

**NETZSCH**

Proven Excellence.

**SmartMode Manual**  
**Proteus 8.0**  
for  
**DSC Instruments**

53623 V1.0 / August 2019

DOCUMENTATION



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## General Information

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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.

Any electronic or mechanical duplication and distribution of these instructions requires prior written authorization of NETZSCH-Gerätebau GmbH.

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:

NETZSCH-Gerätebau GmbH Wittelsbacherstraße 42 <b>D - 95100 SELB</b> Telephone: +49 (0) 9287 881- 0 Telefax: +49 (0) 9287 881- 505 E-Mail: at@netzsch.com Internet: <a href="http://www.netzsch-thermal-analysis.com">http://www.netzsch-thermal-analysis.com</a>
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## Software Manual – Smart Mode Measurement Proteus 8.0

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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.

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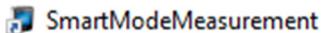
## Start SmartMode Software

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NETZSCH-Proteus-80

Open the NETZSCH-Proteus group on desktop.



SmartModeMeasurement

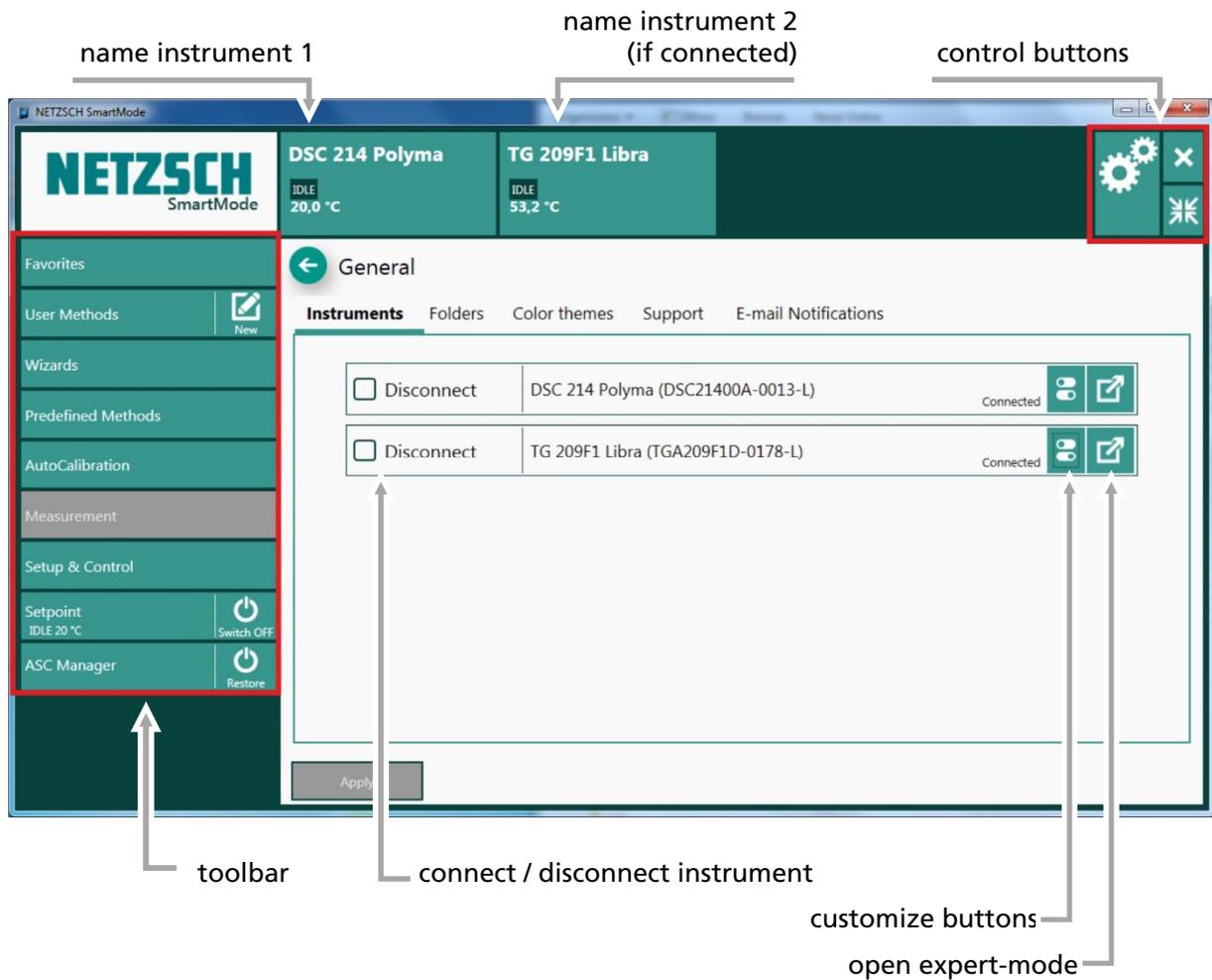
Open the SmartMode Measurement.

## 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.

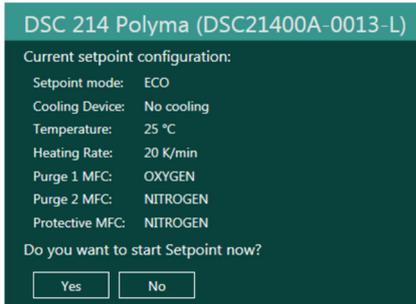


Control buttons:



## Activate Setpoint

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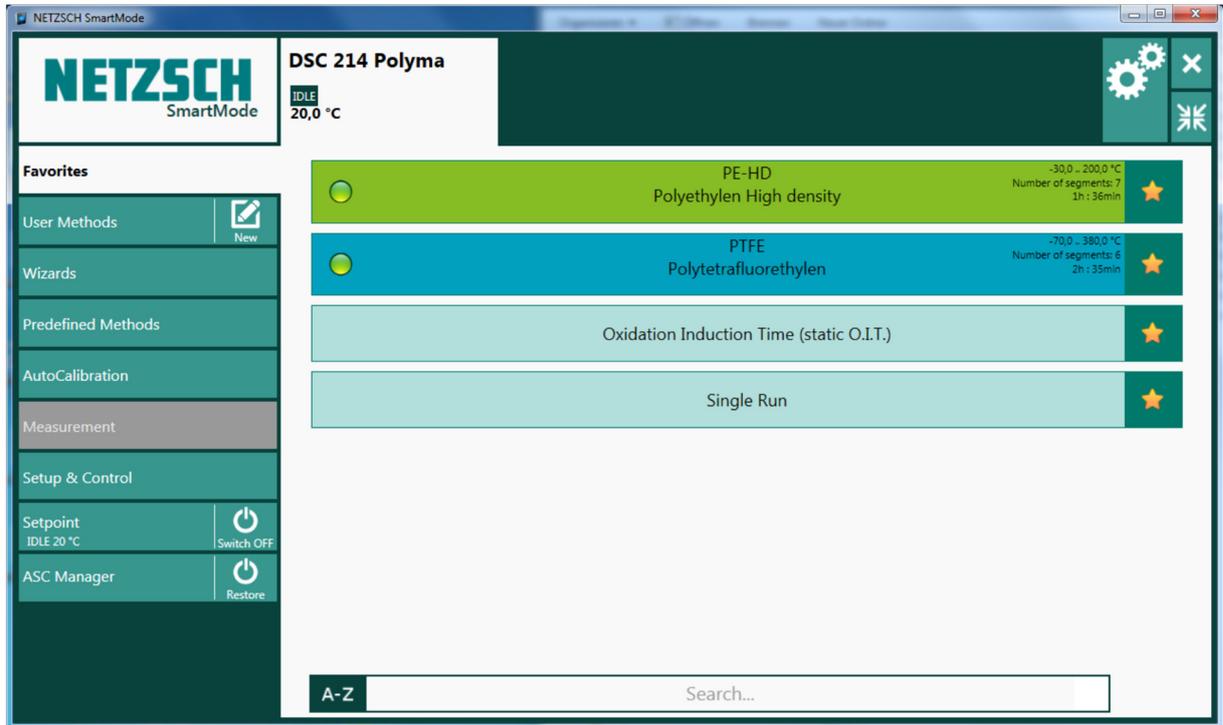


Enable setpoint to keep the instrument always under controlled conditions (protective/ purge gas flow, coolings). For DSC instruments with connected intracooler (IC) the setpoint prohibits a frozen cell when the IC is active, but heater is inactive. In auto sampler mode the sample is inserted/ removed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

Method Name	Status	Temperature Range	Number of Segments	Measurement Time
PolymerPoster	OK			
Training	OK			
140714 AS abc.ngb-s-dsc	OK	200 - 111.0 °C	Number of segments: 1	0h: 8min
140714 AS abcd.ngb-s-dsc	OK	300 - 180.0 °C	Number of segments: 4	0h: 12min
140729 AS PLA 10K.ngb-s-dsc	OK	0.0 - 200.0 °C	Number of segments: 9	0h: 25min
2014-07-24-DS-Base.ngb-s-dsc	OK	25.0 - 400.0 °C	Number of segments: 9	0h: 45min
3943-U-14-OIT.ngb-s-dsc	OK	-70.0 - 210.0 °C	Number of segments: 3	0h: 33min
LDPE - fast first, slow second heating -03.ngb-s-dsc	OK	0.0 - 190.0 °C	Number of segments: 4	0h: 12min
LDPE fast first, slow second 02.ngb-s-dsc	OK	0.0 - 190.0 °C	Number of segments: 4	0h: 12min
PA6.ngb-s-dsc	OK	-70.0 - 260.0 °C	Number of segments: 3	0h: 53min
PA66.ngb-s-dsc	OK	-70.0 - 290.0 °C	Number of segments: 3	0h: 16min
PA6GF30 fast first, slow second.ngb-s-dsc	OK	0.0 - 280.0 °C	Number of segments: 4	0h: 22min

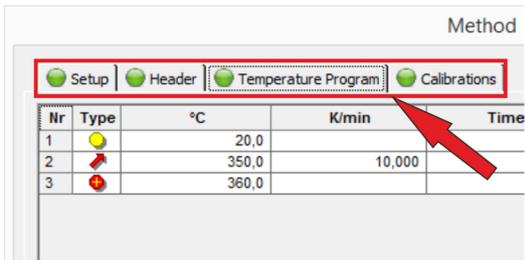
Created methods can not be copied or moved from one method to another method.

