NETZSCH

Proven Excellence.

SmartMode Manual
Proteus 8.0

for

DSC Instruments

53623 V1.0 / August 2019

DOCUMENTATION

206030005 80032061 2020-12-03

206030005 80032061 2020-12-03

Content

General Information

In the design of your instrument, we endeavour to take individual solutions into account and to include these in the documentation.

However, in order to keep the scope of the technical documentation at a reasonable level, we must limit the description to a standard model.

We ask for your understanding, if additional information particular to your instrument is not included within the standard instructions.
Additional information can always be found on the corresponding information sheets.

All technical data, instrument features and other information described in these operating instructions are presented to the best of our knowledge and in accordance with the technical standards of the instrument at the time of printing.

We welcome any comments, suggestions or new ideas concerning the instrument and its operating instructions. Please address them to:

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Software Manual – Smart Mode Measurement Proteus 8.0

The Smart Mode is designed for easily starting measurements based on predefined methods optionally with automatic evaluation. Moreover, Smart Mode offers wizards for typical operations. It is also possible to create new methods or to use analysis states based methods created in Proteus analysis.

'Smart Mode Measurement' is a standard feature of Proteus 8.0 for the DSC instruments Series 200 (DSC 214 Polyma, DSC 204 F1 Phoenix, DSC 3500 Sirius and DSC 200 F3 Maya).

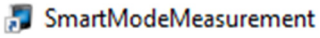
The Smart Mode does not support special applications, e.g. instrument coupling with UV devices (e.g. Omnicure), mass spectrometer (Aeolos), FTIR or GC-MS. In those cases Expert mode has to be applied to perform measurements.

DSC-BeFlat, DSC-correction (TAU-R) are software extensions which are delivered free of charge for some instruments (DSC 214, DSC 204 F1) or as an extra price option for other DSC instruments (DSC 3500, DSC 200 F3). If available, the Smart Mode supports using these calibrations in methods as well as wizards, or allows to renewal and also create calibrations.

Start SmartMode Software

NETZSCH-Proteus-80

Open the NETZSCH-Proteus group on desktop.

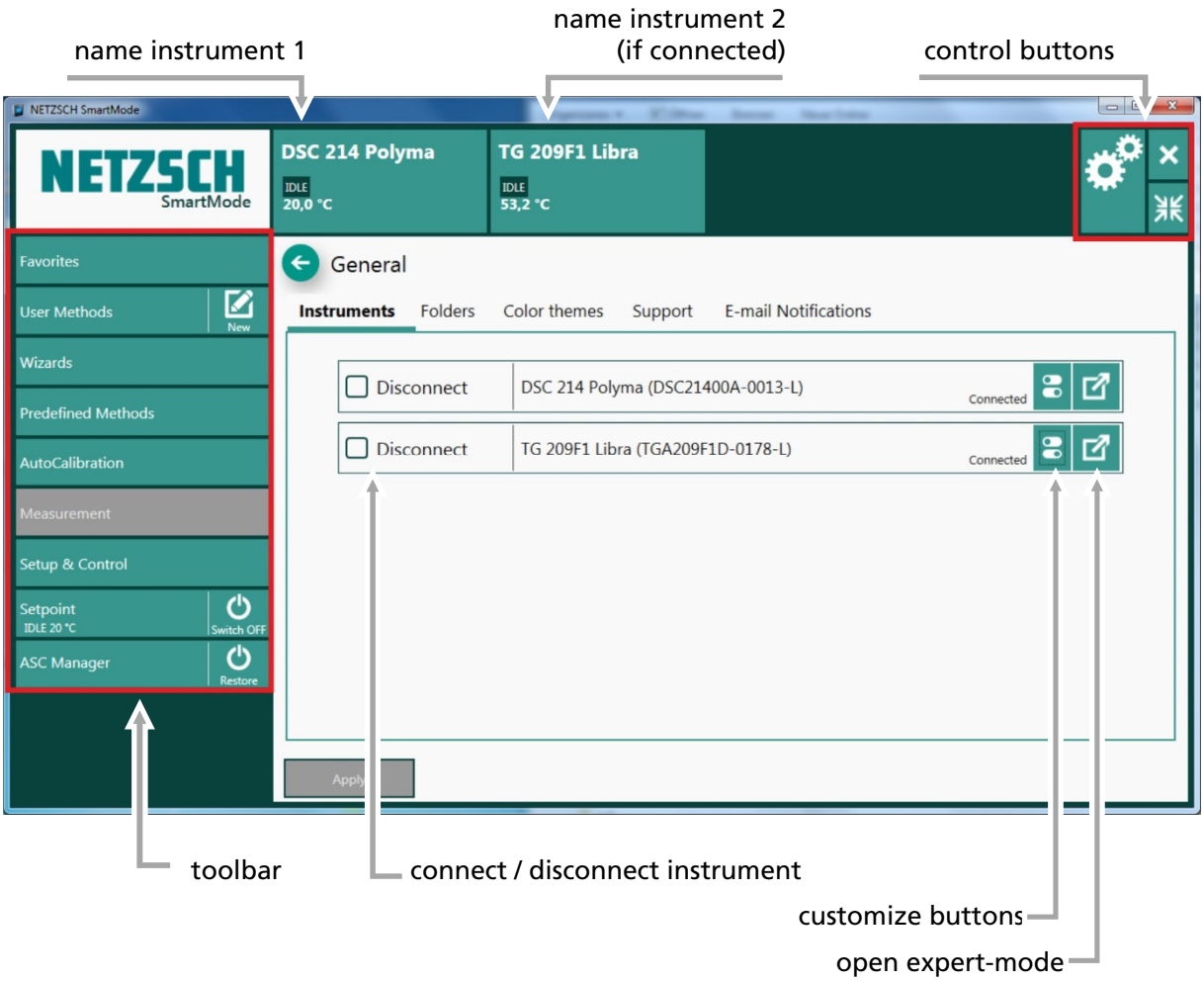


Open the SmartMode Measurement.

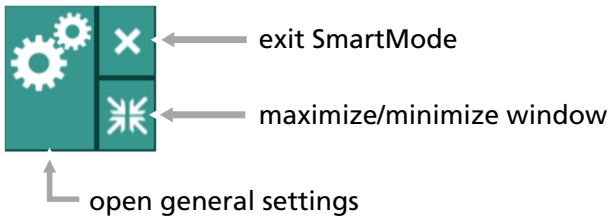
SmartMode-Screen

Screen below appears when starting the SmartMode-software.
It may vary depending on the connected instrument(s). It is possible to connect up to four instruments.

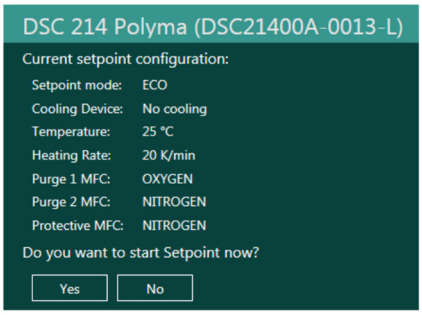
White tabs on toolbar are active, green tabs can be selected and grey tabs are not active right now.



Control buttons:



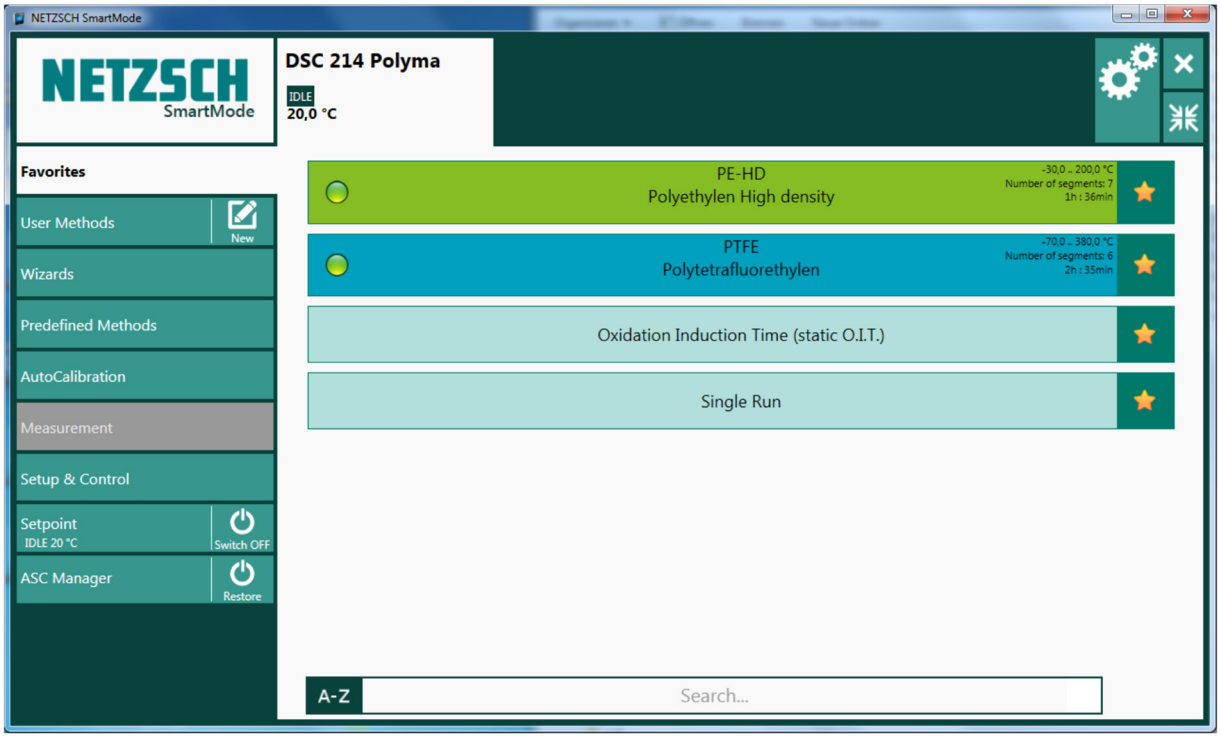
Activate Setpoint



Enable setpoint to keep the instrument always under controlled conditions (protective/ purge gas flow, coolings). For DSC instruments with connected intracooler (IC) the setpoint prohibits a frozen cell when the IC is active, but heater is inactive. In auto sampler mode the sample is inserted/ removed during setpoints only.

For more details regarding the setpoint refer to page 30.

SmartMode Tab Favorites



Favorites are shortcuts to methods, tests or calibrations, which are predefined and selected by the user.

To define a file as a favorite it must be marked with the symbol in the specific menu. The yellow star shows the marked state. Only marked methods are listed at Favorites tab.

SmartMode Tab User Methods

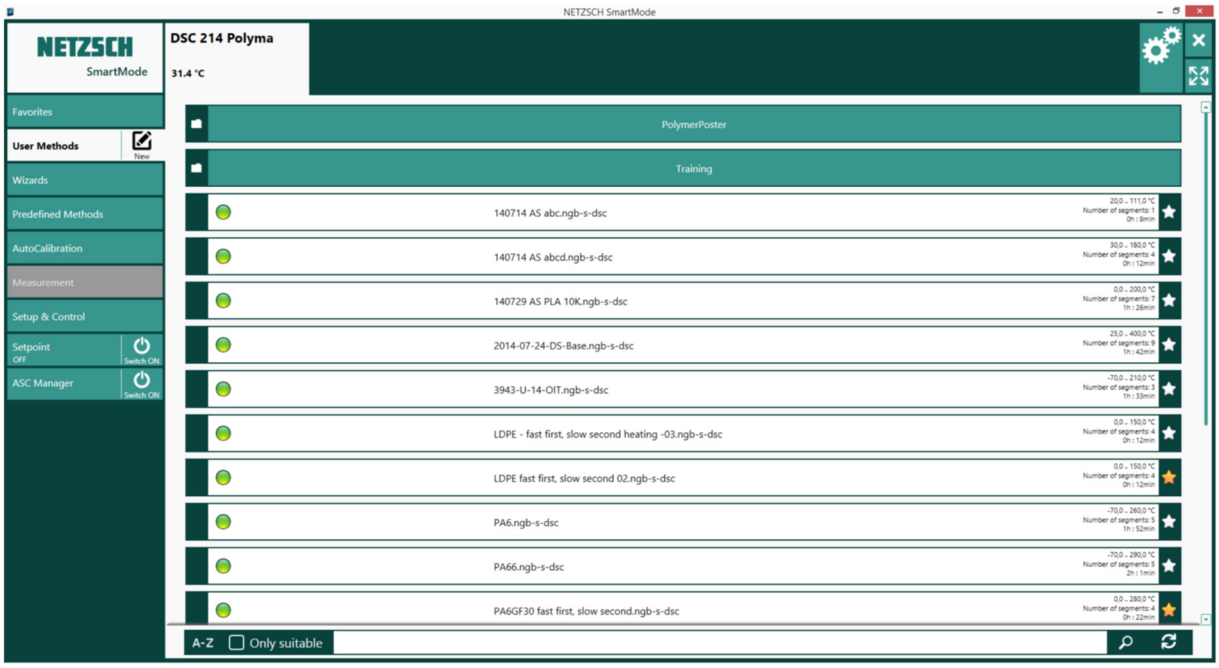
NOTE:

Methods can not be edited when created. Rename, open (start measurement) or delete methods is possible via methods manager.

Red highlighted headers must be completed, green highlighted headers are OK.

Instrument returns to Idle setpoint temperature after measurement.

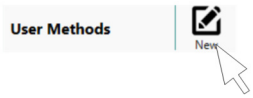
All user methods up to now are listed here (independent of creation via expert mode or smart mode)



Created methods can not be copied or moved from one method to another method.

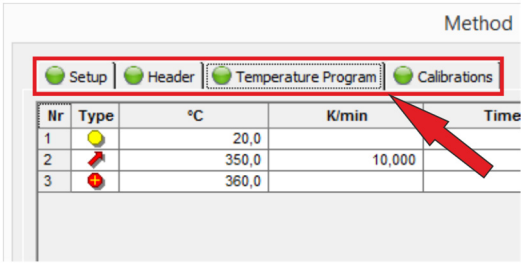
On the right hand side of the method you see the temperature range, number of segments and the required measurement time. To see more measurement settings of each method, click on the method.

