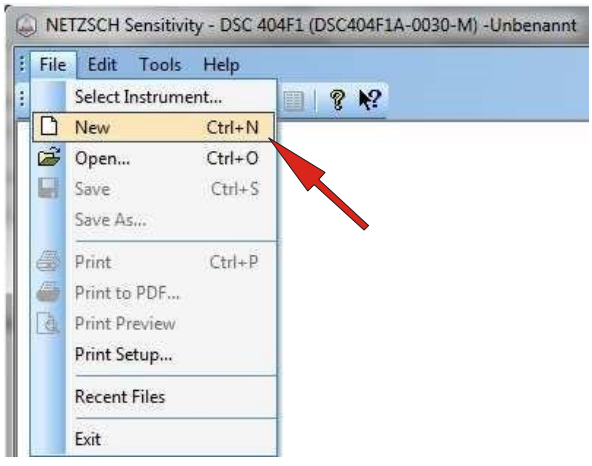
32. Open **Sensitivity Calibration** in the NETZSCH-Proteus group.



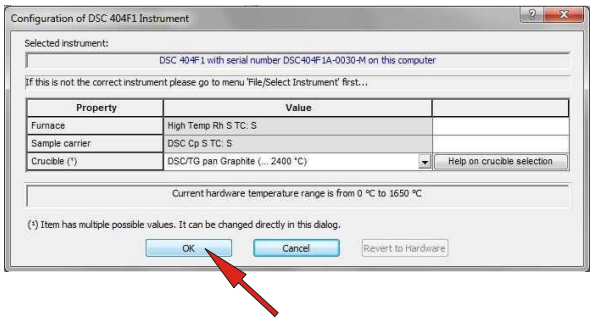
33. Open **Select Instrument** in the **File** menu.



34. Select your instrument and click **OK**.

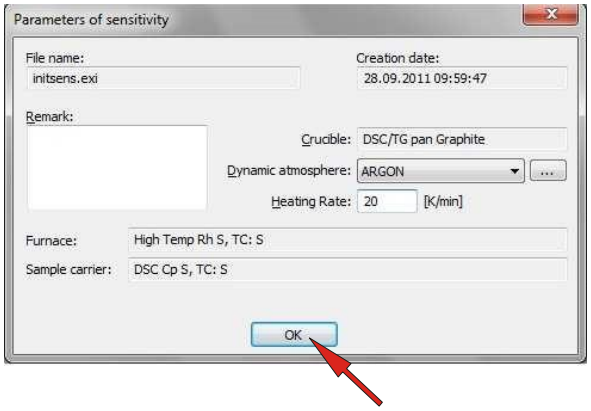


35. Select **New** in the **File** menu.



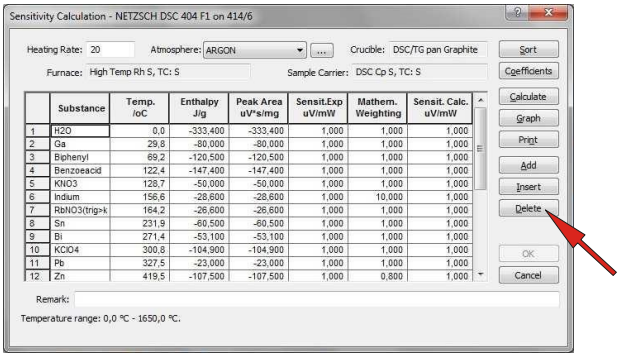
36. Check the instrument configuration (change if necessary).

    Continue with **OK**.

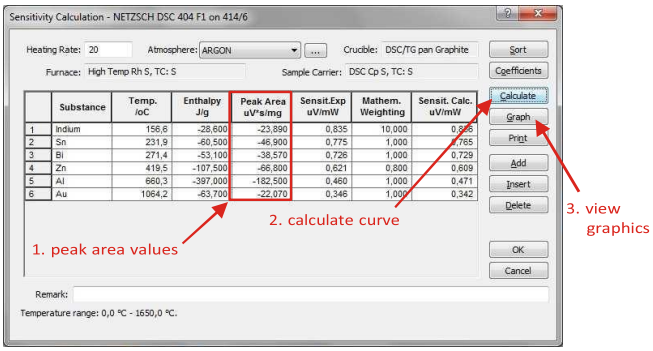


37. Define both the **atmosphere** and the **heating rate** used for the calibration measurements.

    Continue with **OK**.