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.

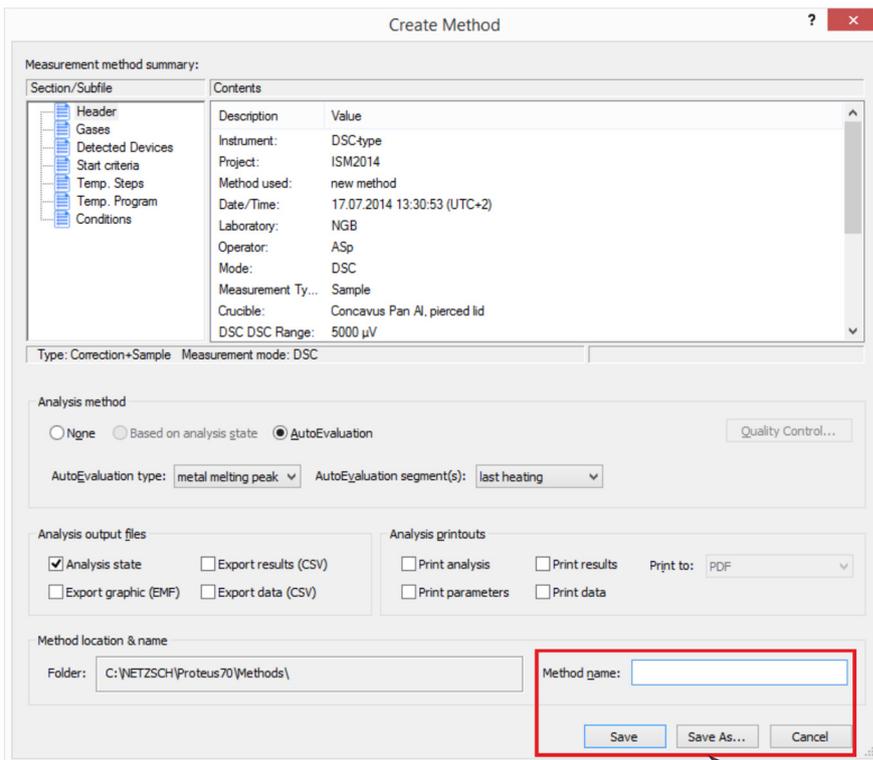
## Create a new method



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



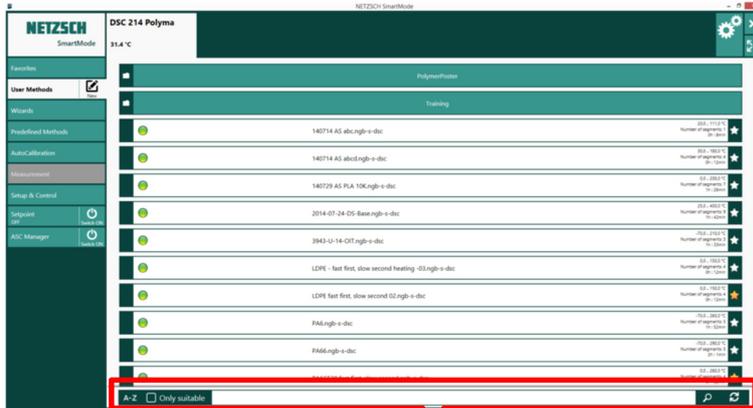
Define setup, header, temperature program, calibrations and save the method. If calibrations are applied later on when using the method, suitable calibrations are precondition.



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.

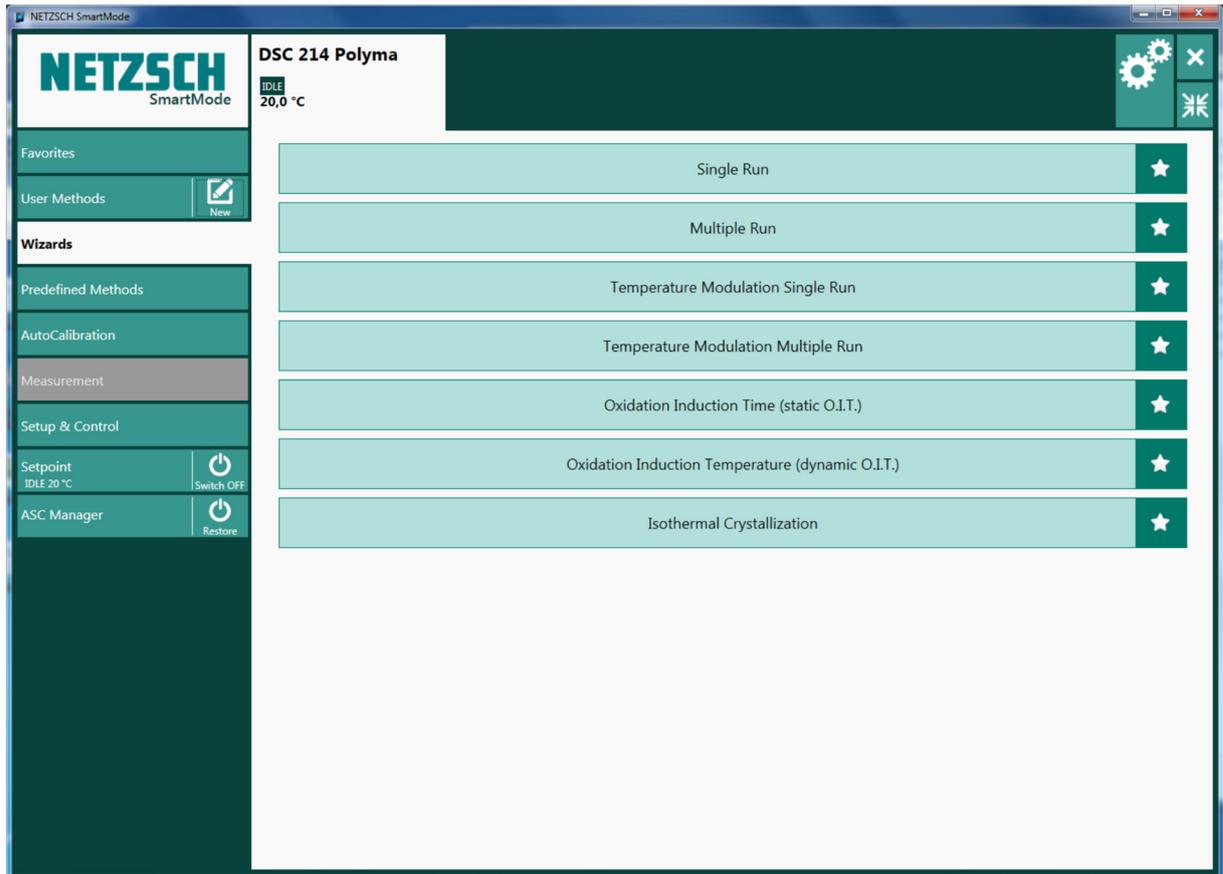


- ↑ search input box
- ↑ only files which are suitable with configuration of instrument will be displayed
- ↑ **A-Z** arrange files by name a-z
- ↑ **Z-A** arrange files by name z-a
- ↑ arrange files by usage date/time (last used on top)
- ↑ arrange files by usage counter (most frequently used on top)

↑ search  
↑ update list

## SmartMode Tab – Wizards

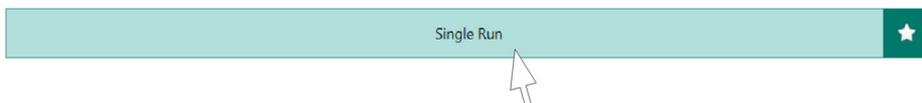
Structurally predefined measurement programs can be selected for typical operations like single run, multiple run, O.I.T., isothermal crystallization, TM-DSC (option). Moreover, crucible type and gas flow can be selected.



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.