Create a new method

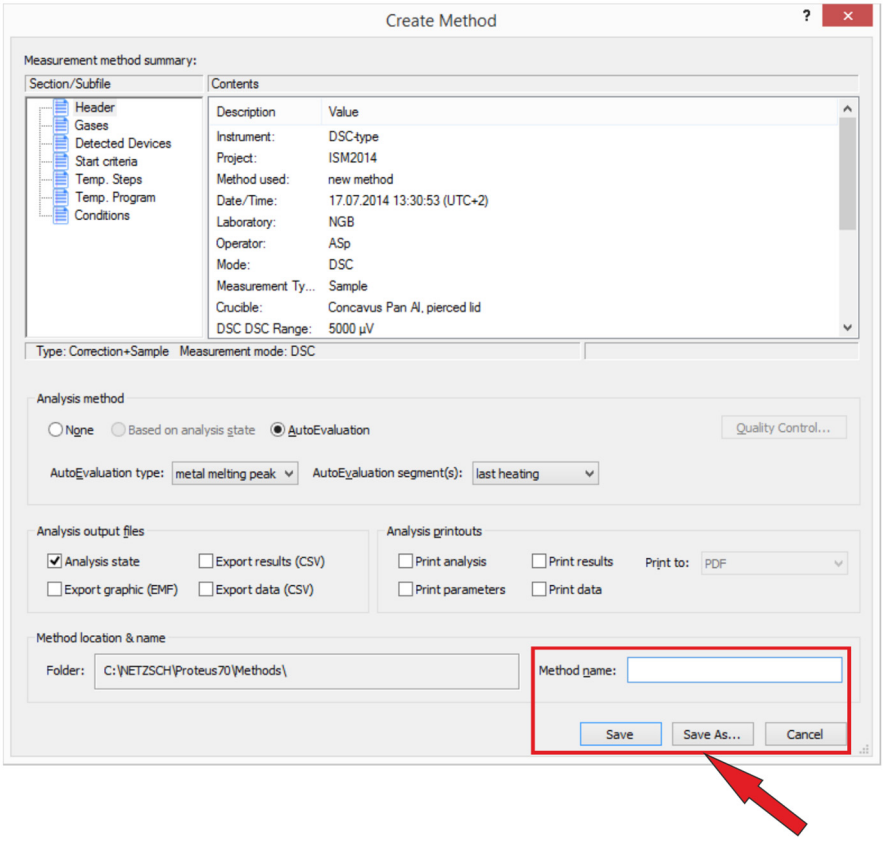


Click on 

, a pop up window will start to create a new method.



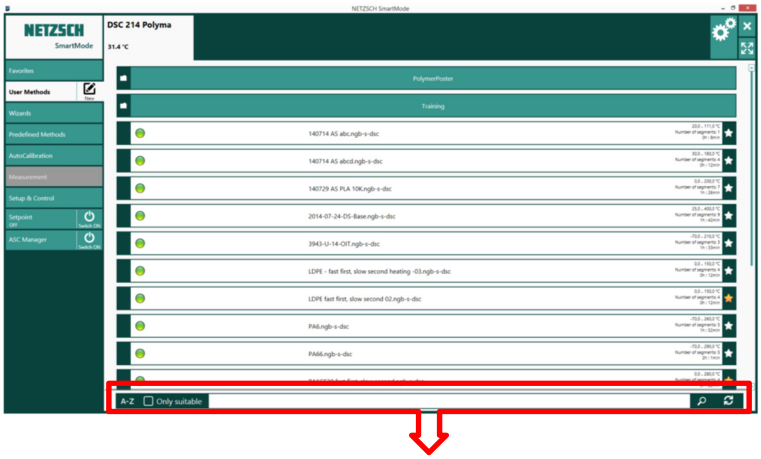
Define setup, header, temperature program, calibrations and save the method. If calibrations are applied later on when using the method, suitable calibrations are precondition.

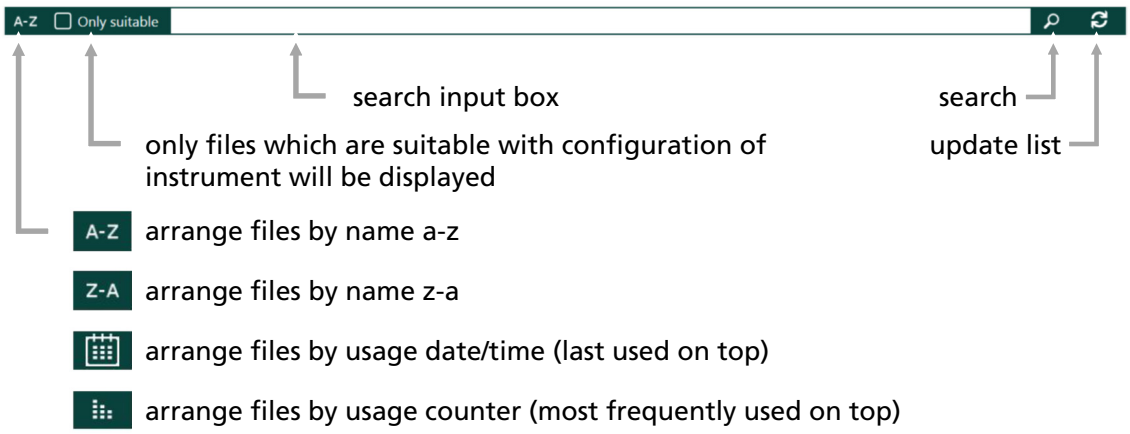


The new created method will also be listed in the user methods tab.

Search toolbar

Search toolbar appears on different screens, e.g. on “User Methods” screen.

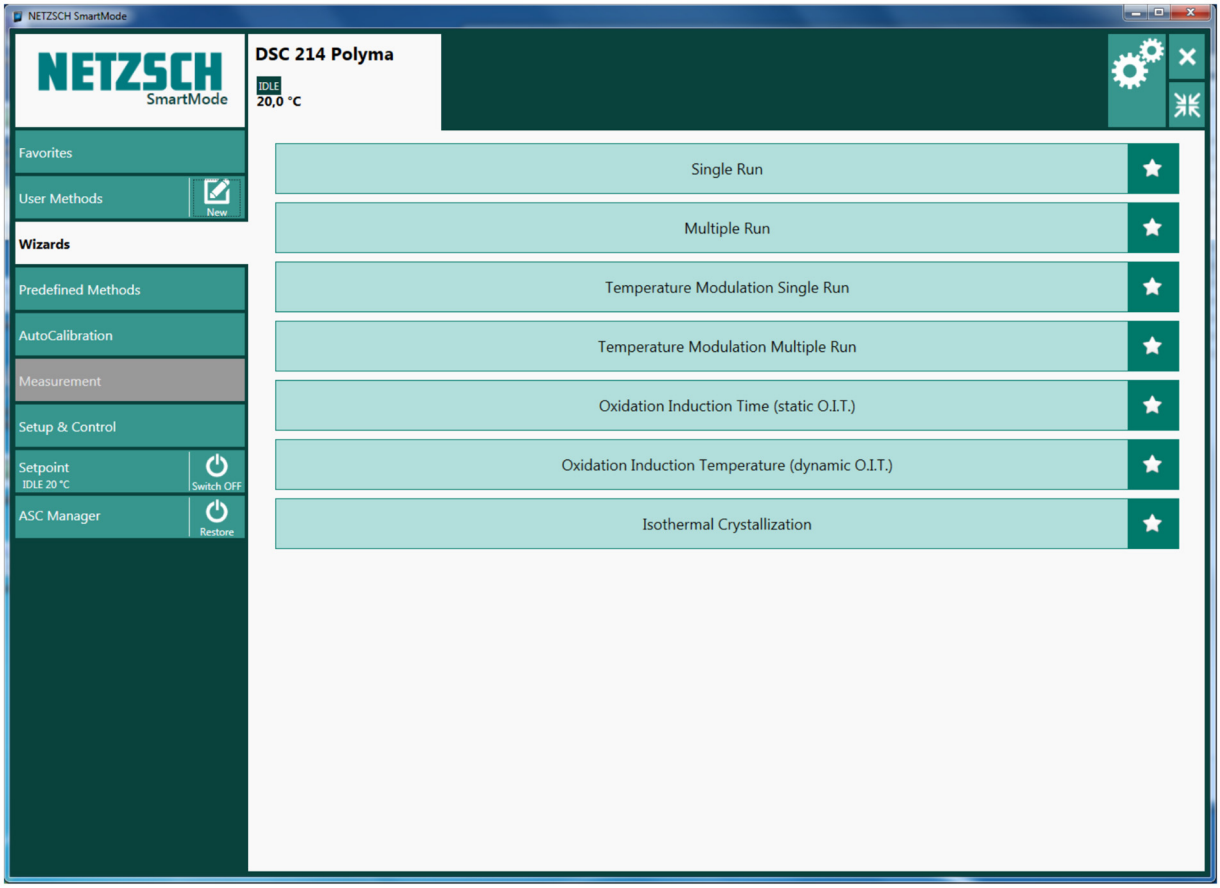




206030005 80032061 2020-12-03

SmartMode Tab – Wizards

Structurally predefined measurement programs can be selected for typical operations like single run, multiple run, O.I.T., isothermal crystallization, TM-DSC (option). Moreover, crucible type and gas flow can be selected.

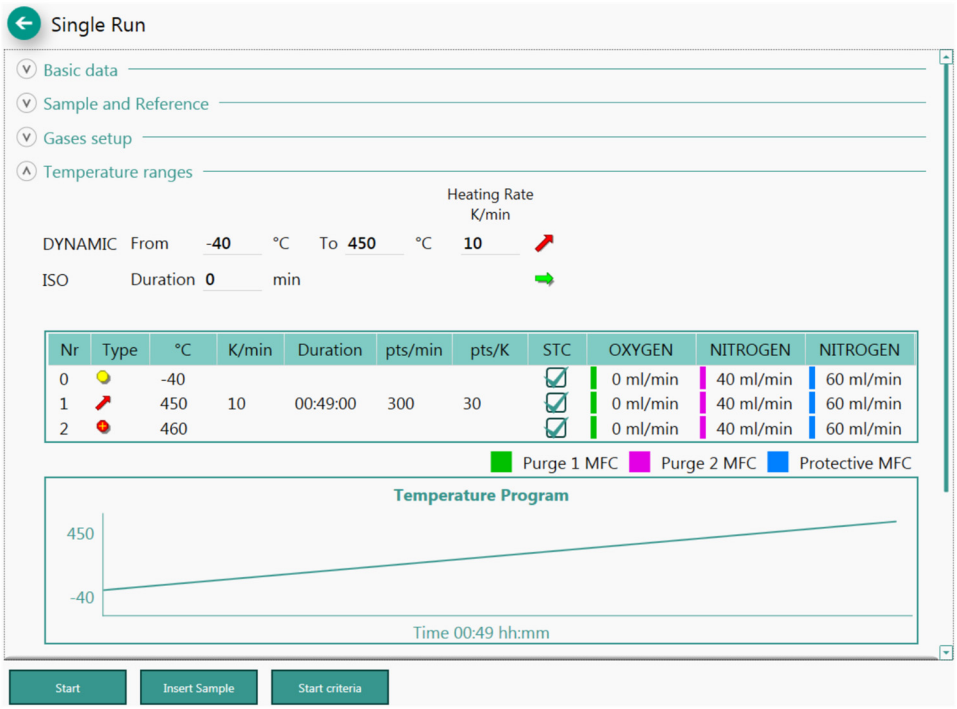


After finishing a measurement the program switches to the setpoint mode, if activated before. In this mode all predefined gas settings are activated. If the setpoint mode is not activated the gas settings from the measurement are still active after finishing the measurement (except O.I.T. mode). According to the temperature program and measurement conditions (crucible type, gases, …) the software select a suitable calibration file for the measurement. If no calibration file is found the measurement can also be carried out (without calibration).

Single Run

For starting a single run press

It is possible to define one dynamic and one isothermal segment.

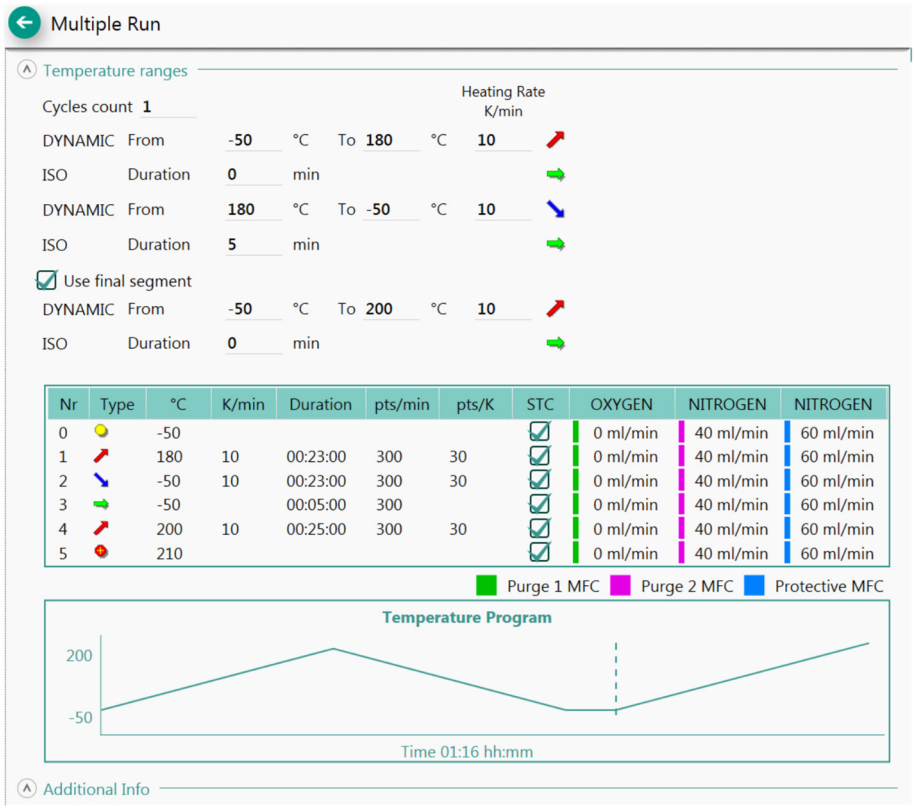


Example for temperature ranges

Multiple Run

For start of a multiple run press

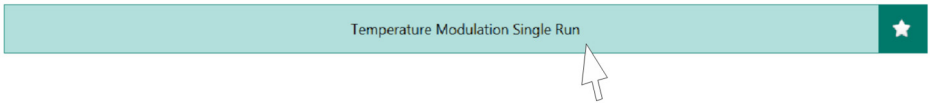
Possibility to define two dynamic and two isothermal segments that can be repeated (cycle count).



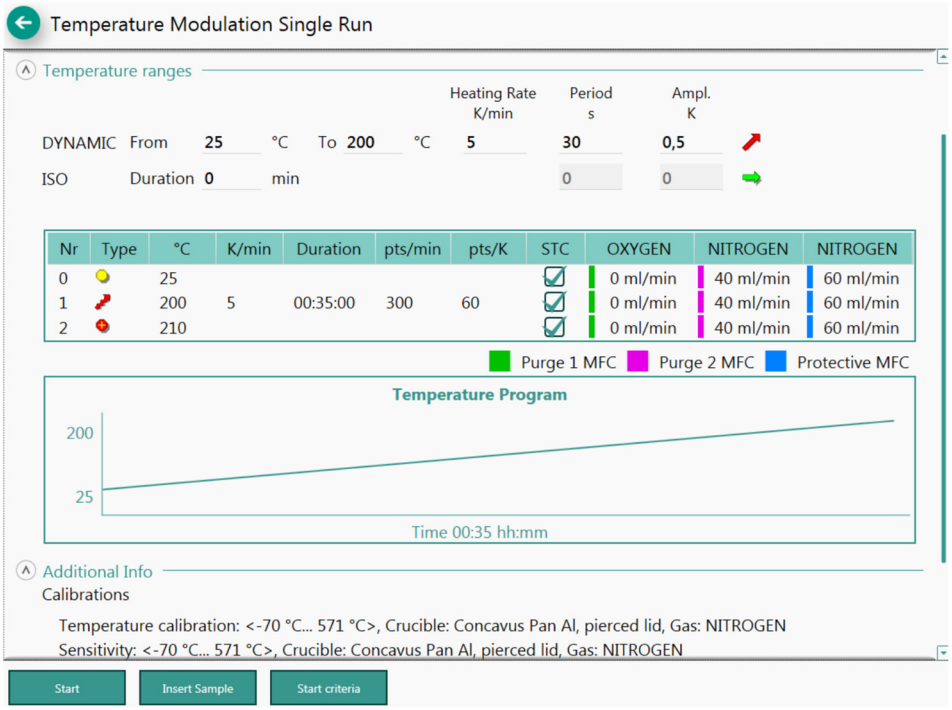
Example for temperature ranges

TM-DSC Single Run

This software feature is optional available.