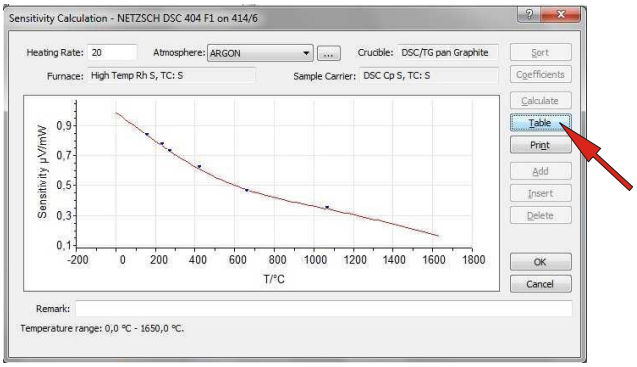
38. Click **Delete** to remove all materials you have not measured.



39. Enter the determined values for the peak area for all calibration materials.

    Click **Calculate** to evaluate the curve.

    Click **Graph** to see the calibration curve.



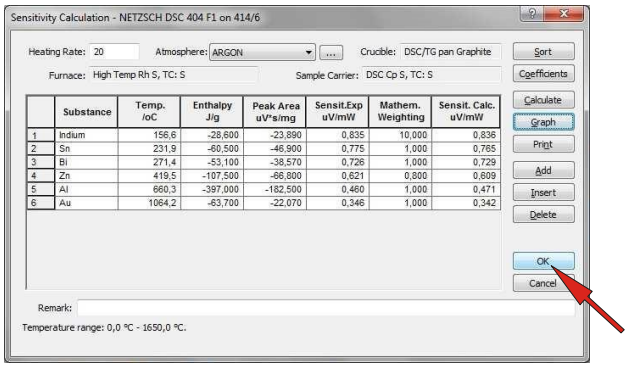
40. Check the correct form of the curve.
    The curve is acceptable if the deviation between **Sensit. Calc.** and **Sensit. Exp.** is less than ± 3%.

Peak Area uV*s/mg	Sensit.Exp uV/mW	Mathem. Weighting	Sensit. Calc. uV/mW
-23,890	0,835	10,000	0,836
-46,900	0,775	1,000	0,765
-38,570	0,726	1,000	0,729
-66,800	0,621	0,800	0,609
-182,500	0,460	1,000	0,471
-22,070	0,346	1,000	0,342

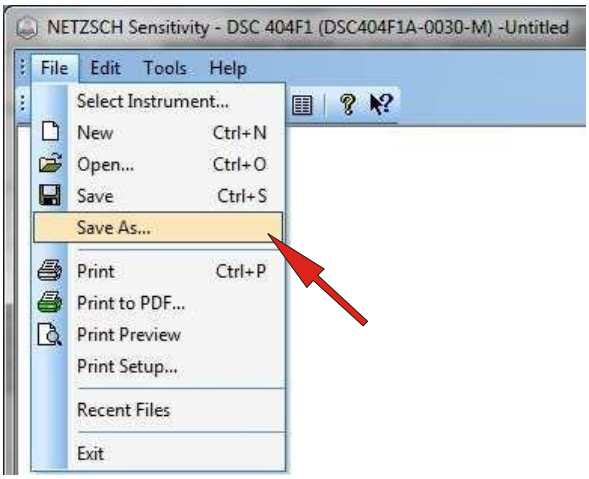
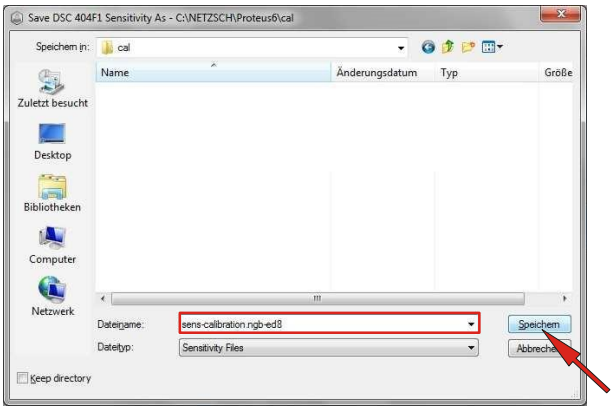
Sensit. Exp Sensit. Calc.

    The column **Mathem. Weighing** contains the mathematical weighing for the fit. It can be edited for each standard to change the shape of the calibration curve. From all calibration standards Indium can be measured with highest accuracy ⇒ Mathem. Weighing 10.

    Click **Table** to change the input or accept the settings.



41. Click **OK** to save the calculation.

	42. Select **Save as** in the **File** menu to save the data.
	43. Define the file name of the **sensitivity calibration file** and click **Save**.