Single Run

- Basic data
- Sample and Reference
- Gases setup
- Temperature ranges

DYNAMIC From **-40** °C To **450** °C Heating Rate **10** K/min

ISO Duration **0** min

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	●	-40					<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
1	↗	450	10	00:49:00	300	30	<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
2	●	460					<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min

■ Purge 1 MFC 
 ■ Purge 2 MFC 
 ■ Protective MFC

**Temperature Program**

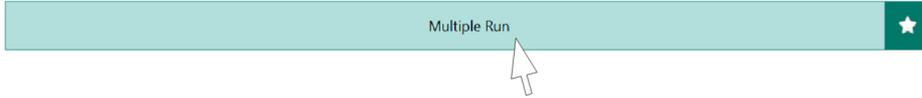
Time 00:49 hh:mm

Start    Insert Sample    Start criteria

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).

Multiple Run

Temperature ranges

Cycles count **1**

Type	From °C	To °C	Heating Rate K/min
DYNAMIC	-50	180	10
ISO	Duration	0	min
DYNAMIC	180	-50	10
ISO	Duration	5	min
<input checked="" type="checkbox"/> Use final segment			
DYNAMIC	-50	200	10
ISO	Duration	0	min

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	●	-50					<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
1	↗	180	10	00:23:00	300	30	<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
2	↘	-50	10	00:23:00	300	30	<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
3	→	-50		00:05:00	300		<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
4	↗	200	10	00:25:00	300	30	<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
5	●	210					<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min

■ Purge 1 MFC   
 ■ Purge 2 MFC   
 ■ Protective MFC

Temperature Program

Time 01:16 hh:mm

Additional Info

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.

**Temperature Modulation Single Run**

Temperature ranges

	From	To	Heating Rate K/min	Period s	Ampl. K
DYNAMIC	25 °C	200 °C	5	30	0,5
ISO	Duration 0 min			0	0

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	●	25					<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
1	⚡	200	5	00:35:00	300	60	<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min
2	●	210					<input checked="" type="checkbox"/>	0 ml/min	40 ml/min	60 ml/min

Purge 1 MFC  
  Purge 2 MFC  
  Protective MFC

**Temperature Program**

Graph showing temperature (°C) vs Time (00:35 hh:mm). The temperature starts at 25 °C and increases linearly to 200 °C.

**Additional Info**

Calibrations

Temperature calibration: <-70 °C... 571 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN  
 Sensitivity: <-70 °C... 571 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

Start   Insert Sample   Start criteria

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  $DSC_o$ , amplitude  $A_{DSC}$ , phase shift  $\varphi$  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 Multiple Run

This software feature is optional available.



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

Temperature Modulation Multiple Run

Temperature ranges

Cycles count **1**

		From	°C	To	°C	Heating Rate K/min	Period s	Ampl. K	
DYNAMIC	From	20	°C	To 200	°C	5	30	0,5	↗
ISO	Duration	10	min				30	0,5	→
DYNAMIC	From	200	°C	To 20	°C	5			↘
ISO	Duration	20	min				30	0,5	→
<input checked="" type="checkbox"/> Use final segment									
DYNAMIC	From	20	°C	To 400	°C	5	30	0,5	↗
ISO	Duration	20	min				30	0,5	→

[10...300] and: 0

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	●	20					<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
1	↗	200	5	00:36:00	300	60	<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
2	→	200		00:10:00	300		<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
3	↘	20	5	00:36:00	300	60	<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
4	→	20		00:20:00	300		<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
5	↗	400	5	01:16:00	300	60	<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
6	→	400		00:20:00	300		<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min
7	●	410					<input checked="" type="checkbox"/>	40 ml/min	40 ml/min	60 ml/min

■ Purge 1 MFC
 ■ Purge 2 MFC
 ■ Protective MFC

Temperature Program

Time 03:18 hh:mm

Additional Info

Start Insert Sample Start criteria

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  $DSC_o$ , amplitude  $A_{DSC}$  and phase shift  $\varphi$  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.

Oxidation Induction Time (static O.I.T.)
★

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← Oxidation Induction Time (static O.I.T.)

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**Gases setup**

Purge 1 MFC: OXYGEN Flow Rate: 50 ml/min Oxidizing gas

Purge 2 MFC: NITROGEN Flow Rate: 50 ml/min Inert gas

Protective MFC: NITROGEN Flow Rate: 60 ml/min

**Temperature settings**

Start Temperature: 25 °C    Iso Temperature: 200 °C    Heating Rate: 20 K/min

Inert Time: 0 : 03 : 00 hh:mm:ss

Please enter value between 00:01:00 and 99:59:00

Oxidation Time: 120 min

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	🟡	25					<input type="checkbox"/>	0 ml/min	50 ml/min	60 ml/min
1	🔴	200	20	00:08:45	150	7,50	<input checked="" type="checkbox"/>	0 ml/min	50 ml/min	60 ml/min
2	🟢	200		00:03:00	150		<input checked="" type="checkbox"/>	0 ml/min	50 ml/min	60 ml/min
3	🟢	200		02:00:00	150		<input checked="" type="checkbox"/>	50 ml/min	0 ml/min	60 ml/min
4	🔴	210					<input type="checkbox"/>	0 ml/min	50 ml/min	60 ml/min

■ Purge 1 MFC
■ Purge 2 MFC
■ Protective MFC

**Temperature Program**

**Additional Info**

Calibrations

Temperature calibration: <-70 °C... 571 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

Sensitivity: <-70 °C... 571 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

Tau-R calibration: <-70 °C... 571 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

Sample temperature stability threshold: 5 K

Sample temperature stability rate: 0,1 K/min

Start delay after stability: 00:00:30 hh:mm:ss

**OIT settings**

OIT detection settings:

OIT start delay: 0 : 01 : 00 hh:mm:ss

Please enter value between 00:01:00 and 01:59:45.

End Delay Time: 0 : 05 : 00 hh:mm:ss

Please enter value between 00:00:00 and 01:58:45.

DSC Threshold: 0,5 mW/mg

Start
Insert Sample
Start criteria

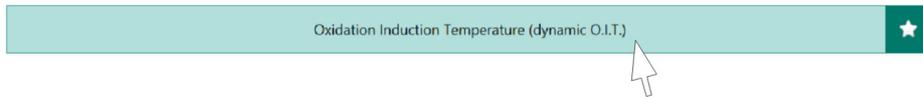
Example for O.I.T. static settings

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  $\mu\text{V}/\text{mg}$  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.

← Oxidation Induction Temperature (dynamic O.I.T.)  
[according to ASTM E 2009 / ISO 1357-6]

Ⓐ Gases setup

Purge 1 MFC: OXYGEN Flow Rate 50 ml/min Oxidizing gas

Purge 2 MFC: NITROGEN Flow Rate 0 ml/min Final Conditioning

Protective MFC: NITROGEN Flow Rate 60 ml/min

Ⓐ Temperature settings

Start Temperature 25 °C End Temperature 300 °C Heating Rate 20 K/min

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	●	25					<input type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
1	⚡	300	20	00:13:45	150	7,50	<input checked="" type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
2	●	310					<input type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min

■ Purge 1 MFC ■ Purge 2 MFC ■ Protective MFC

Temperature Program

Time 00:14 hh:mm

Ⓐ OIT settings

OIT detection settings:

OIT start delay 0 : 00 : 00 + - hh:mm:ss 25 °C  
Please enter value between 00:00:00 and 00:13:30.

End Delay Time 0 : 05 : 00 + - hh:mm:ss  
Please enter value between 00:00:00 and 00:13:30.

DSC Threshold 0,5 mW/mg

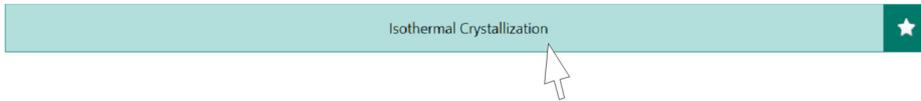
Start Insert Sample Start criteria

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  $\mu\text{W/mg}$  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.

← Isothermal Crystallization

Ⓐ Temperature settings

Start Temperature 25 °C Heating Rate 10 K/min  
 Upper Temperature 200 °C Stabilization Time 3 min  
 Cryst. Temperature 145 °C Cooling Rate 250 K/min  
 Crystallization Time 30 min

Nr	Type	°C	K/min	Duration	pts/min	pts/K	STC	OXYGEN	NITROGEN	NITROGEN
0	●	25					<input type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
1	↗	200	10	00:17:30	600	60	<input checked="" type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
2	→	200		00:03:00	600		<input checked="" type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
3	↘	145	250	00:00:13	600	2,40	<input type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
4	→	145		00:30:00	600		<input checked="" type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min
5	●	210					<input checked="" type="checkbox"/>	0 ml/min	0 ml/min	0 ml/min