Define one modulated dynamic and one modulated isothermal segment is possible.



Predefined dialog for TM-DSC measurements

Temperature-modulated DSC is the method of the thermal analysis similar to the usual DSC, but the temperature program has harmonic oscillations which superposes the constant underlying dynamic or isothermal temperature program. Typical temperature amplitudes are 0.2 to 1K, period 10s to 60s.

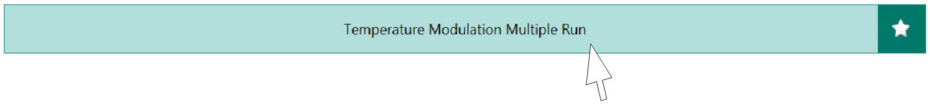
In Proteus analysis software TM-DSC method allows to make fourier analysis and to find underlying (total) signal DSC_0, amplitude A_{DSC}, phase shift φ between oscillating heating rate and measured DSC, reversing and nonreversing DSC.

During temperature modulated DSC measurement all predefined gases are the same for all segments. Purge 1 and/or purge 2 and protective must be activated.
A connected cooling device is absolutely required.

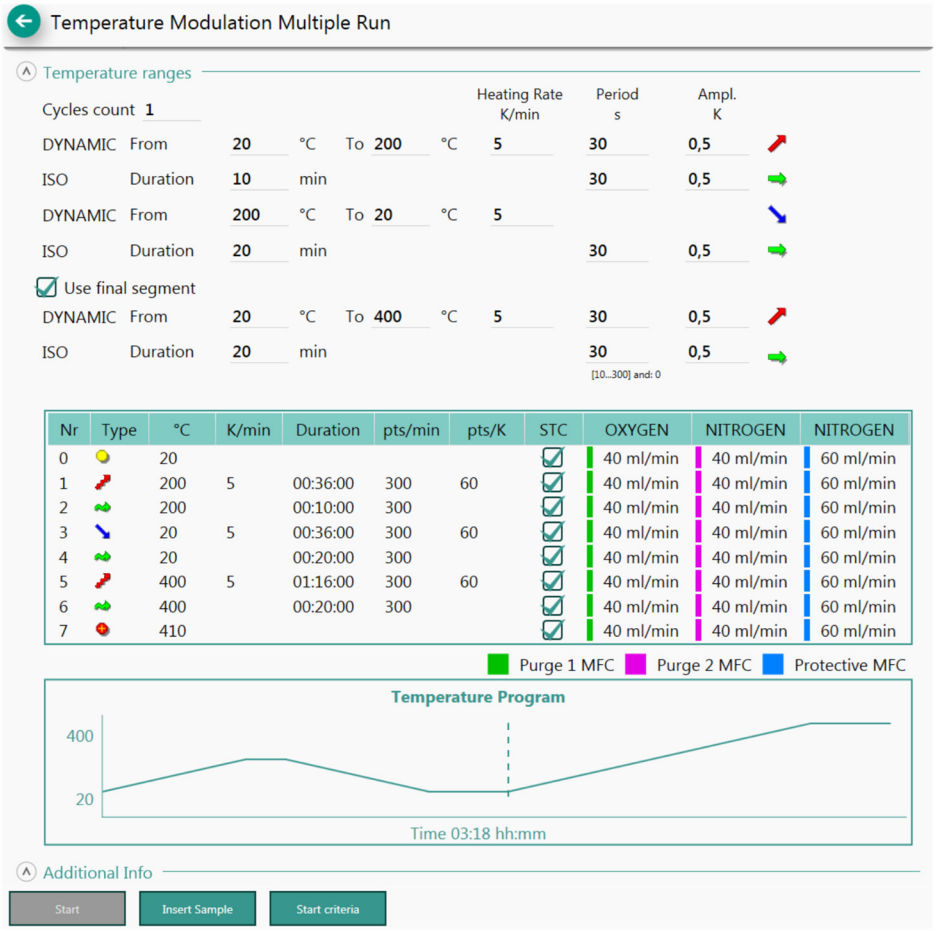
NOTE: A suitable heat flow calibration is always necessary!

TM-DSC Mutiple Run

This software feature is optional available.



It is possible to define two modulated dynamic and two modulated isothermal segments that can repeated (cycle count).



Temperature-modulated DSC is the method of the Thermal analysis similar to the usual DSC, but the temperature program has harmonic oscillations which superposes the constant underlying dynamic or isothermal temperature program. Typical temperature amplitudes are 0.2 to 1K, period 10s to 60s.

In Proteus analysis software TM-DSC method allows to make Fourier analysis and to find underlying (total) signal DSC_0, amplitude A_{DSC} and phase shift φ between oscillating heating rate and measured DSC, reversing and nonreversing DSC.

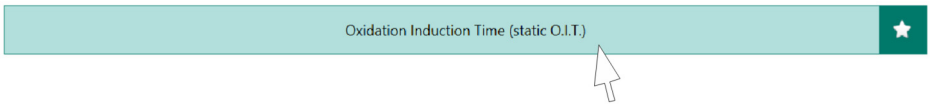
During temperature modulated DSC measurements all predefined gases are switched on for all segments. The use of purge 1 is optional, purge 2 and protective must be activated.
A connected cooling device is absolutely required.

NOTE:

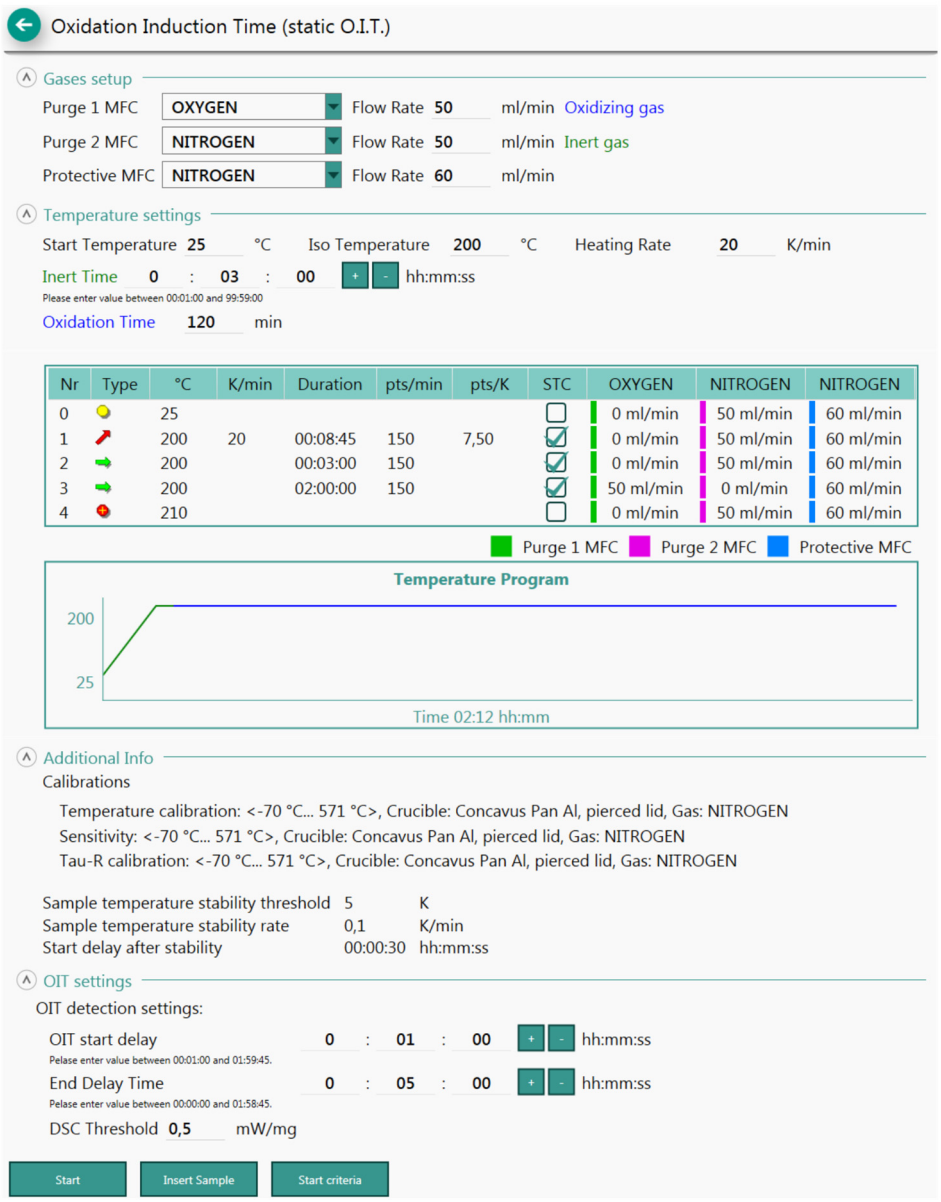
Temperature modulation cannot be applied for cooling segments!

A suitable heat flow calibration file is always necessary!

Static Oxidation Induction Time (O.I.T.)



Predefined dialog for starting O.I.T. measurements.



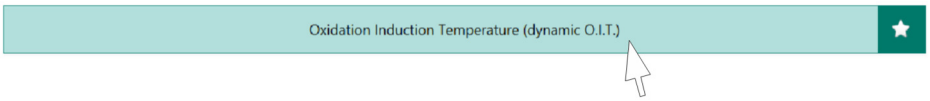
Example for O.I.T. static settings

The wizard O.I.T. static enables the definition of a program in which you can define a gas switch from inert to oxidizing in an isothermal segment after a dynamic heating.

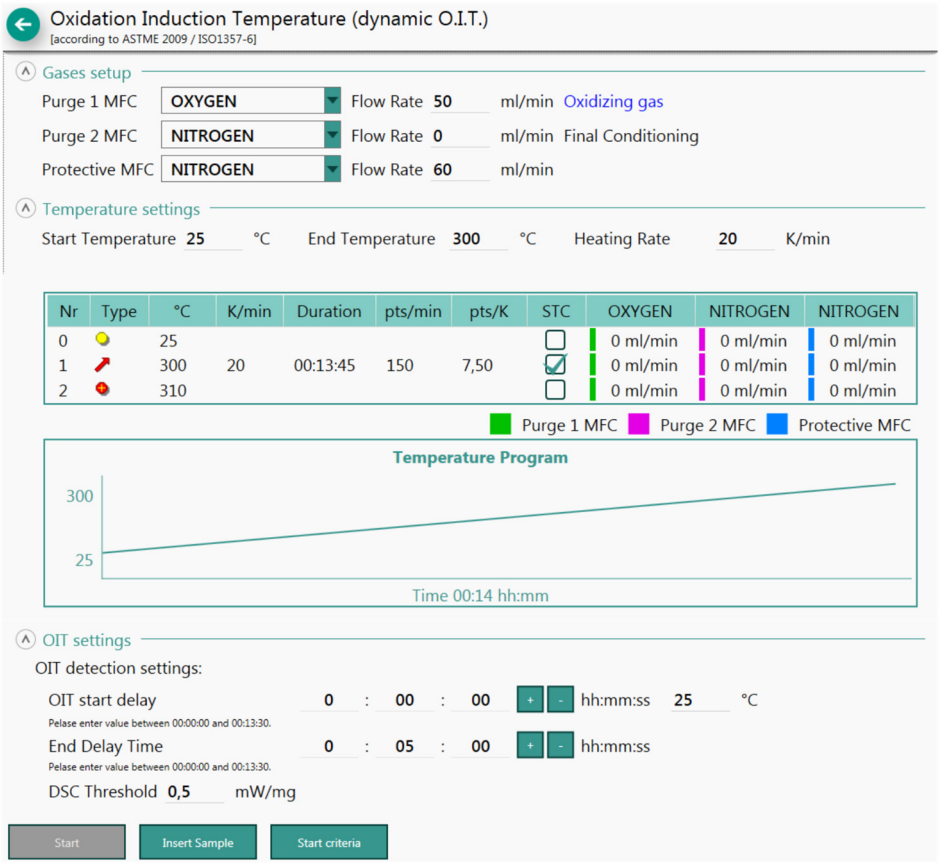
- The colours in the figure show the active purge gases. The protective gas remains permanently on.
- The O.I.T. start and end delays refer to the oxidation segment.

- For O.I.T. it is strongly recommended to have a valid heat flow calibration file available. The O.I.T. experiment will be controlled using the DSC threshold value in mW/mg. If no valid sensitivity file is found a threshold in µV/mg will be checked.
- During the O.I.T. measurement the actual DSC signal is monitored and compared to the DSC signal after the O.I.T. start delay. The termination of the measurement occurs as soon as an oxidation reaction reaches the predefined DSC threshold and the end delay time is finished.
- After finishing the measurement the program switches automatically to the setpoint mode, if activated before. In this mode all predefined gas settings are activated. If the setpoint mode is not activated the oxidation gas is switched off and the inert gas is activated.

Dynamic Oxidation Induction Time (O.I.T.)



Predefined dialog for starting O.I.T. measurements.

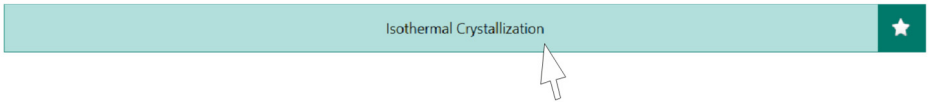


Example for O.I.T. dynamic settings

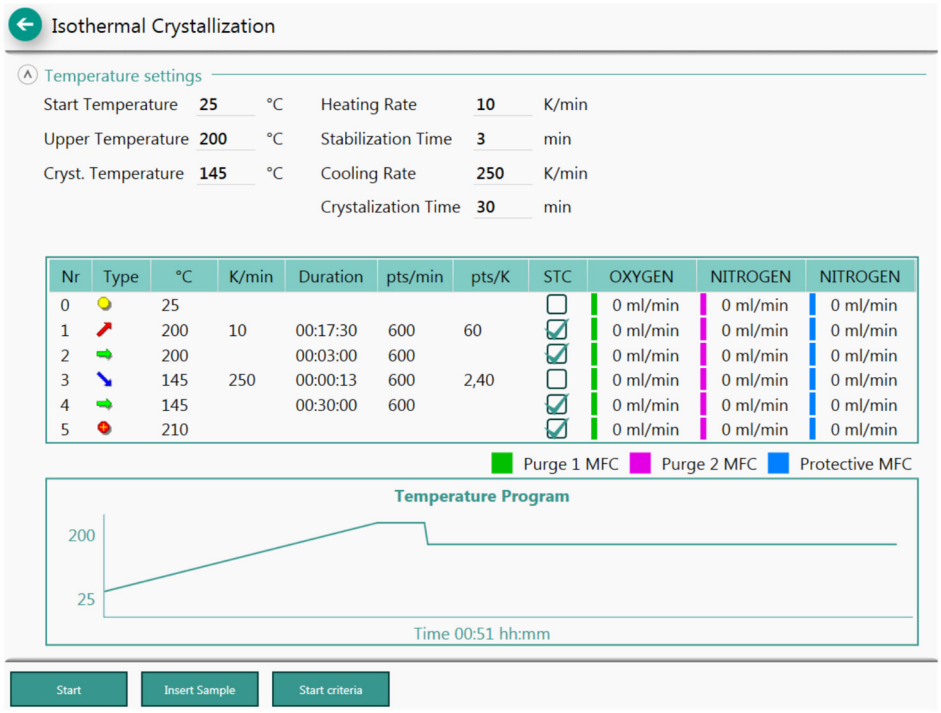
The wizard O.I.T. dynamic enables the definition of a program in which you can define an oxidizing gas in a dynamic heating segment.