Purge 1 MFC
  Purge 2 MFC
  Protective MFC

Temperature Program

Time 00:51 hh:mm

Start Insert Sample Start criteria

Example for isothermal crystallization

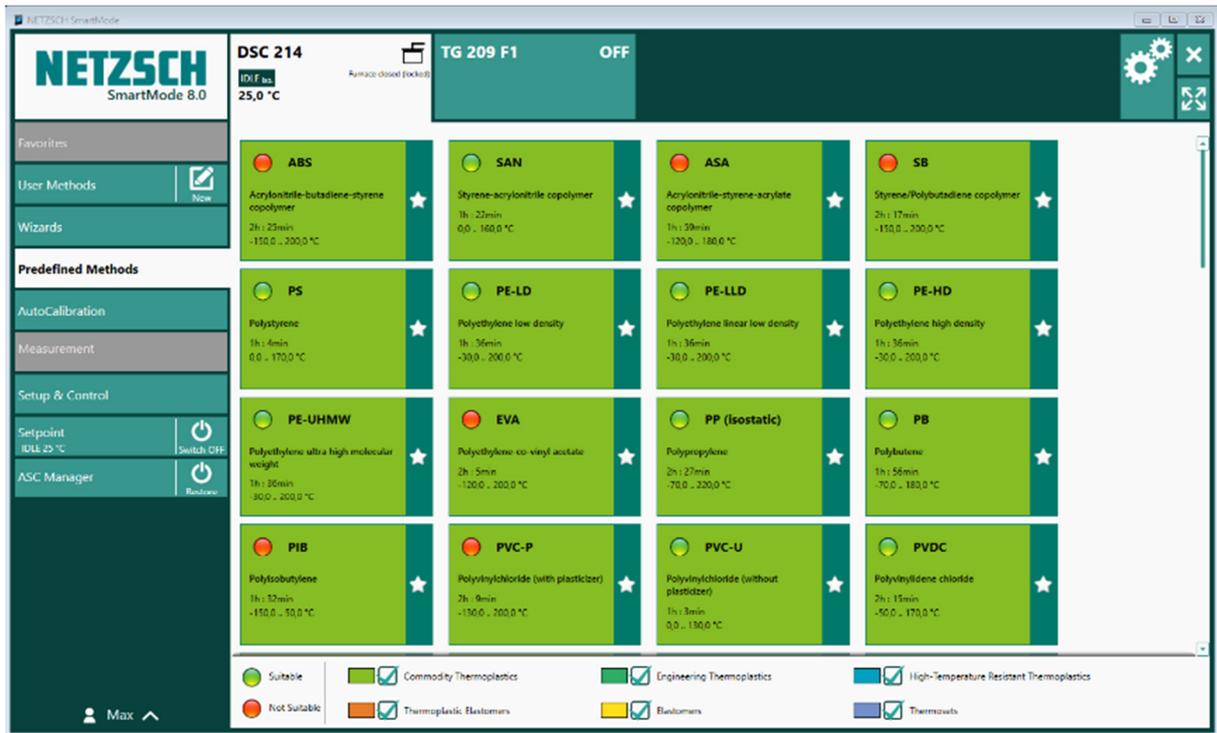
This wizard helps for defining experiments for the investigation of crystallization of semicrystalline polymers. The method includes a very fast cooling segment from an upper temperature (molten polymer) to a crystallization temperature followed by an isothermal segment in which crystallization occurs.

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!

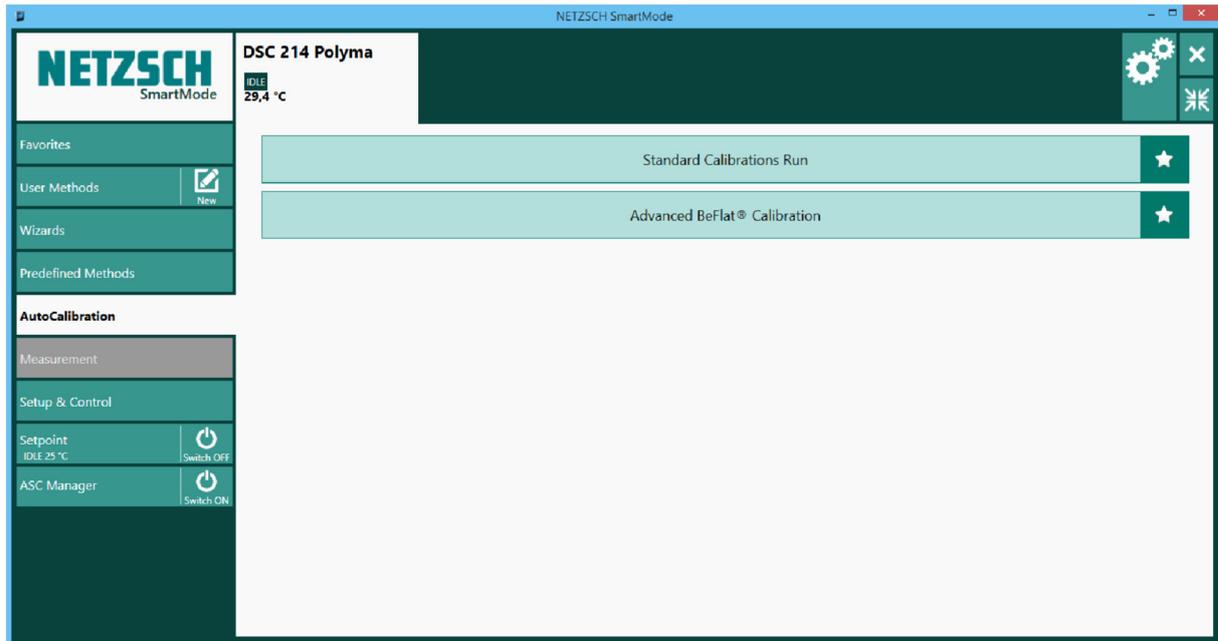


### Method Validation Failed!

- **Temperature calibration** Calibration file is required but no suitable calibration for the current instrument is found.  
Required calibration: Temperature range <-50,0 °C... 285,0 °C>, Crucible: High pressure, steel, Gas: NITROGEN
- **Heat flow calibration** Calibration file is required but no suitable calibration for the current instrument is found.  
Required calibration: Temperature range <-50,0 °C... 285,0 °C>, Crucible: High pressure, steel, Gas: NITROGEN

## 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

← Standard Calibrations Run

**Autosampler settings** Calibrations Standards Results

Activate autosampler mode

Max removal temperature: 150 °C

Final removal: Remove sample

Reference crucible: 19 - Concavus Pan Al, pierced

Reference crucibles

Position	Name	Mass [mg]	Crucible Mass [mg]	Crucible	
19	----	50	22,2	Concavus Pan Al, pierced lid 610 °C	<input type="button" value="Add"/> <input type="button" value="Remove"/>

This reference is used in Standard Calibrations.

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

Standard Calibrations Run

Autosampler settings **Calibrations** Standards Results

Full calibration  
 Fast recalibration  
 Verification run

Select calibrations:

Temperature Done  
 Heat Flow Done  
 Tau-R Not yet performed

Autosampler mode is active. Available calibration sets and crucibles are limited by the configuration of the reference crucible.

Calibration set: \*\*\* All Standards \*\*\*  
 20 standard(s) available

Crucible: Concavus Al, pierced lid Confirm

Temperature limit: 600 °C

Purge1: Confirm

Purge2: NITROGEN Confirm

Protective: NITROGEN Confirm

Purge2 flow: 40 ml/min  
 Protective flow: 60 ml/min

Temp. range: -80 - 600 °C  
 Range: -80 °C ... 600 °C

Heating Rate: 10 K/min Change

MFC flow: Active

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

← Standard Calibrations Run

Autosampler settings   Calibrations   **Standards**   Results

Name	ASC Position	T. Melt. °C	Enthalpy J/g	Sample Mass mg	
<input type="checkbox"/> Adamantane		-64.5	22.000		
<input checked="" type="checkbox"/> Indium <small>Temperature program for Indium : starts at 80 °C, heating to 180 °C, cooling to 100 °C, isothermal for 5 minutes and heating to 180 °C with 10 K/ min.</small>	1	156.6	28.600	11,53	●
<input checked="" type="checkbox"/> Tin	2	231.9	60.500	12,91	●
<input checked="" type="checkbox"/> Bismuth	3	271.4	53.100	8,96	●
<input checked="" type="checkbox"/> Zinc	4	419.5	107.500	9,33	●
<input checked="" type="checkbox"/> Cesium Chloride	5	476.0	17.200	11,74 <small>(0.001..50000)</small>	●

Measurement: Impossible Succeeded Ready Obligatory Failed

Evaluation: Needs verification Failed Succeeded Not used Threshold exceeded Reset

Instrument is OK.

Instrument configuration   Reset all   Reset selected   Start   Stop   Save & Finish

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!

## DSC 214 Polyma (240-20-0772-L)

Please make sure 'Indium' sample is inserted for measurement.

OK

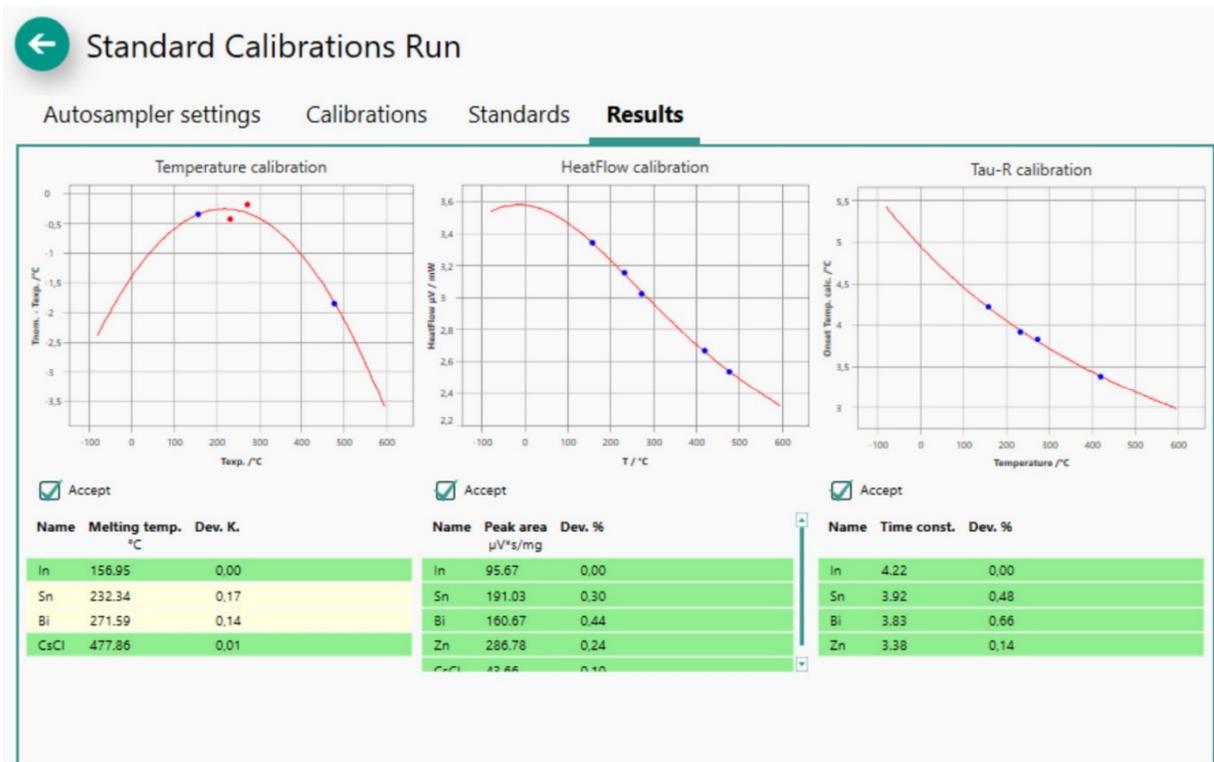
Cancel



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 its 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 adaptations 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.

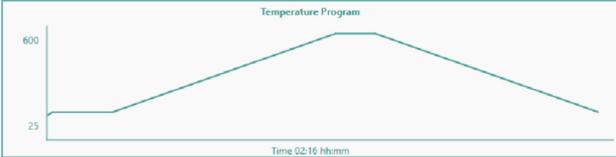
Advanced BeFlat® Calibration

← Advanced BeFlat® Calibration

- Advanced BeFlat calibration missing
- Empty sample position  
Not yet performed.
- Empty crucible on sample position  
Not yet performed.



Temperature program: start at 25 °C, heating to 50 °C, isothermal for 15 minutes, heating to 600 °C, isothermal for 10 minutes and cooling to 50 °C.



Cooling device: No cooling

Temp. range: 50 - 600 °C  
range: 5 °C ... 605 °C except 25 °C

Crucible: Concavus Pan Al, pierced lid   
temperature limit: 610 °C

Reference crucible mass: 20 mg  
range: 5 mg ... 50000 mg

Sample crucible mass: 0 mg  
range: 5 mg ... 50000 mg

Purge1: HELIUM

Purge2: NITROGEN

Protective: NITROGEN   
Purge2 flow: 40 ml/min  
Protective flow: 60 ml/min

MFC flow: Active

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.

### ***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:

#### Additional Info

##### Calibrations

Temperature calibration: <-100 °C... 500 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

Sensitivity: <-30 °C... 605 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

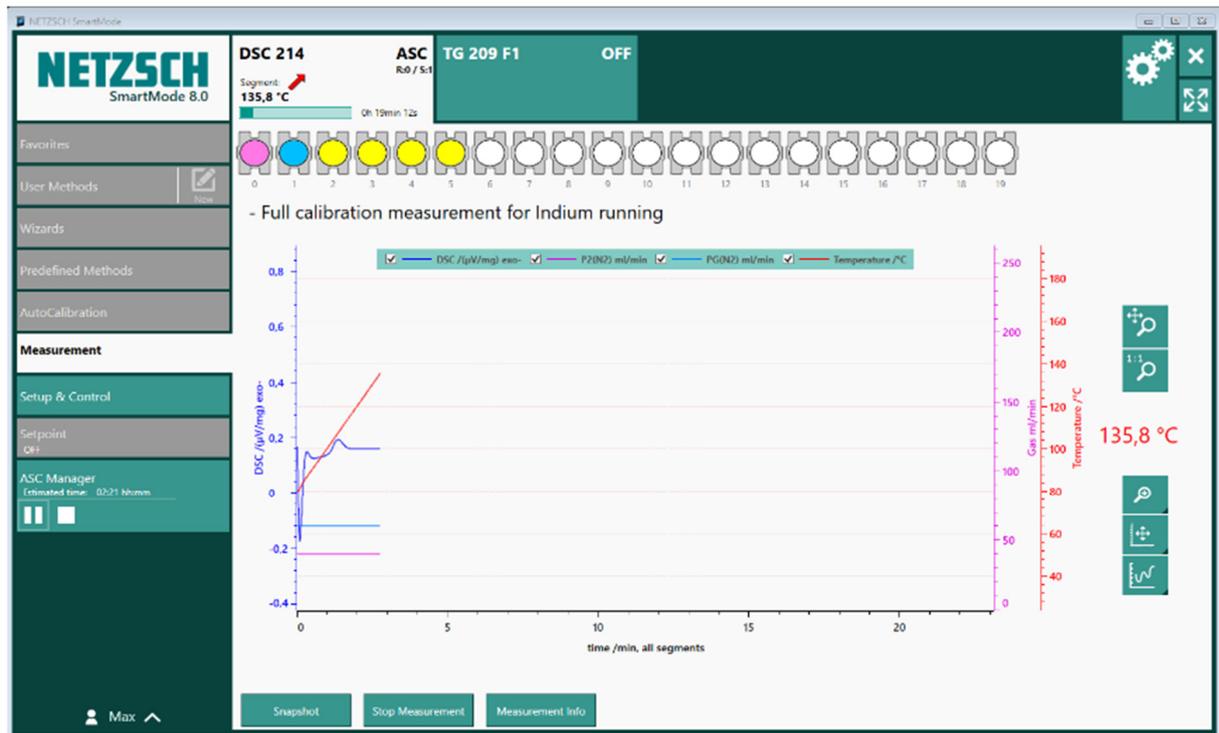
Tau-R calibration: <-30 °C... 605 °C>, Crucible: Concavus Pan Al, pierced lid, Gas: NITROGEN

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



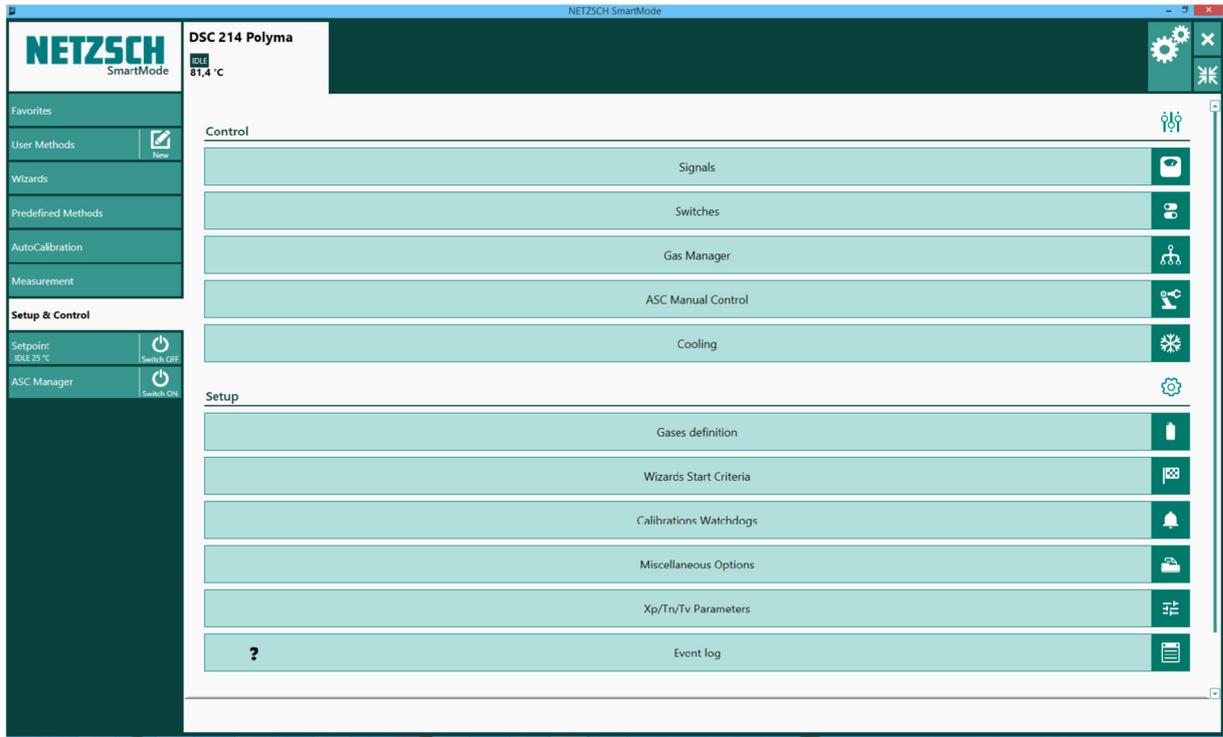
The tab Measurement shows a graphical chart of the recent running measurement. If no measurement is running, the tab is grey and cannot be activated.