- A single measurement is carried out. During the measurement the oxidation gas (purge 1) and protective gas are permanently switched on.
- The O.I.T. monitoring of the DSC signal starts after finishing the start O.I.T. start delay.
- For O.I.T. it is strongly recommended to have a valid sensitivity calibration file available. The O.I.T. experiment will be controlled using the DSC threshold value in mW/mg. If no valid sensitivity file is found a threshold in µV/mg will be checked.
- During the O.I.T. measurement the actual DSC signal is monitored and compared to the DSC signal after the O.I.T. start delay. The termination of the measurement occurs as soon as an oxidation reaction reaches the predefined DSC threshold and the end delay time is finished.
- After finishing the measurement the program switches automatically to the setpoint mode, if activated before. In this mode all predefined gas settings are activated. If the setpoint mode is not activated the oxidation gas is switched off and the inert gas is activated.

Isothermal Crystallization



Predefined dialog for isothermal crystallization measurements.



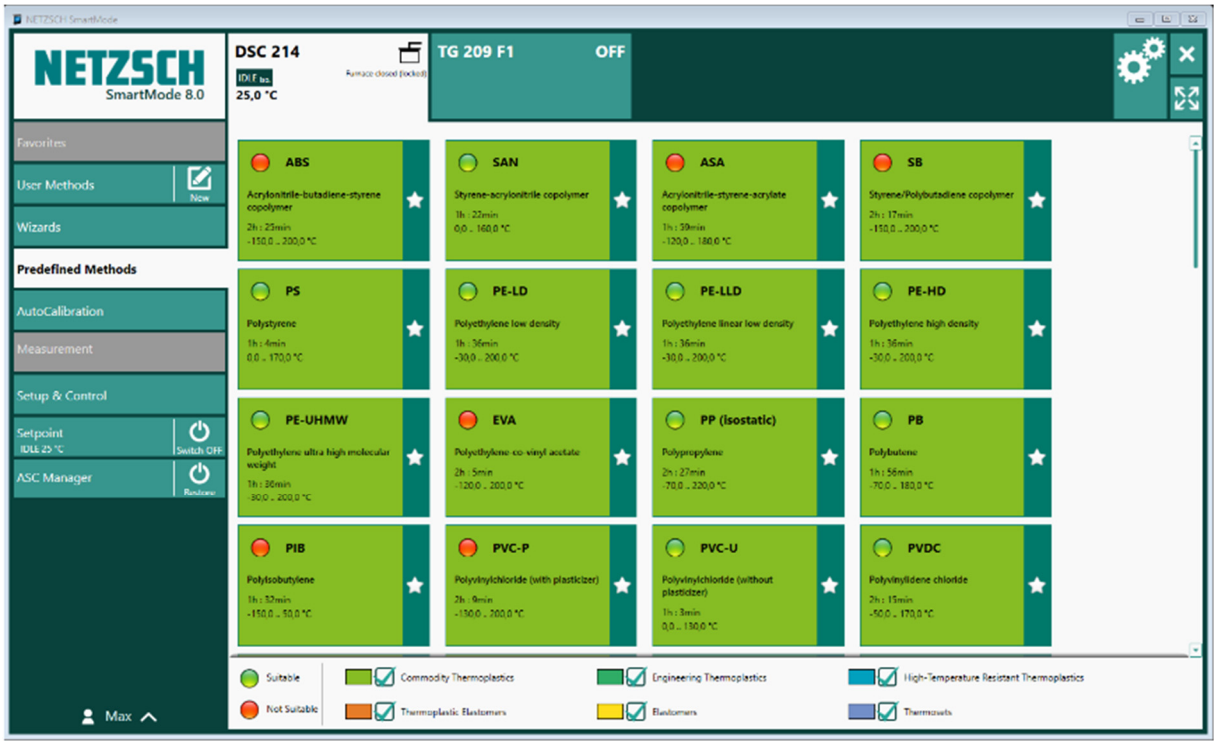
Example for isothermal crystallization

This wizard helps for defining experiments for the investigation of crystallization of semicrystalline polymers. The method includes a very fast cooling segment from an upper temperature (molten polymer) to a crystallization temperature followed by an isothermal segment in which crystallization occurs.

Additional Information:
During the isothermal crystallization all predefined gases are switched on for all segments. The use of purge 1 is optional, purge 2 and protective must be activated. During the cooling segment the STC (Sample Temperature Controller) is switched off.

SmartMode Tab – Predefined Methods

In this tab you can find a list with predefined measurement methods for the 66 most commonly used neat polymers. No additional methods for blends or special compounds are included but the standard methods are also applicable.



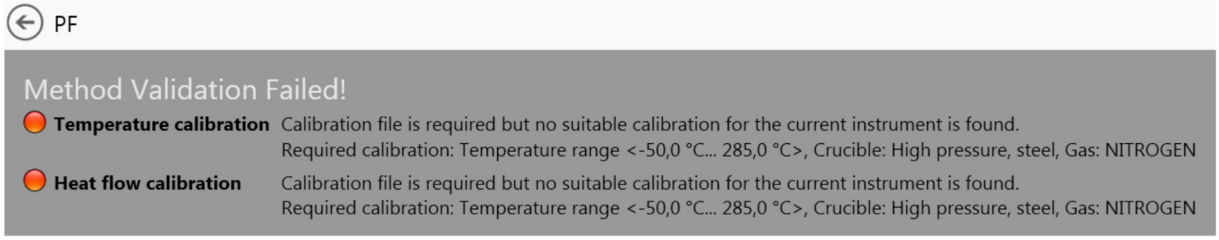
Click on the method to see measurement settings.

The predefined methods include a temperature range to investigate all typical effects of the selected polymer (glass transition, melting, recrystallization or curing). A method contains two heating and one cooling segment with typical heating rates of 10 K/min or 20 K/min. Moreover, the gas flow is also predefined. User can select crucible type and has to enter basic sample data. Predefined methods can only be started if the customer DSC fulfils the predefined cooling requirements.

NOTE:

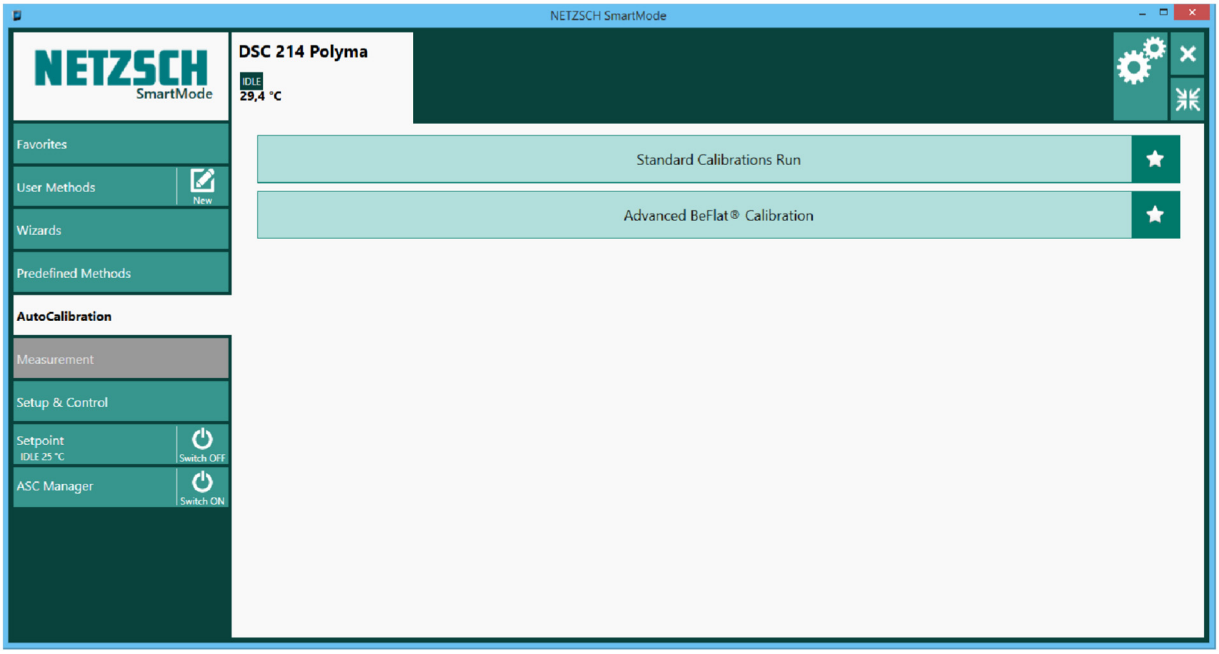
Method with green ball fit to your hardware.
Method with red ball cannot be used (e.g. missing cooling device).

It is absolutely necessary that temperature and sensitivity calibration in the temperature range of the selected polymer method is available!



SmartMode Tab – AutoCalibration

AutoCalibration offers a complete new calibration routine which includes defined temperature programs and an autonomous curve analysis for the melting standard materials.



To perform a calibration use of AutoCalibration function is necessary.

Standard Calibrations Run:

The 'Standard Calibrations Run' includes calibrations for temperature, enthalpy, Tau-R (optional) and automatic evaluation of onset and area of DSC melting peaks.

Advanced DSC-BeFlat® Calibration:

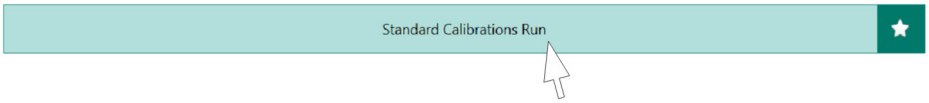
User can perform a calibration to ensure flat baselines for DSC instruments.

Prior to the measurement, the calibrations of the instrument are managed with the module Standard Calibrations Run. It reminds when a calibration is necessary and carefully guides through the calibration procedure.

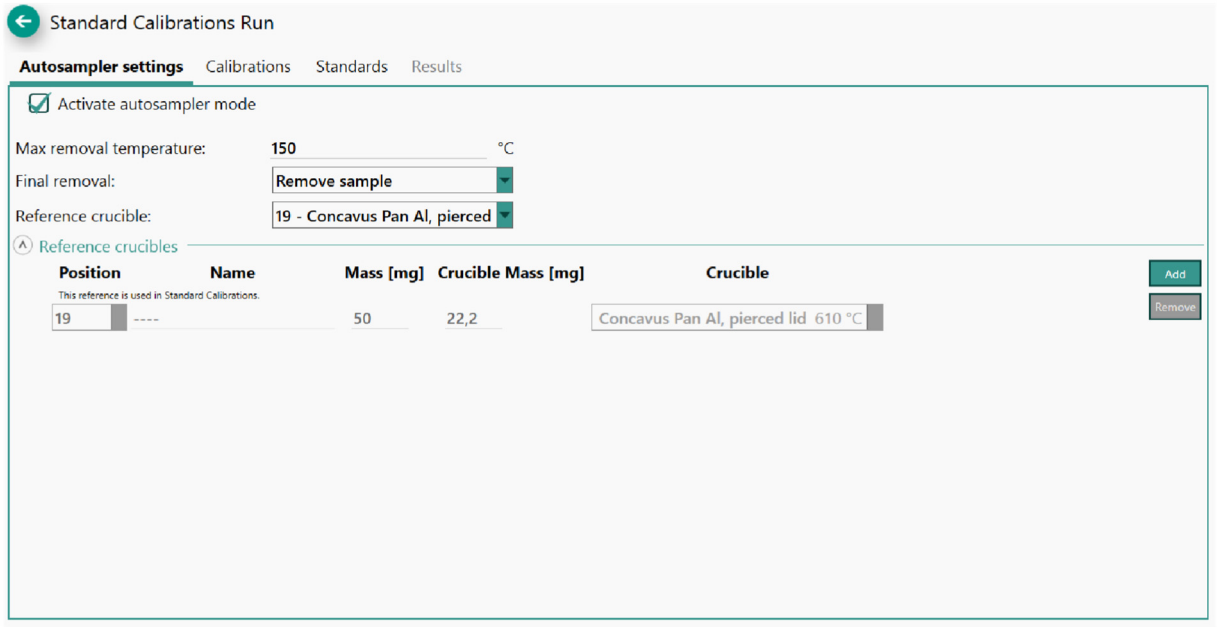
Being interested in more details we recommend to operate "Set point", "Advanced DSC BeFlat Calibration" and "Autocalibration" in the Expert modus of the Proteus software and read the description in the help system which is context sensitive linked to it.

Standard Calibrations Run

The ‘Standard Calibrations Run’ includes calibrations for temperature, enthalpy, Tau-R (optional) and automatic evaluation of onset and area of DSC melting peaks.

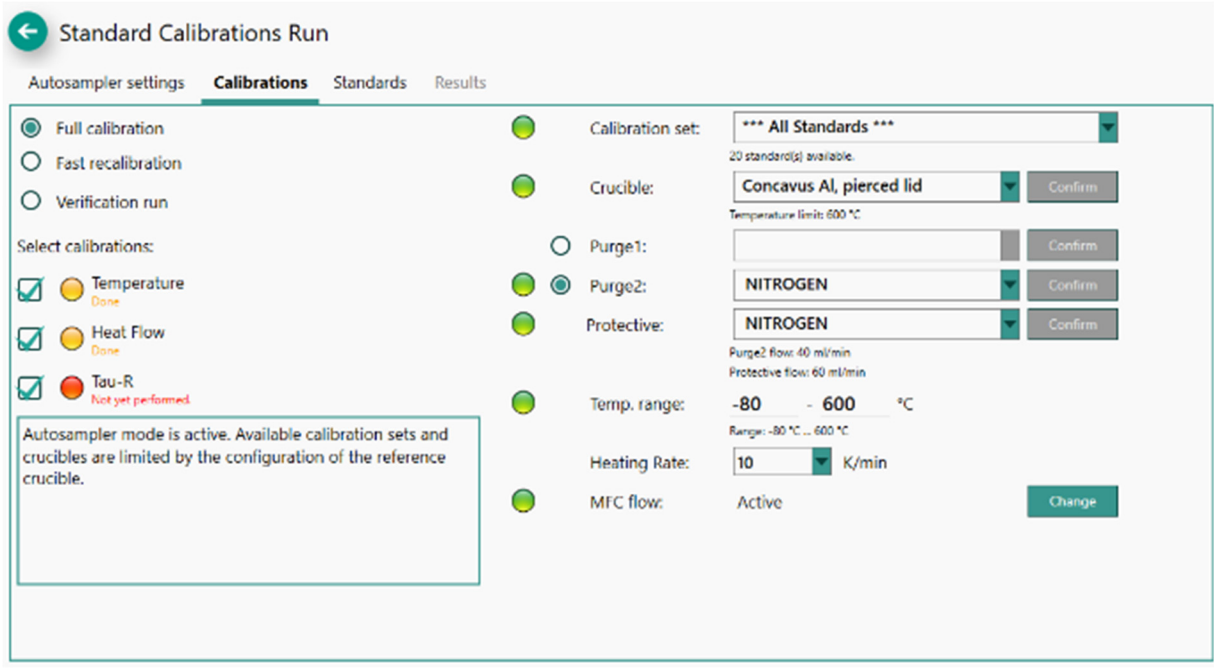


Autosampler settings



With ‚active autosampler mode‘ the selected reference crucible type is taken as a preselection for the ‘calibration set’ to be defined on ‘calibrations’ folder. ➔ Only calibration sets which fit to reference crucible type can be selected in ASC mode.