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.

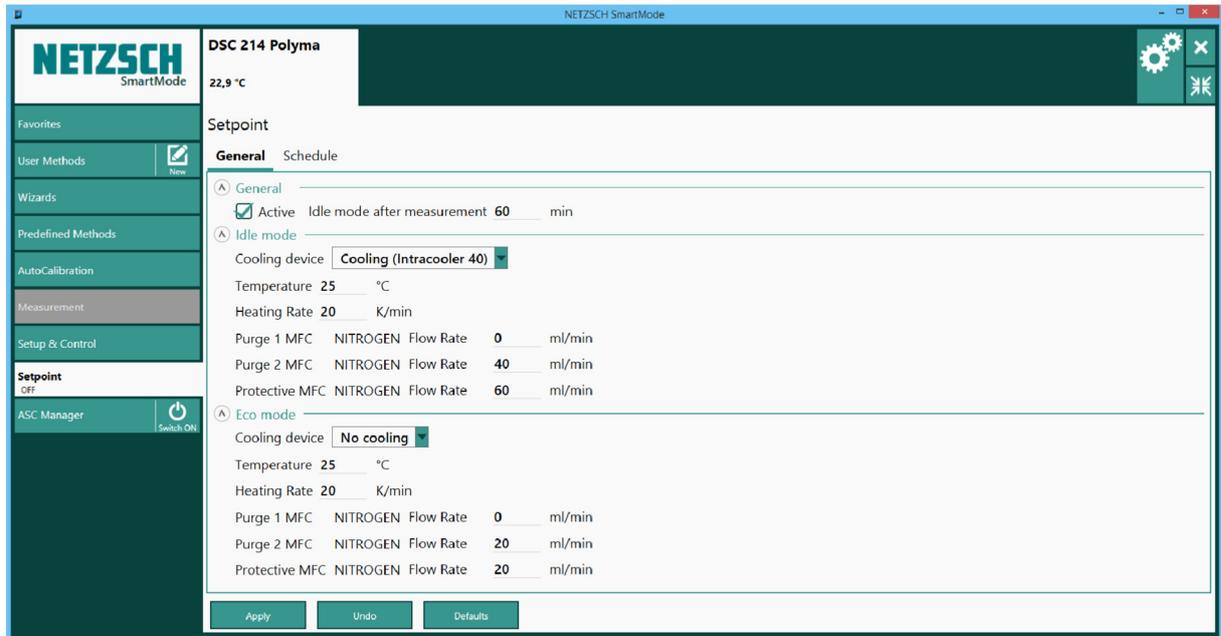
By means of the dialog Xp/Tn/Tv Parameters the predefined settings can be changed before or during a measurement (if necessary). These parameter changes are stored for Predefined Methods in a common parameter set. For any Wizard (e.g. Single Run, Multiple Run...) an individual parameter set is stored. 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**

Setpoint

General **Schedule**

	Monday	Tuesday	Wednesday	Thursday	Friday	Saturday	Sunday
00:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
01:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
02:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
03:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
04:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
05:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
06:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
07:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
08:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
09:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
10:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
11:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
12:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
13:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
14:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
15:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
16:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
17:00	IDLE	IDLE	IDLE	IDLE	IDLE	ECO	ECO
18:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
19:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
20:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
21:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
22:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO
23:00	ECO	ECO	ECO	ECO	ECO	ECO	ECO

Eco mode  Idle mode

Apply Undo Defaults

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

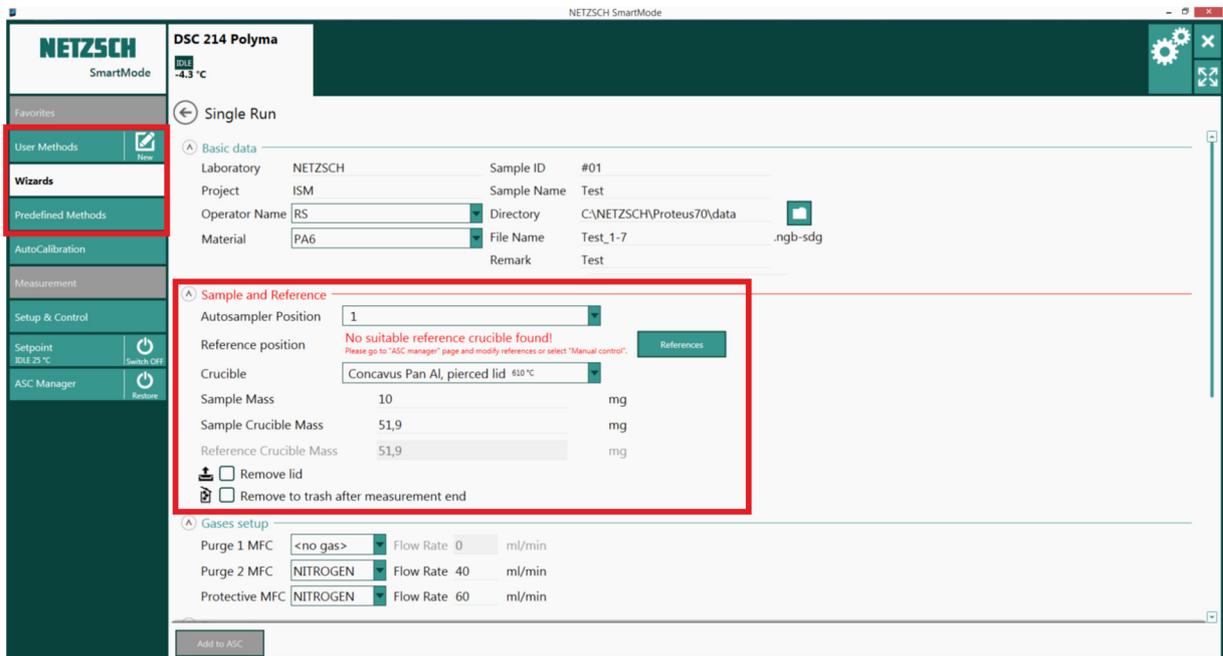
	defined
	done
	done (analysis failed or results are out of the predefined boundaries, e.g. for quality control)
	failed
	measurement active
	reference

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.

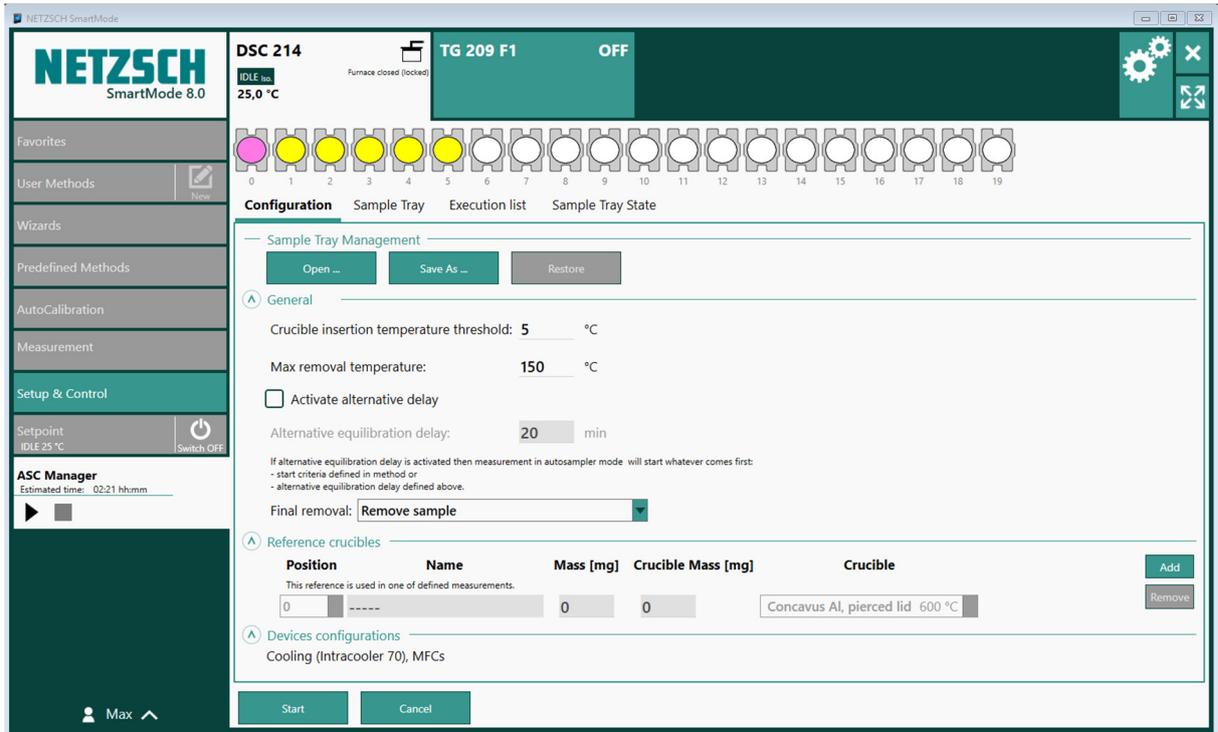


It is also possible activate the ASC by clicking "Restore", see page 39.