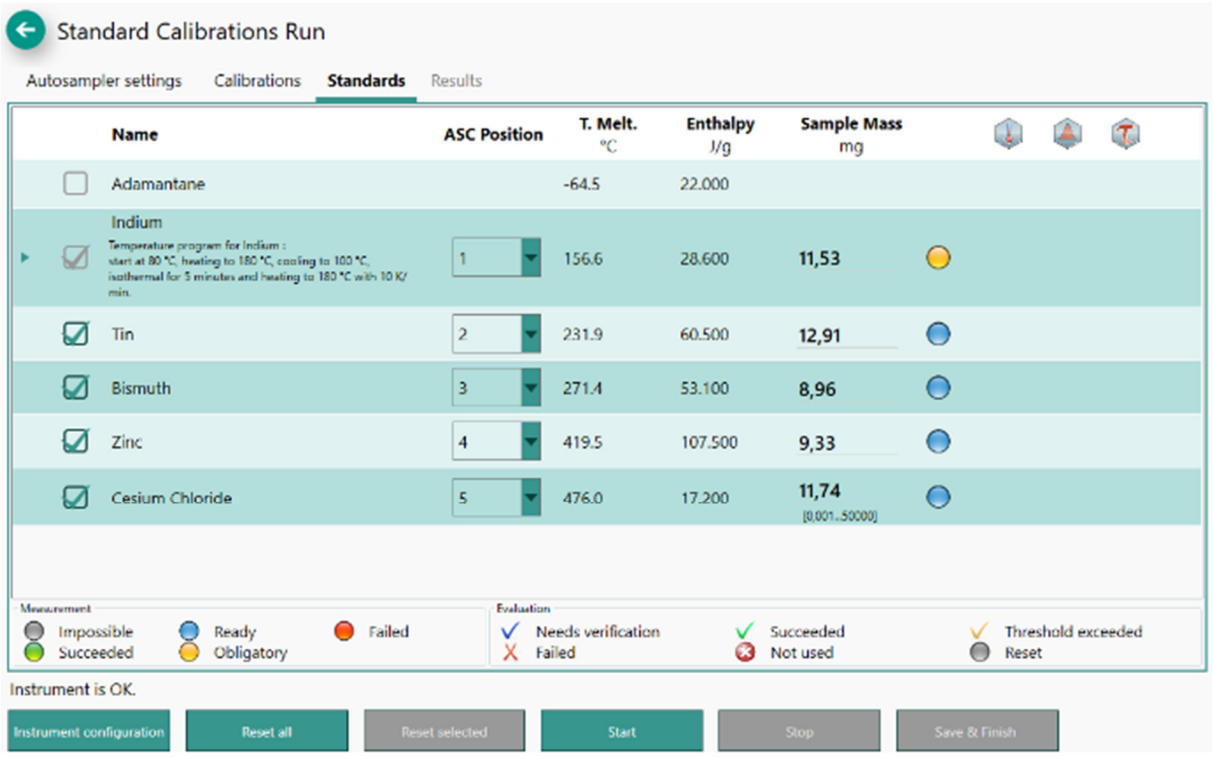
Calibrations



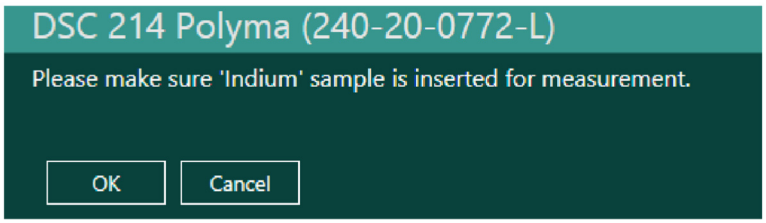
Later on, only calibrations which fit the selected “crucible”, can be used for the corresponding methods.

Full calibration:	Execute a complete calibration of the instrument using a standard set.
Fast calibration:	Edit an existing calibration by re-measuring one calibration standard.
Verification run:	Check an existing calibration by re-measuring one or more calibration standard(s). An already existing calibration will be used. The results can be compared with literature values.

Standards



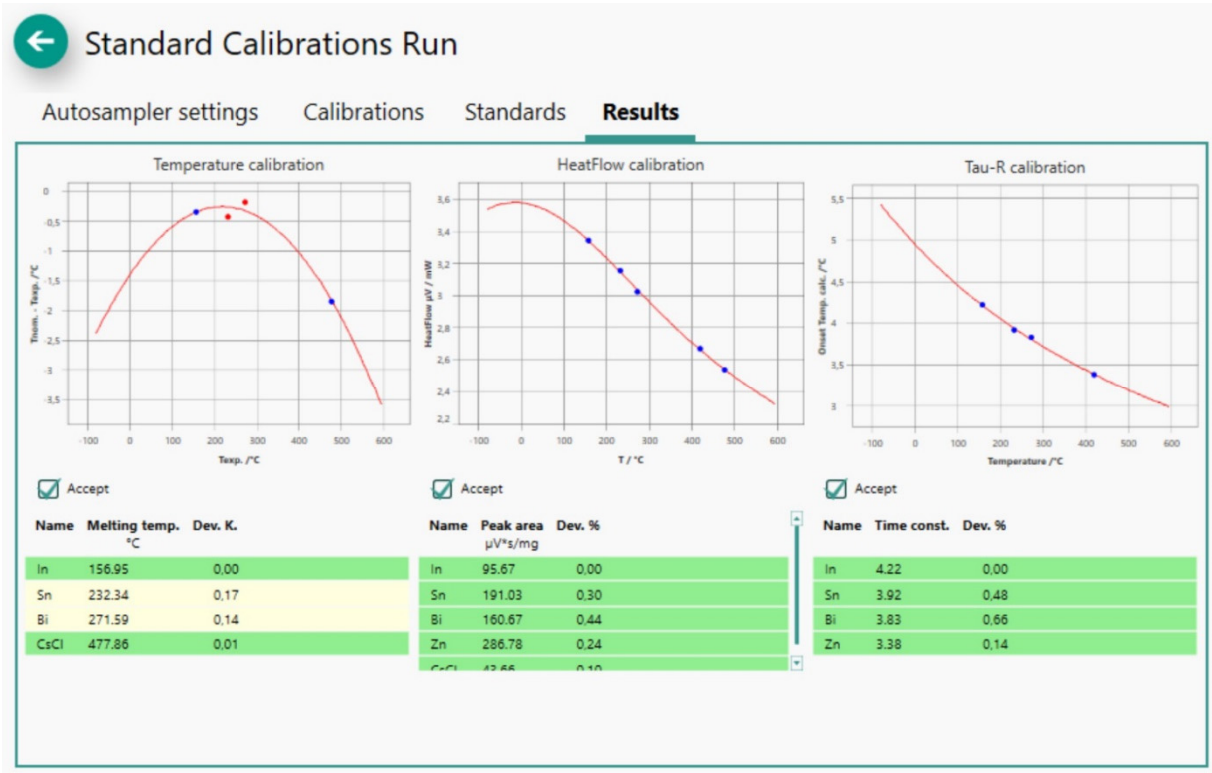
The calibration standards are listed with increasing temperature and predefined temperature programs. Adamantane can only be measured with connected LN2 cooling device. The measurement of Indium is absolutely required!



NOTE: Ensure that the correct sample is inserted.

NOTE: NETZSCH takes no warranty for overheating of wrong standard materials!

Results



Visual check of the calibration curves: Measuring points marked with yellow checkmarks, which indicate exceed specified threshold values, can be excluded from the curve calculation at standards tab (indicated as a red point in graph at results tab).

Note: If a single result has to be excluded from the calculation, it may be “reset” in tab “Standards” by clicking it´s green checkmark.

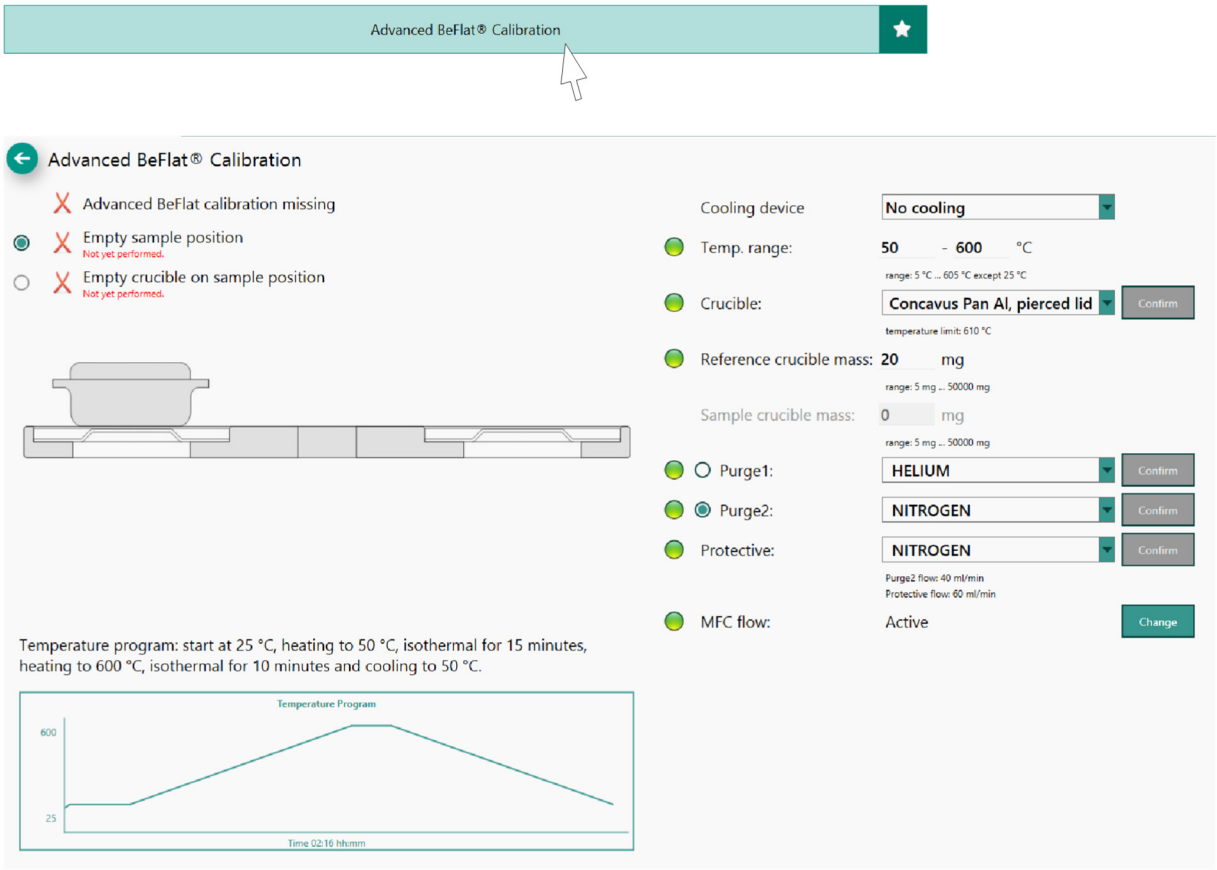
Set “Accept” checkmarks if the calibration curves are valid to be used later.

Finally press “Save & Finish” button to finish and close the calibration process.

If necessary adaptions may be applied opening the saved calibration files in the separate tools temperature, sensitivity or TAU-R calibration.

Advanced DSC-BeFlat Calibration

The user can perform a calibration to ensure flat baselines for DSC instruments here.



The DSC-BeFlat calibration allows a program controlled creation of a DSC baseline in 2 steps.

Being interested in more details we recommend to operate “Set point”, “Advanced DSC BeFlat Calibration” and “Autocalibration” in the Expert modus of the Proteus software and read the description in the help system which is context sensitive linked to it.

206030005 80032061 2020-12-03

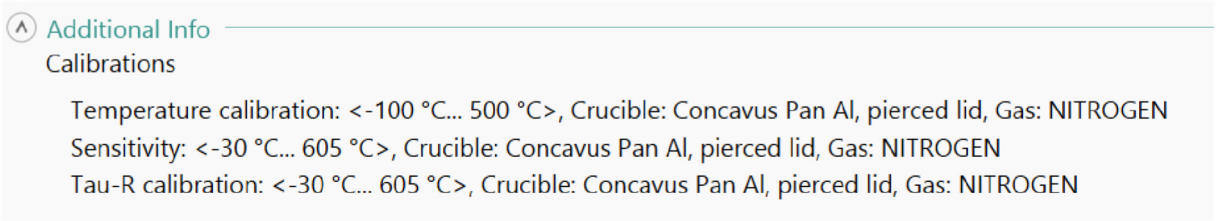
How calibrations are applied in Smart Mode measurements

When performing Smart Mode measurements the Proteus software mostly requires calibration files which will later be applied to correct the measurement data and the instrument control.

A calibration created for this instrument and sensor type is suitable if the temperature range of the prepared measurement is covered by the calibration file and gas types and crucible material are adequate.

- When operating with Wizards or Predefined Methods Proteus always searches for the most suitable calibration files and auto selects the respective calibration files.
- When operating with User Methods Proteus will use the calibrations defined inside the user method (if available in the directory …\calibrations…). If no calibration file is found in this directory then measurement can be executed without calibration.

Found calibrations file are indicated as below:

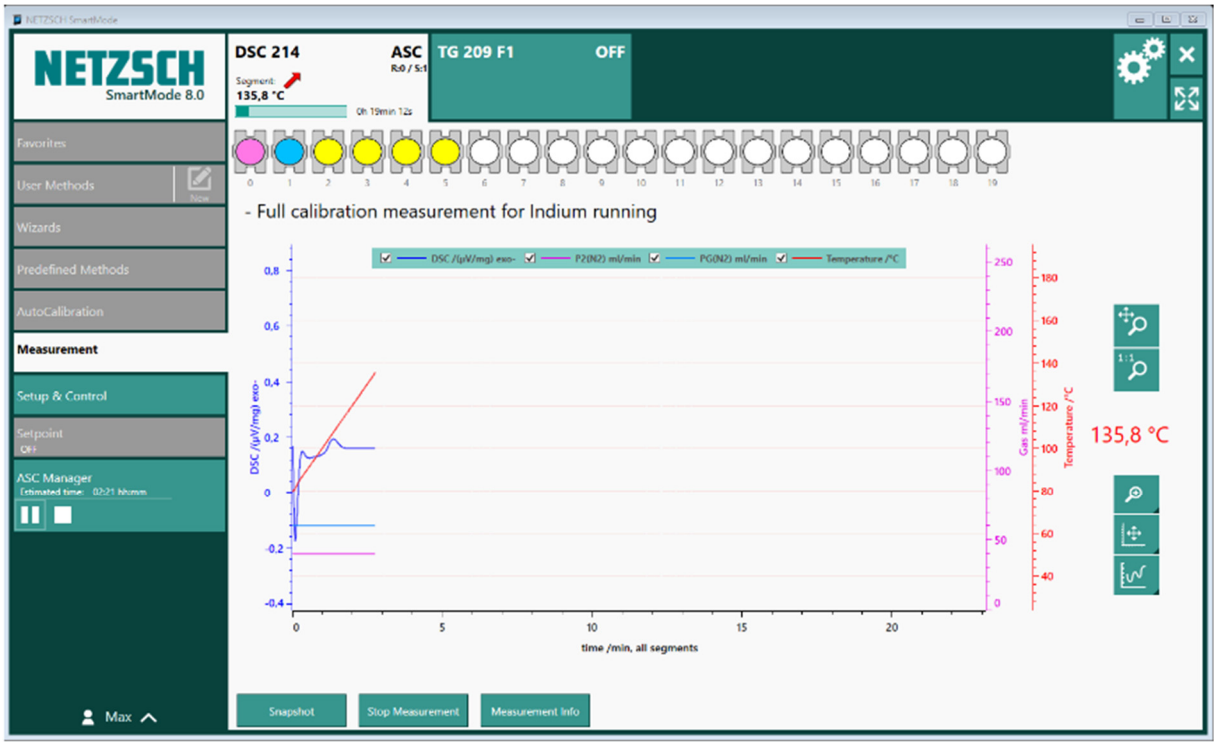


The Auto Selection of the calibration files in the Smart Mode is always active and can never be deactivated. For the individual selection of calibration files you have to use the Expert Mode.