**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.

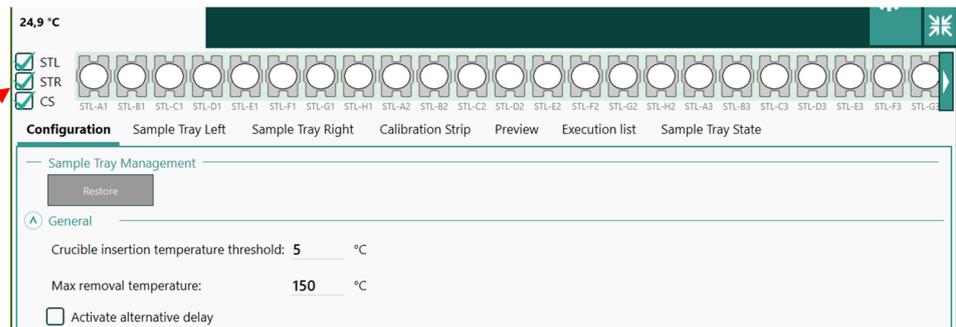
**Configuration**



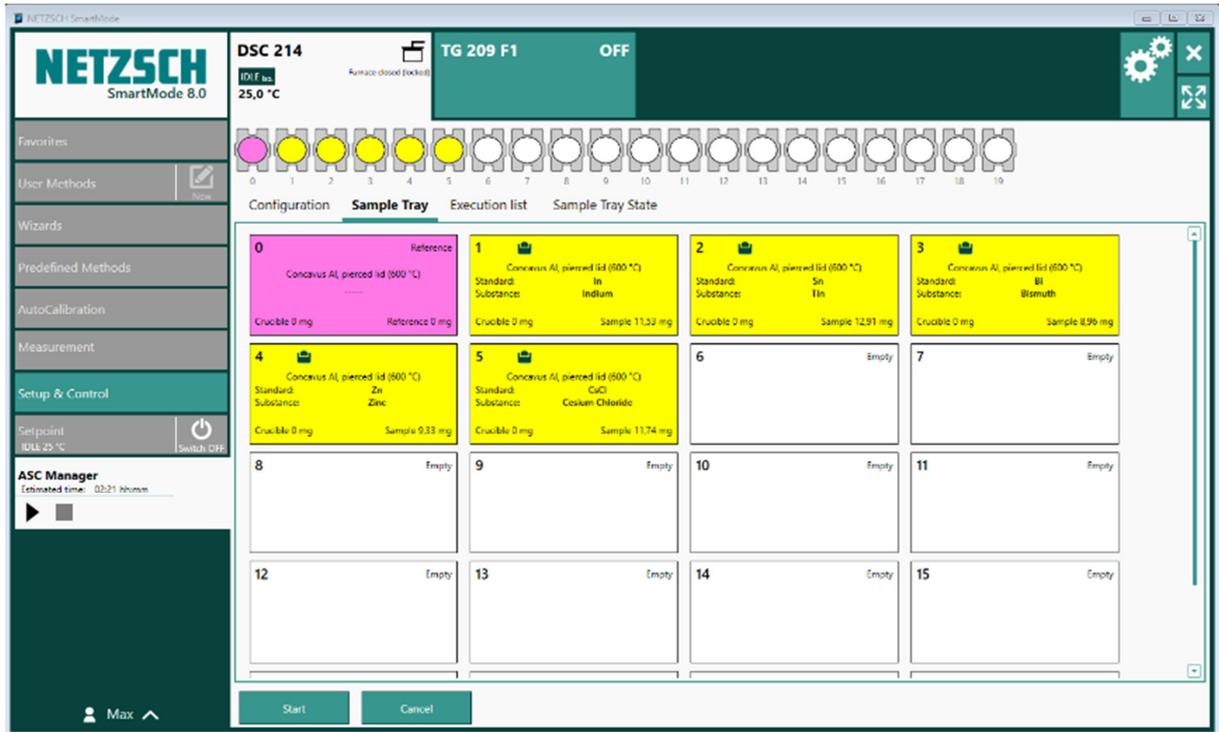
In the Configuration list of the ASC manager define the general ASC settings and the reference crucible (position, mass, crucible mass, crucible type).

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**



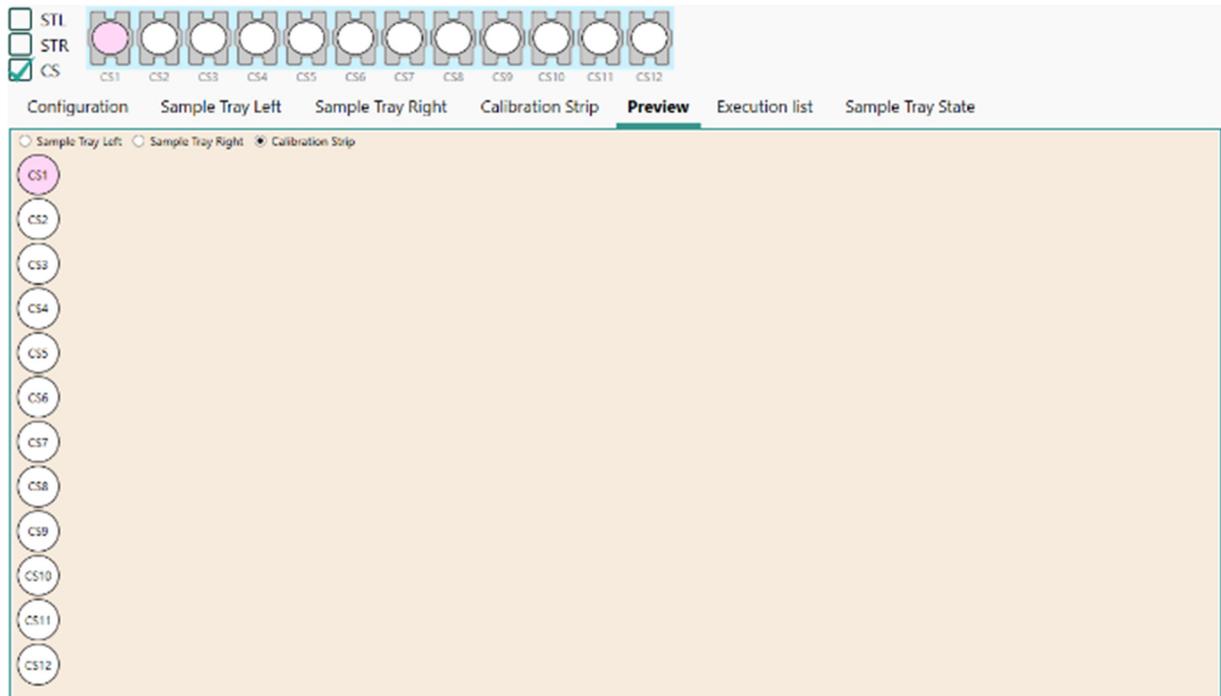
In the ASC manager you find the sample tray list with all defined positions. By means of the toolbar in the right corner each sample tray position can be edited and copied/pasted.

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

The screenshot displays the NETZSCH SmartMode 8.0 software interface. The top status bar shows 'DSC 214' with 'IDLE 10s' and '25,0 °C', and 'TG 209 F1' with 'OFF'. Below this is a sample tray visualization with 20 positions (0-19). The 'Execution list' tab is active, showing a table of measurement steps:

Position	Reference	Sample	Crucible	Sample name	Sample ID	Crucible	Step status
1	0	11,53 mg	0 mg	In	Indium	Concavus Al, pierced lid (600 °C)	Not yet run
2	0	12,91 mg	0 mg	Sn	Tin	Concavus Al, pierced lid (600 °C)	Not yet run
3	0	8,96 mg	0 mg	Bi	Bismuth	Concavus Al, pierced lid (600 °C)	Not yet run
4	0	9,33 mg	0 mg	Zn	Zinc	Concavus Al, pierced lid (600 °C)	Not yet run
5	0	11,74 mg	0 mg	CsCl	Cesium Chloride	Concavus Al, pierced lid (600 °C)	Not yet run

Below the table is a toolbar with icons for: Remove, Remove measurement, Move top, Move up, Move down, Move bottom, Edit, Redefine, Add pause, Copy, Paste, and Run analysis. At the bottom of the interface are 'Start' and 'Cancel' buttons.