If more than one calibration file is available in (…\calibrations …) and the 'most suitable' calibration file Proteus selects automatically is not 'the best one', such (historical calibration files) may be 'moved to archive'. To perform this operation access the 'File open' dialog in the calibration tools (Temperature Calibration, Sensitivity Calibration or TAU-R calibration) mark this file and press button 'Move to archive'. If such calibrations are moved to archive they will be not used and found automatically. If necessary these archived files may be restored any day.

SmartMode Tab – Measurement

General



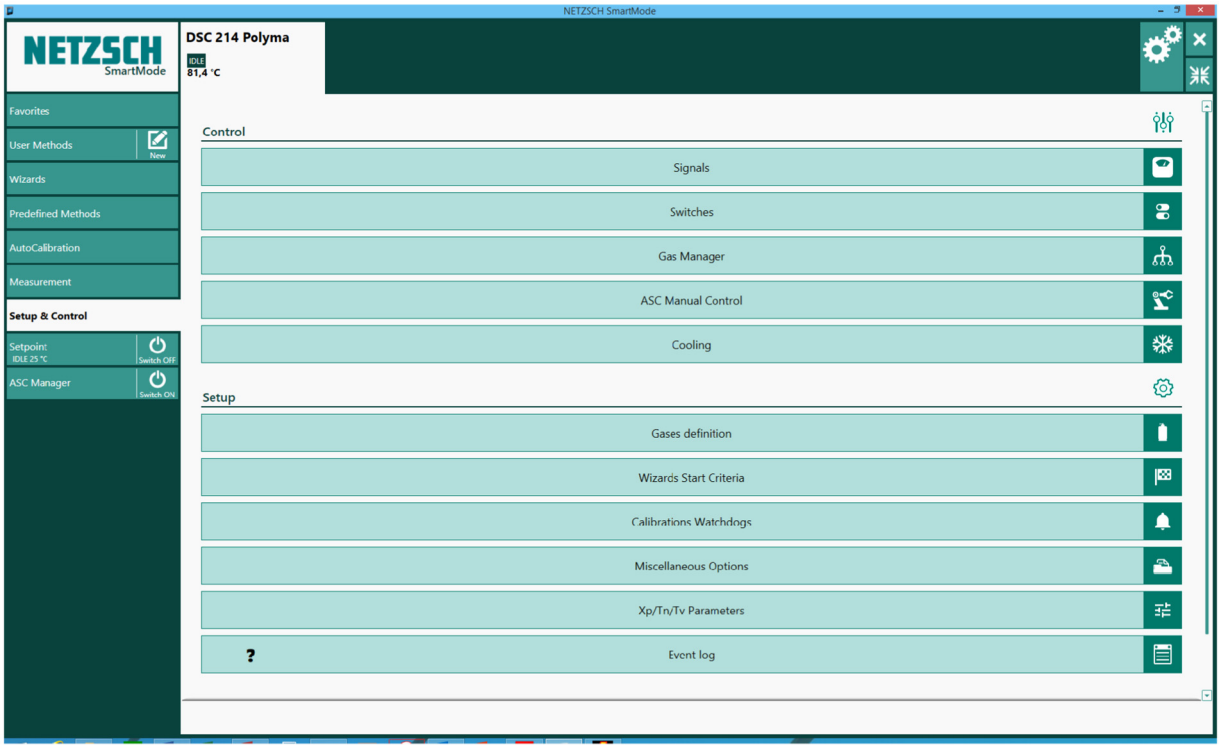
The tab Measurement shows a graphical chart of the recent running measurement. If no measurement is running, the tab is grey and cannot be activated.

An active measurement shows DSC signal, actual temperature and gas flow as well as the required time to finish measurements. If the AutoEvaluation was activated for a method or a wizard, an evaluated curve will be shown at the end of the measurement.

From this tab the user can switch over into the NETZSCH Analysis software to perform advanced evaluation or already perform a snapshot during the run.

If a method (User or Predefined) or a wizard includes an AutoEvaluation then the measurement analysis will be carried out directly at the end of the measurement. The data will be analyzed in a hidden form and shown in the Smart Mode Measurement window as a result. Not in ASC runs!

SmartMode Tab - Setup & Control



Control

Here you can see and check all instrument functions (e.g. gases, switches, signals…). You can also manage samples if an ASC (Autosampler) is installed.

Setup

Select type of gas, which is connected to purge 1, purge 2 and protective at “Gases definition”.

Under “Wizards Start Criteria” you can define the conditions which are applied for the automatic start of measurements even if the actual sample temperature differs from the defined new start temperature.

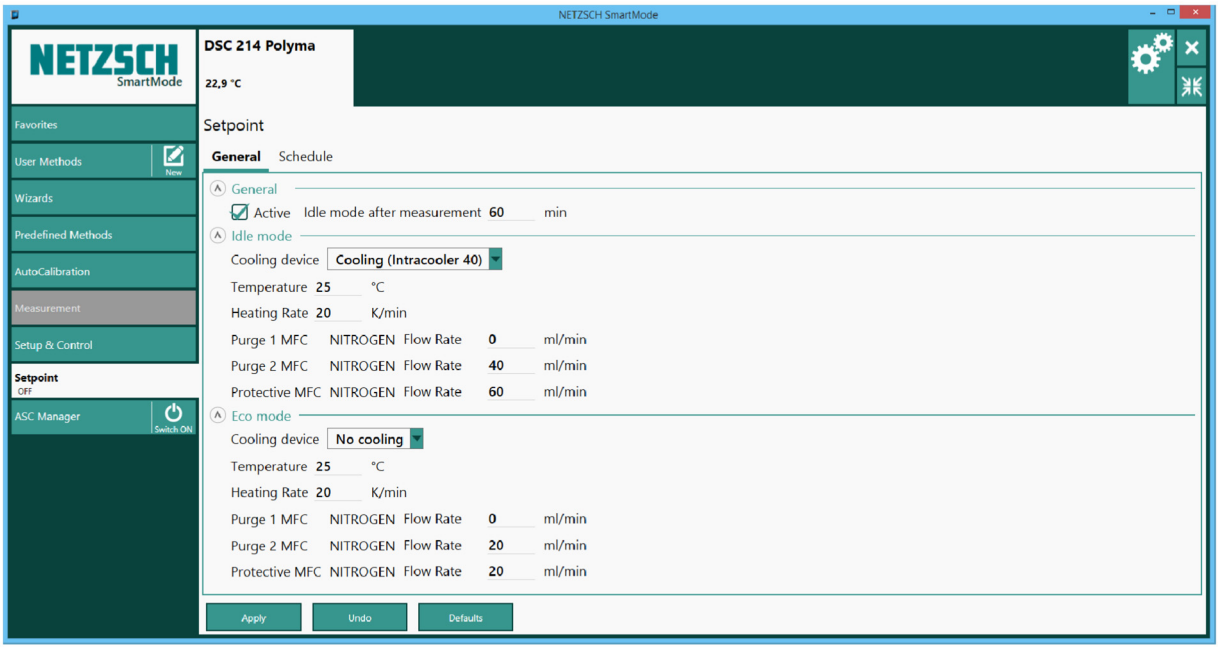
Here it is also possible to check the “Calibrations Watchdogs” which are reminders for necessary recalibrations.

Xp/Tn/Tv Parameters are predefined by NETZSCH (factory settings) and applied to all Wizards and Predefined Methods.
By means of the dialog Xp/Tn/Tv Parameters the predefined settings can be changed before or during a measurement (if necessary). These parameter changes are stored for Predefined Methods in a common parameter set. For any Wizard (e.g. Single Run, Multiple Run…) an individual parameter set is stored. The stored parameters will be applied when carrying out a Wizard or Predefined Method later.
For creation of User Methods you can use predefined parameters from NETZSCH (factory settings) or you can define Xp/Tn/Tv Parameters individually for each separate method. When performing a measurement the respective parameters will be applied.

Incidents (leak of gases, external resets,…) during Smart Mode Measurements are registered in “Event log” tab.

SmartMode Tab – Setpoint

General



The Setpoint functionality is a new feature in the Proteus software. If no measurement program is active, the software controls the status of the DSC cell regarding its temperature and gas flow. Setpoint has the aim that the DSC cell is always under controlled conditions, offering two subsequent modes.

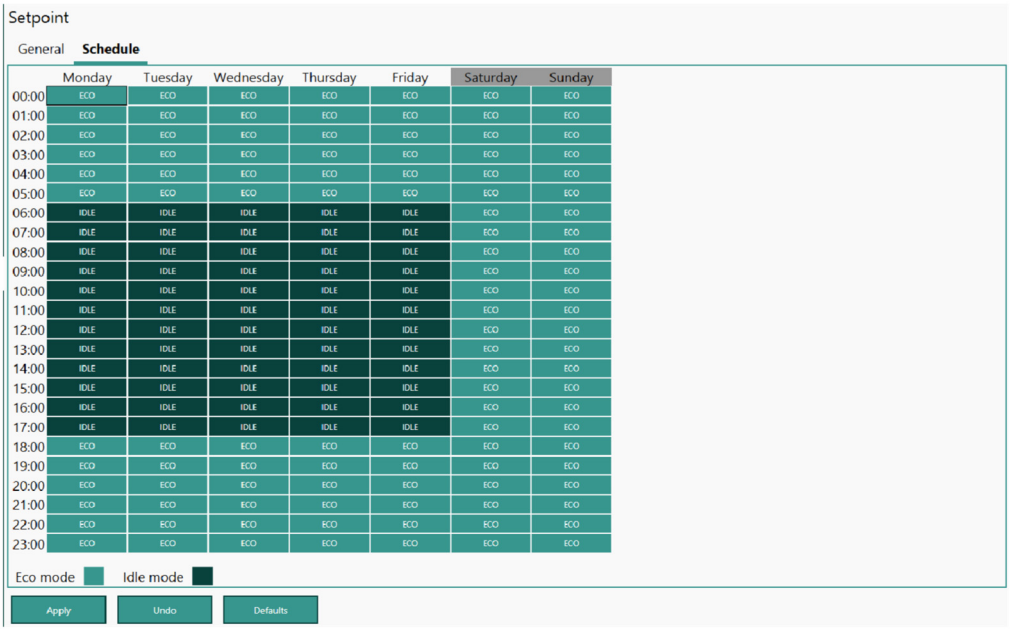
The Idle mode becomes active when starting the software and is in charge before and after the measurement to keep the DSC cell at default values, e.g. 25 °C and a gas flow of 40 ml/min purge and 60 ml/min protective.

The Economy mode can be scheduled for nights or weekends when the DSC is not in use for a longer time, to save energy and gas consumption.

A cooling device may be switched on to precool and stabilize the instrument. It is recommended to use it in Idle mode if the DSC instrument is equipped with an Intracooler. This avoids a possible freezing of the cell.

Being interested in more details we recommend to operate “Set point”, “Advanced DSC BeFlat Calibration” and “Autocalibration” in the Expert modus of the Proteus software and read the description in the help system which is context sensitive linked to it.

Schedule



The Eco mode starts according the above "Schedule" but will be started earliest when the defined "Switch time" has elapsed after the end of a measurement. Customizing the time schedule is possible at anytime.

ASC Manager (programming the ASC) (option)

The ASC (Automatic Sample Changer) must be programmed in the respective tab when selecting a method (User Methods or Predefined Methods) or defining a measurement (Wizards). In the tab ASC manager you can see the single programmed ASC measurements. Here you can define the measurement sequence and it is also possible to edit or change the single measurement parameters.

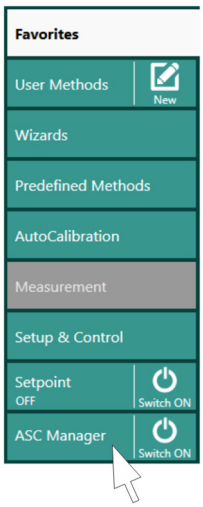
The following screenshots shows the ASC 200 which is assembled on a DSC 214 Polyma, DSC 200 F3 Maya and DSC 3500 Sirius. The screens of the ASC 400 (DSC 204 F1 Phoenix) are quite similar.

Color mode for ASC measurement status

	defined
	done
	done (analysis failed or results are out of the predefined boundaries, e.g. for quality control)
	failed
	measurement active
	reference

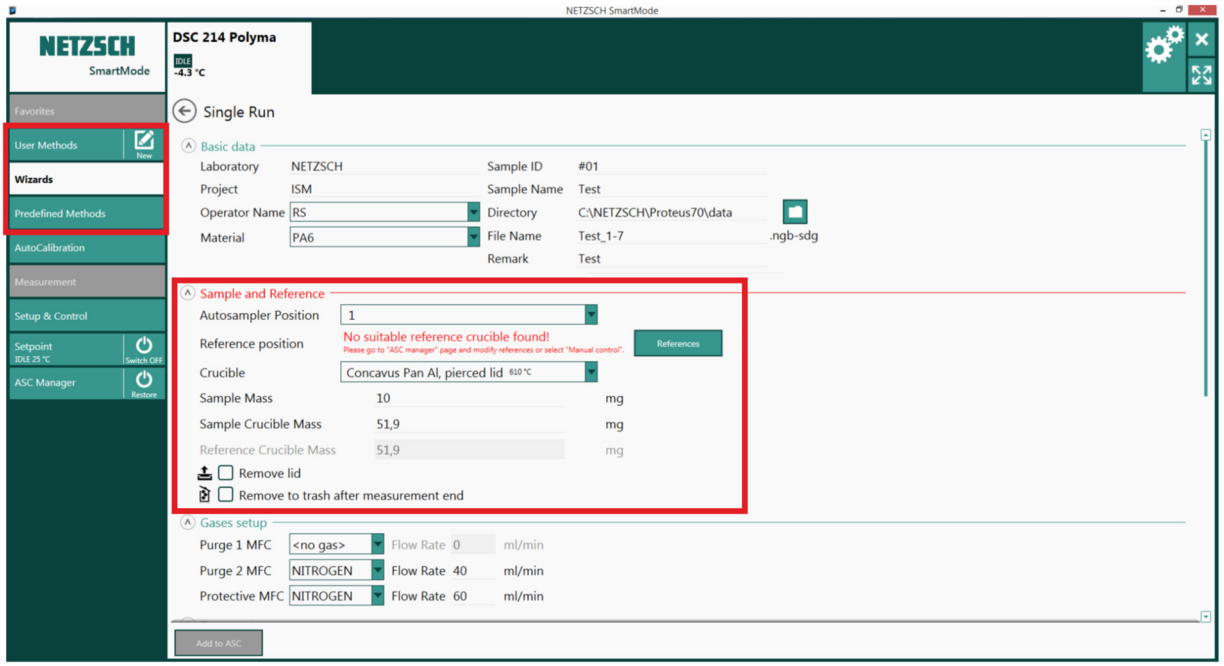
Some screens will also contain a toolbar at the lower right corner. The function of each icon is described below the specific icon.

Click “Switch ON” to activate the ASC.



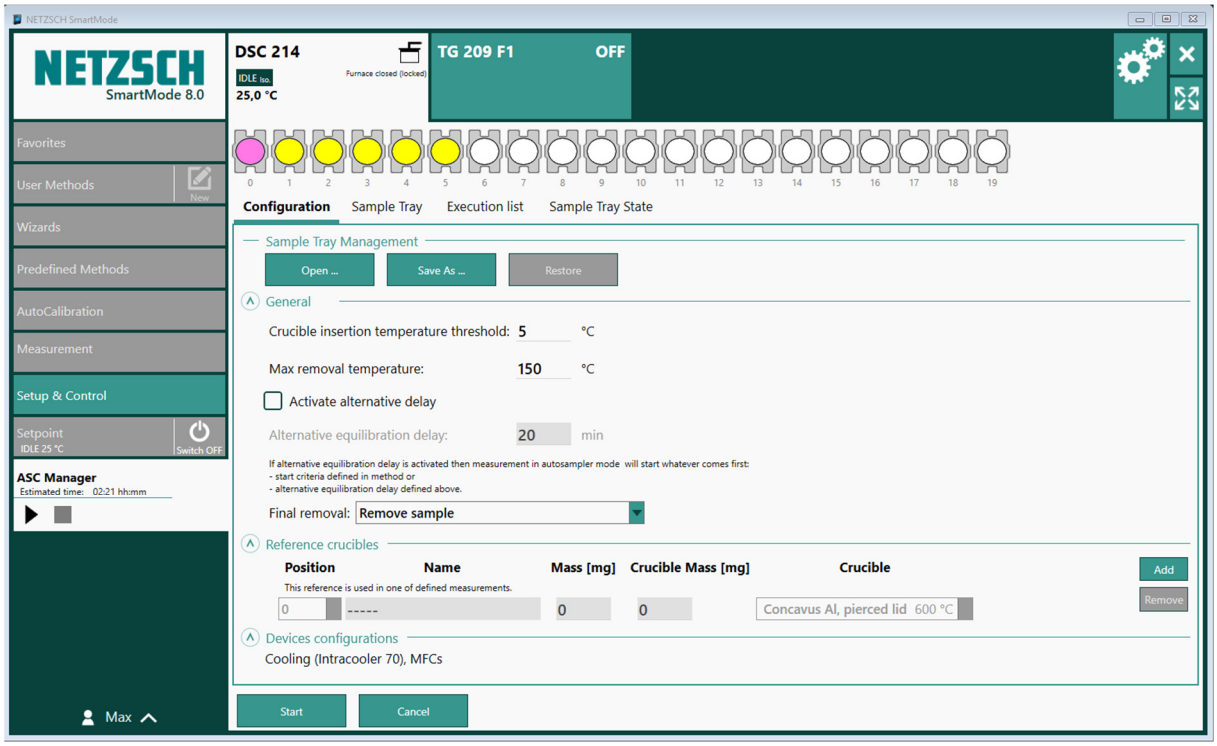
It is also possible activate the ASC by clicking “Restore”, see page 39.

DSC Instruments with ASC



Select a method (User Methods, Predefined Methods) or create a new measurement (Wizards) and define the settings of sample and reference crucibles (position and mass). The ASC manager will be switched on automatically. In this way you can define all sample tray positions. With click on the button References you can also define the reference position.

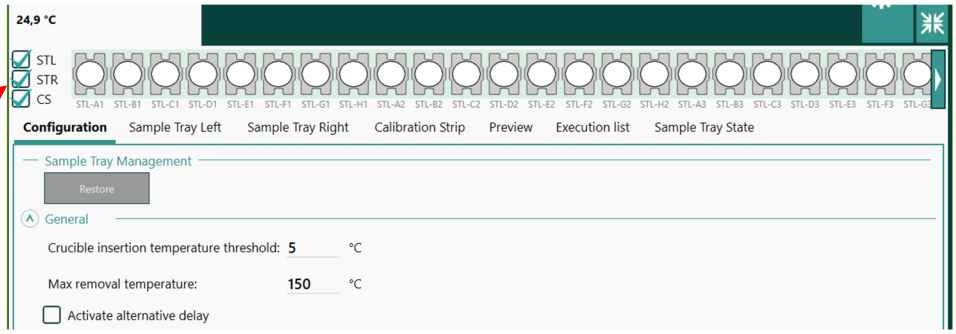
Configuration



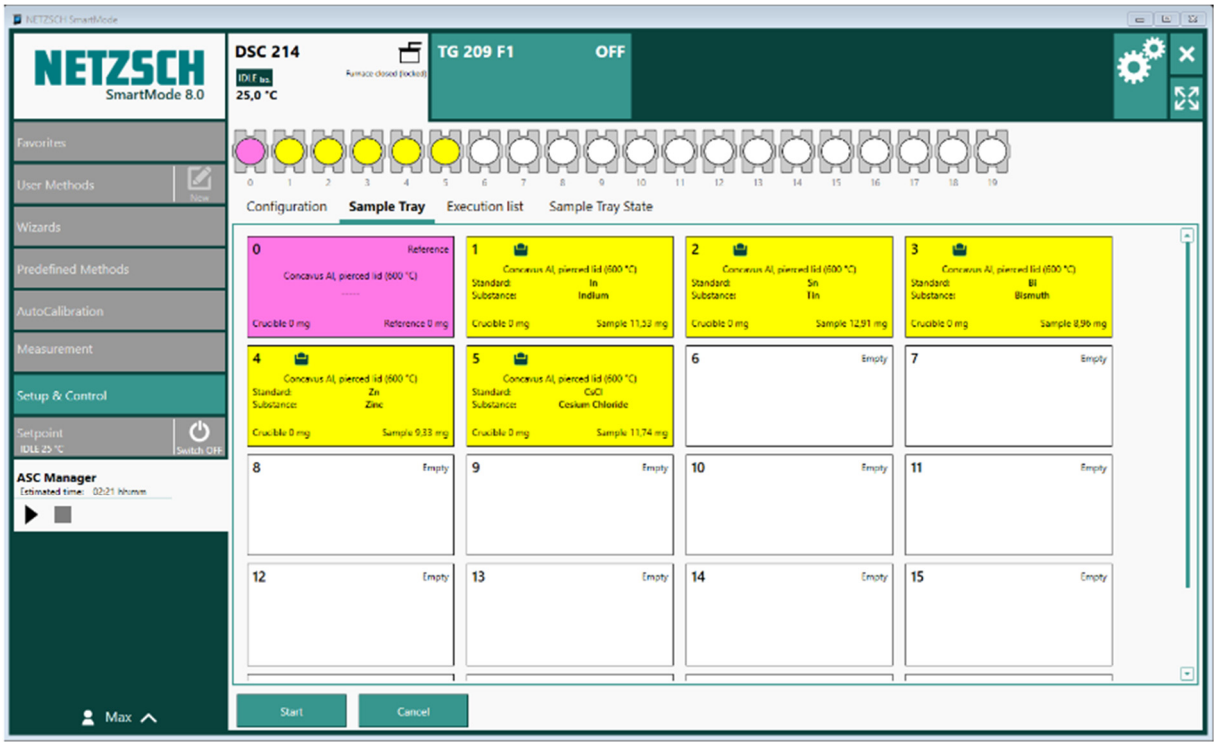
In the Configuration list of the ASC manager define the general ASC settings and the reference crucible (position, mass, crucible mass, crucible type).

When the instrument is equipped with an ASC400:

STL:
sample tray left
STR:
sample tray
right
CS:
calibration strip

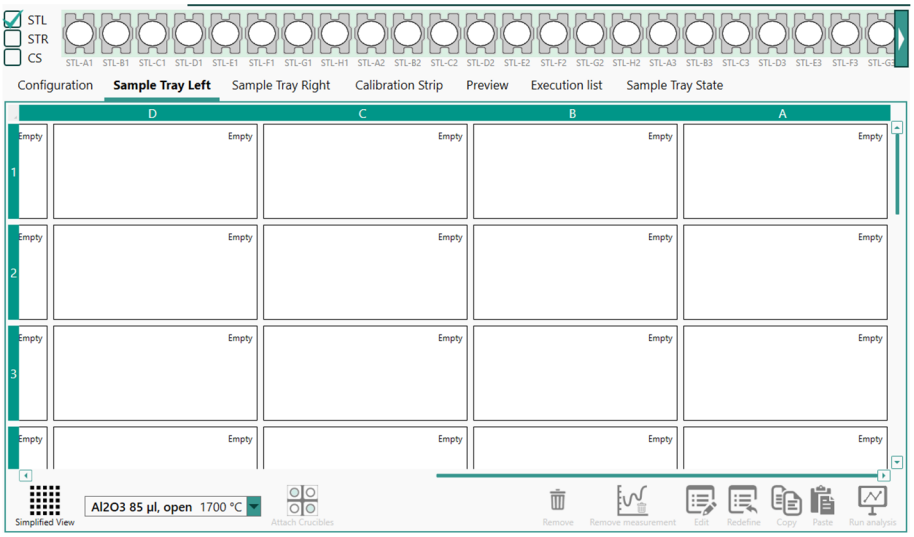


Sample Tray / Sample Tray Left - Sample Tray Right - Calibration Strip



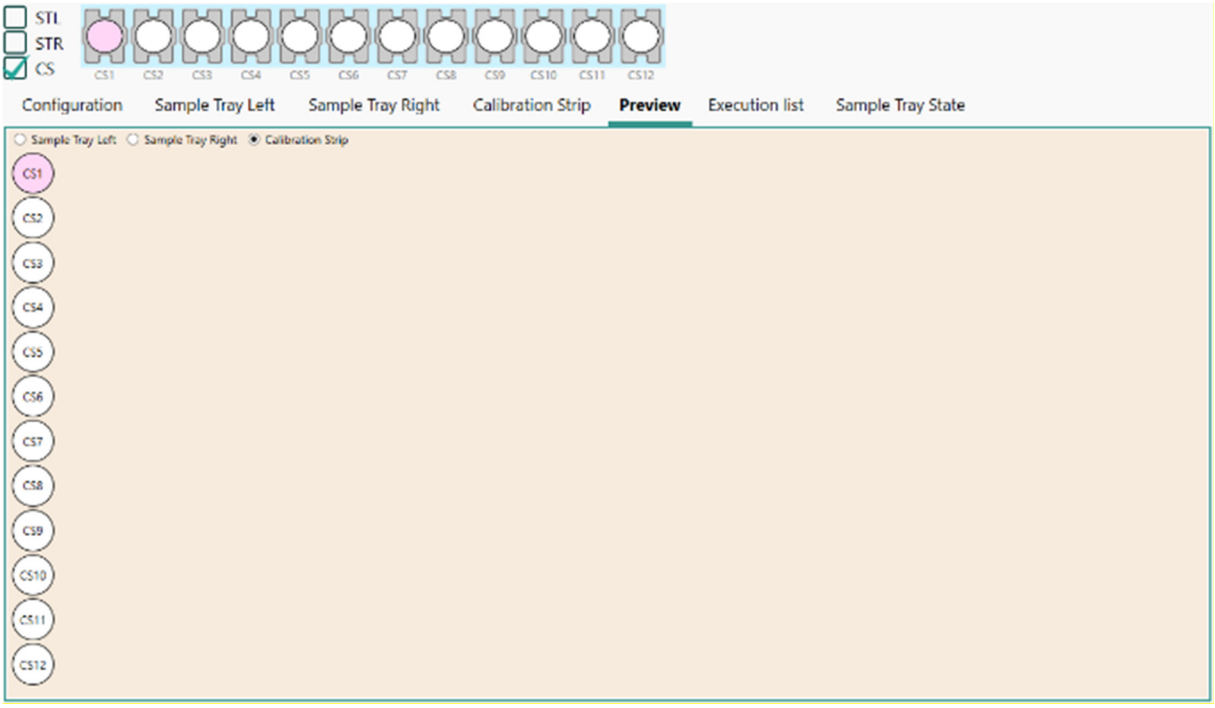
In the ASC manager you find the sample tray list with all defined positions. By means of the toolbar in the right corner each sample tray position can edited and copied/pasted.

Example screen when the instrument is equipped with an ASC400:



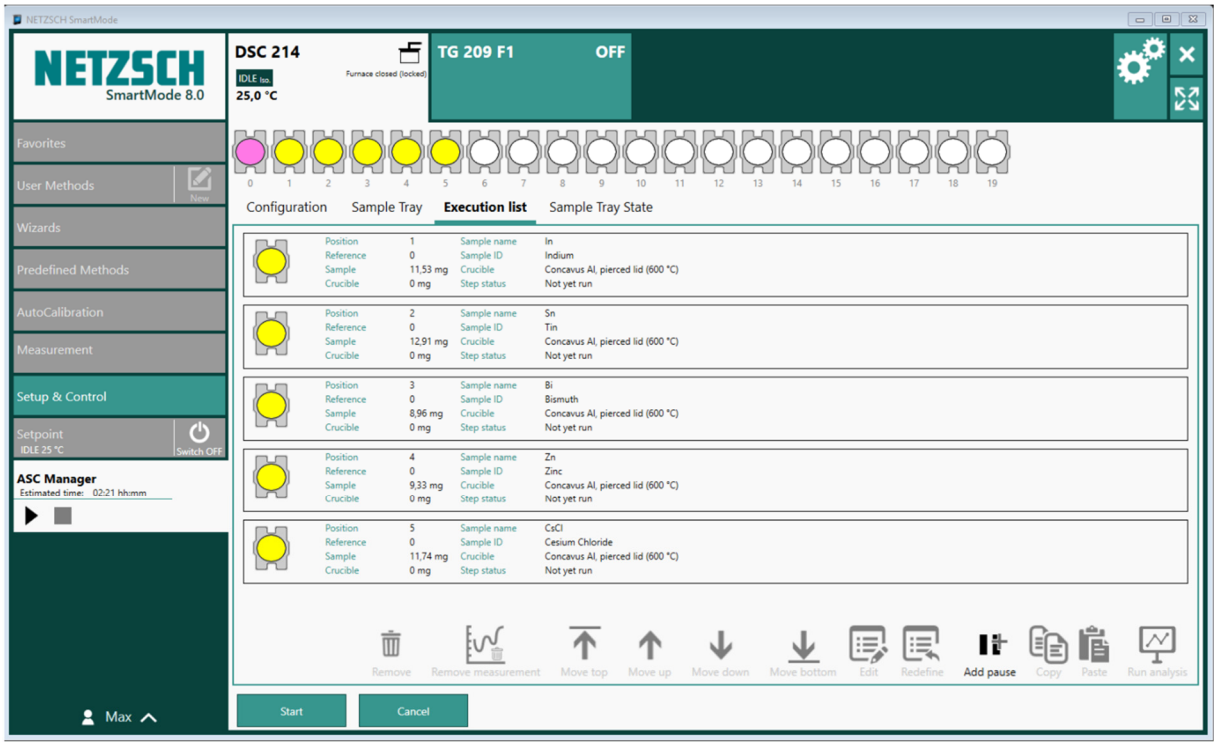
Screen is quite similar for sample tray right and calibration strip.

Preview



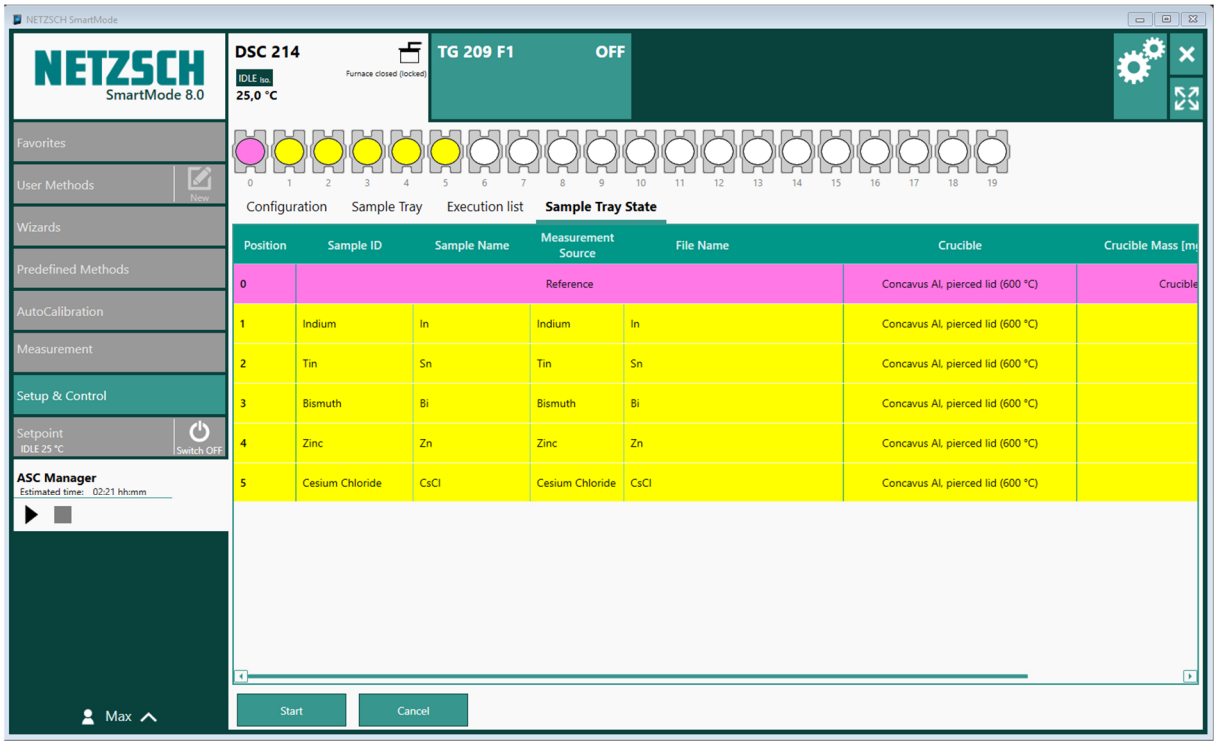
This screen is only visible when an ASC 400 is used and gives a quick overview of defined sample / calibration materials. For editing select the respective position under the header “Sample Tray Left”, “Sample Tray Right” or “Calibration Strip”.

Execution List



In the Execution list of the ASC manager you see the actual sequence of measurement. By means of the toolbar in the right corner each single measurement can be copied, removed or modified. Here you can also define the desired sequence of measurement.

Sample tray state



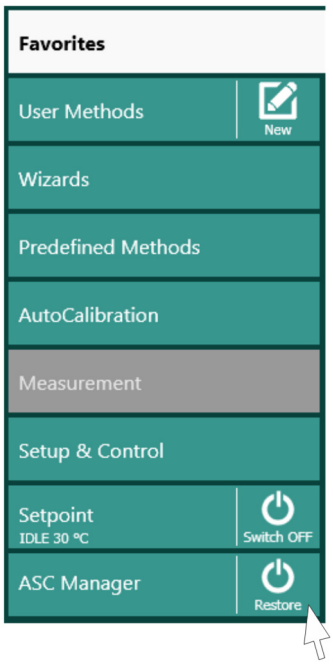
All programmed positions are visible here which will gives an quick overview. For editing select the respective position under the header “Sample Tray Left”, “Sample Tray Right” or “Calibration Strip”.

Restore

After closing the Smart Mode, the latest sample tray status will be saved automatically. When opening the Smart Mode next time, Proteus will offer the possibility to restore the latest sample tray status and to continue where it was stopped.

The saved sample tray status will be lost if any run is performed in manual mode or another new sample tray is defined after having started the Smart Mode.

A saved sample tray status can only be restored if connected gases or cooling devices were not changed.
If measurements no longer exists in the working directories methods will be deleted from the sample tray definition.

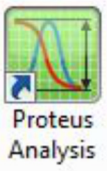
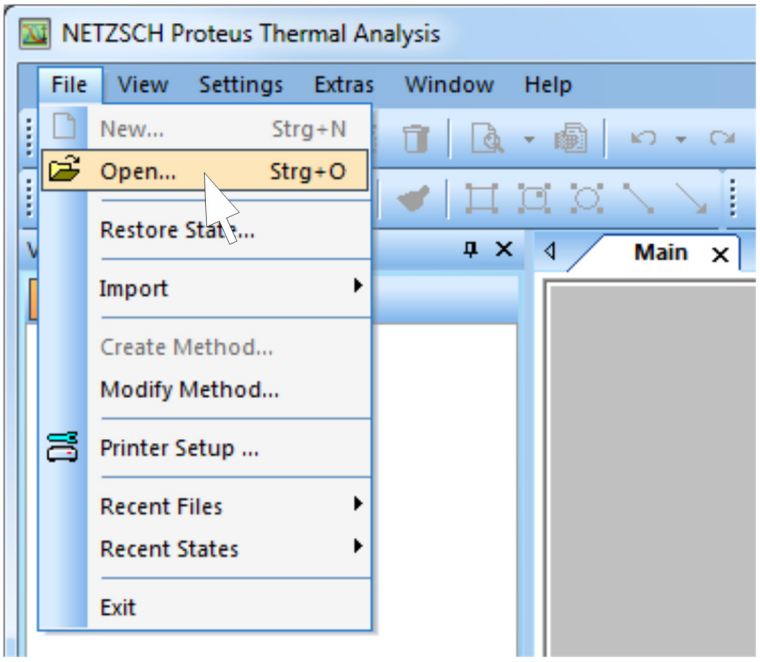


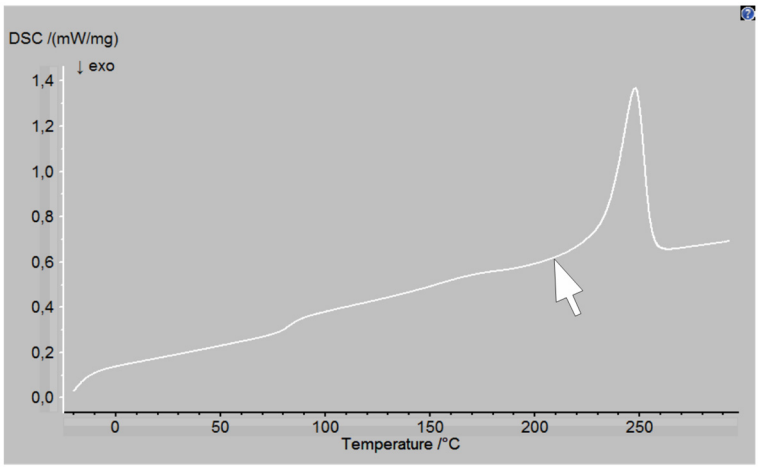
After click on restore, the last restored state screen appears.

Evaluation

NOTE:

Remember!
It is possible to save analysis states also as methods with evaluation. As evaluation you can select either "based on analysis state" (manual evaluation) or "Auto Evaluation"!

	• Open the NETZSCH-Proteus group.
	• Open the Proteus Analysis.
	• Select **Open** in the **File** menu to open your measurement file.



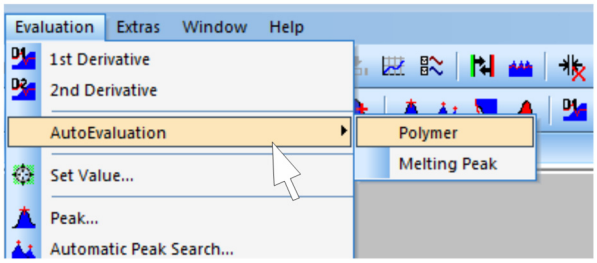
- Click on the DSC curve for selection.

NOTE:

Dark grey bar at axis x or y displays the complete axis range for opened measurement.

Light grey bar at axis x or y displays zoom area.

AutoEvaluation

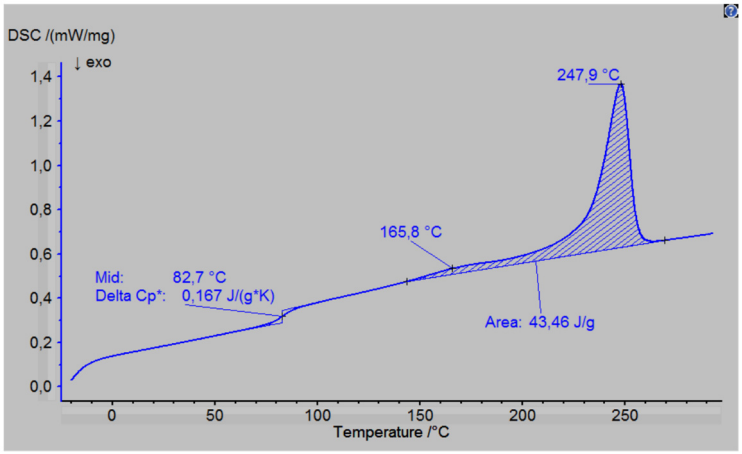


- Select AutoEvaluation in the Evaluation menu.

  DSC AutoEvaluation has subitems:

  Polymer:
  use this option especially for DSC AutoEvaluation of polymer materials.

  Melting Peak:
  use this option for DSC AutoEvaluation of other materials e.g. metals (for calibration)



- The results of the AutoEvaluation are shown in the window.
- With right mouse click on the presented results you can recalculate the values.
- DSC AutoEvaluation shows evaluation results for the current DSC data.

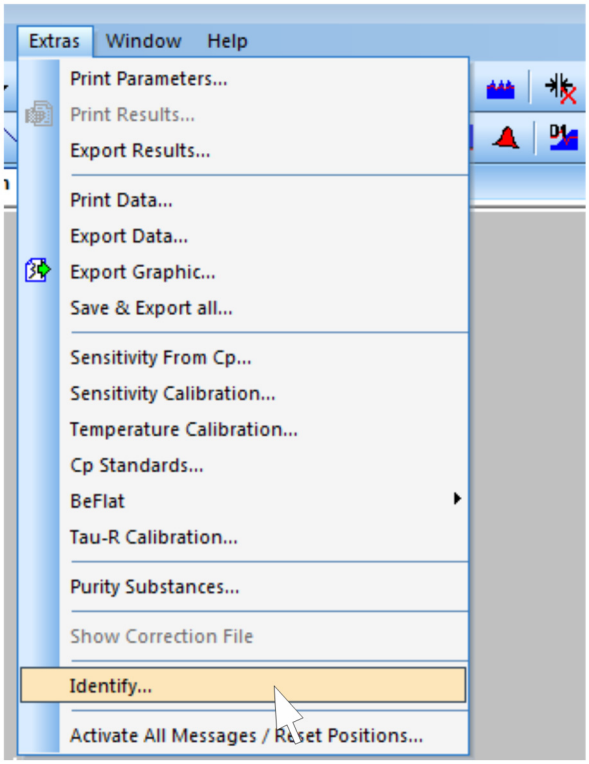
NOTE:

DSC AutoEvaluation is enabled only for heating DSC segments.

AutoEvaluation algorithm is optimized to get the best results for heating rates up to 20K/min.

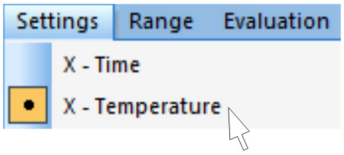
AutoEvaluation and Identify are autonomous mathematical algorithms, which propose an evaluation and an interpretation of DSC data.
Please be aware that these information are only recommendations based on objective algorithms that might not be always correct. The user is always in charge of the final curve interpretation and NETZSCH does not guarantee correctness of the algorithms.

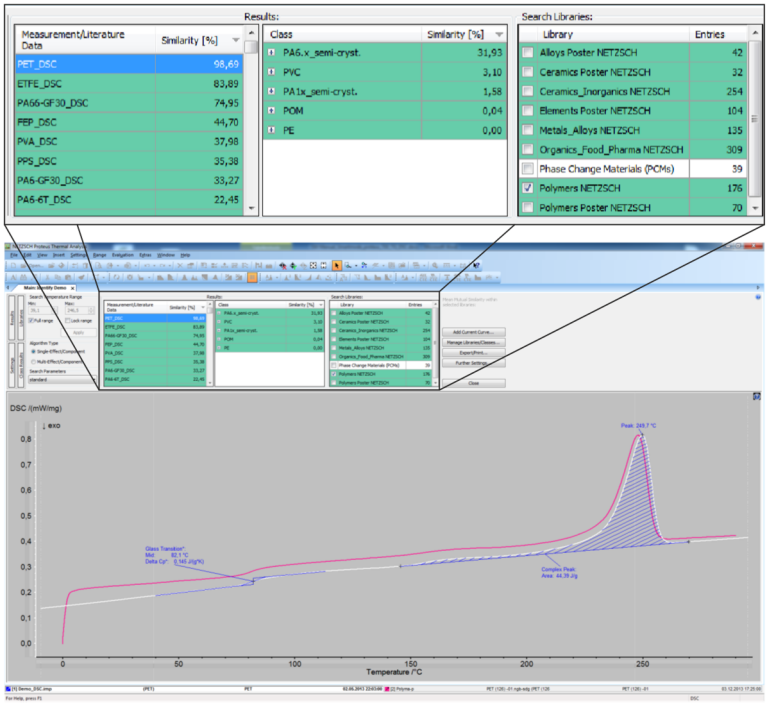
Identify



- Select Identify in the Extras menu after a curve segment was selected. Alternatively, click with right mouse button on a curve segment and select **Identify**.

Check that X-Temperature is selected and that the segments are splitted!
Consider ToolTips!





- The hits are shown in two windows on the left.
- In the window on the right you can select the search libraries.
- Identify can be customized with own measurements.

More details can be found in the Proteus help system!

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206030005 80032061 2020-12-03