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

The screenshot shows the NETZSCH SmartMode 8.0 interface. At the top, it displays 'DSC 214' with 'IDLE' and '25,0 °C' status, and 'TG 209 F1' with 'OFF' status. Below this is a row of 20 sample tray positions (0-19). The 'Sample Tray State' table is as follows:

Position	Sample ID	Sample Name	Measurement Source	File Name	Crucible	Crucible Mass [mg]
0	Reference				Concavus Al, pierced lid (600 °C)	Crucible
1	Indium	In	Indium	In	Concavus Al, pierced lid (600 °C)	
2	Tin	Sn	Tin	Sn	Concavus Al, pierced lid (600 °C)	
3	Bismuth	Bi	Bismuth	Bi	Concavus Al, pierced lid (600 °C)	
4	Zinc	Zn	Zinc	Zn	Concavus Al, pierced lid (600 °C)	
5	Cesium Chloride	CsCl	Cesium Chloride	CsCl	Concavus Al, pierced lid (600 °C)	

At the bottom of the interface, there are 'Start' and 'Cancel' buttons. The user 'Max' is logged in.

All programmed positions are visible here which will give a quick overview. For editing, select the respective position under the header "Sample Tray Left", "Sample Tray Right" or "Calibration Strip".

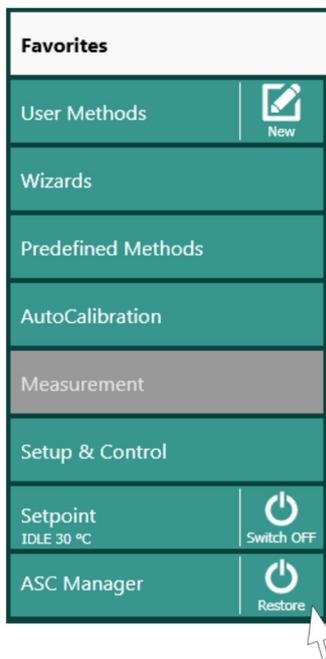
## Restore

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.

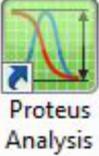
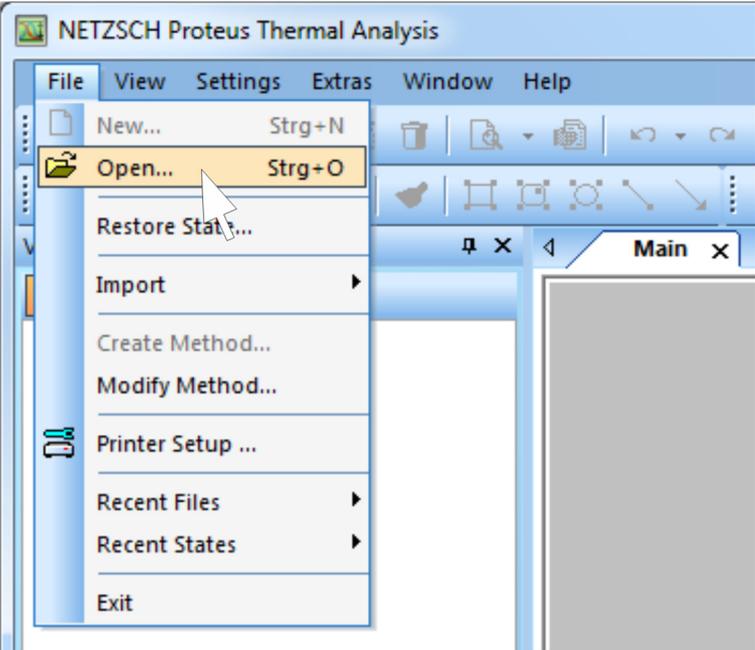
## Evaluation

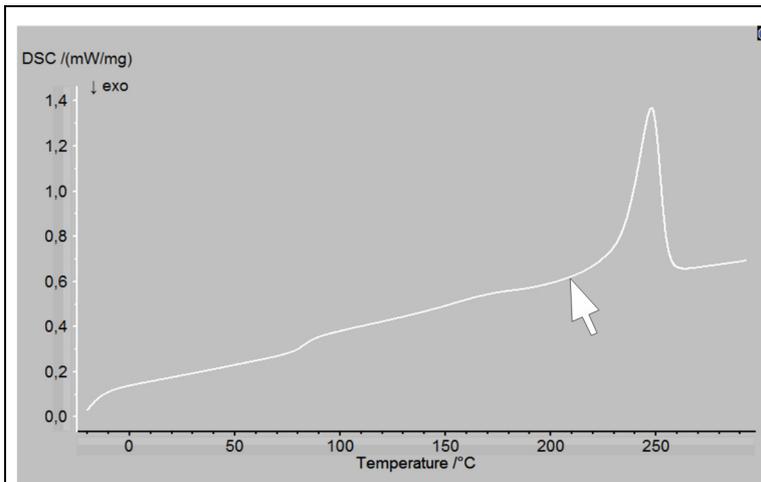


**NOTE:**

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"!

	<ul style="list-style-type: none"> <li>• Open the NETZSCH-Proteus group.</li> </ul>
	<ul style="list-style-type: none"> <li>• Open the Proteus Analysis.</li> </ul>
	<ul style="list-style-type: none"> <li>• Select <b>Open</b> in the <b>File</b> menu to open your measurement file.</li> </ul>



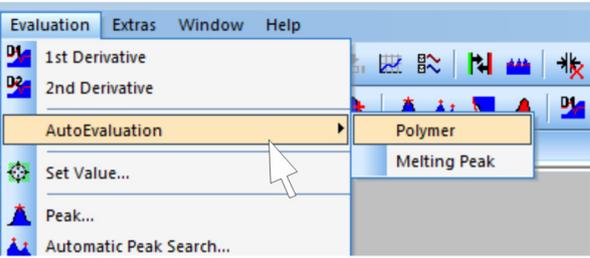
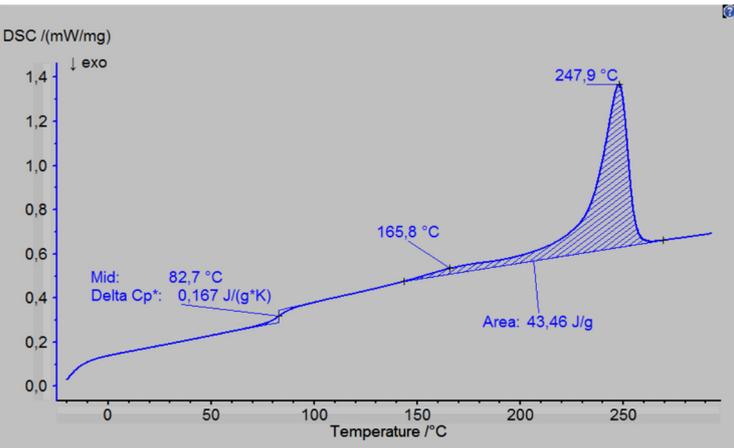
- Click on the DSC curve for selection.

**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

	<ul style="list-style-type: none"> <li>• Select AutoEvaluation in the Evaluation menu.</li> </ul> <p>DSC AutoEvaluation has subitems:</p> <p>Polymer: use this option especially for DSC AutoEvaluation of polymer materials.</p> <p>Melting Peak: use this option for DSC AutoEvaluation of other materials e.g. metals (for calibration)</p>
	<ul style="list-style-type: none"> <li>• The results of the AutoEvaluation are shown in the window.</li> <li>• With right mouse click on the presented results you can recalculate the values.</li> <li>• DSC AutoEvaluation shows evaluation results for the current DSC data.</li> </ul>



**NOTE:**

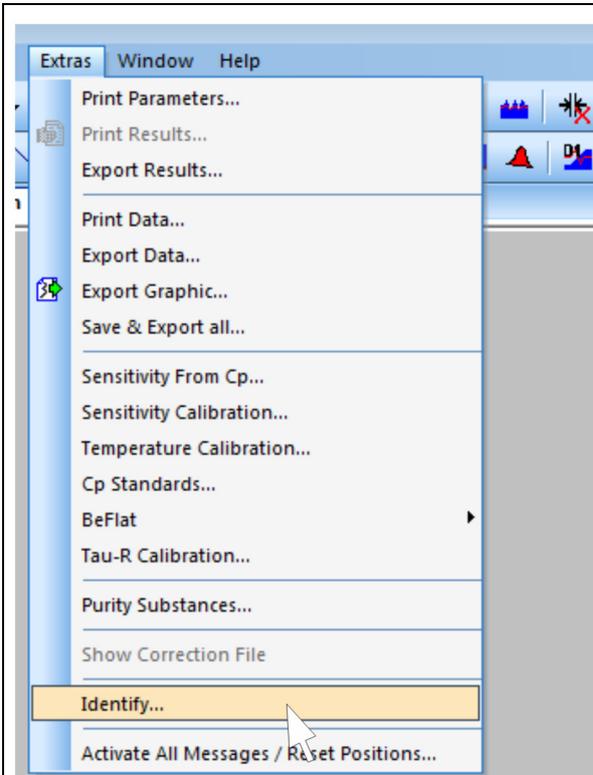
DSC AutoEvaluation is enabled only for heating DSC segments.

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.

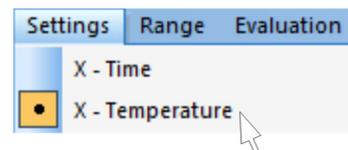
**Identify**



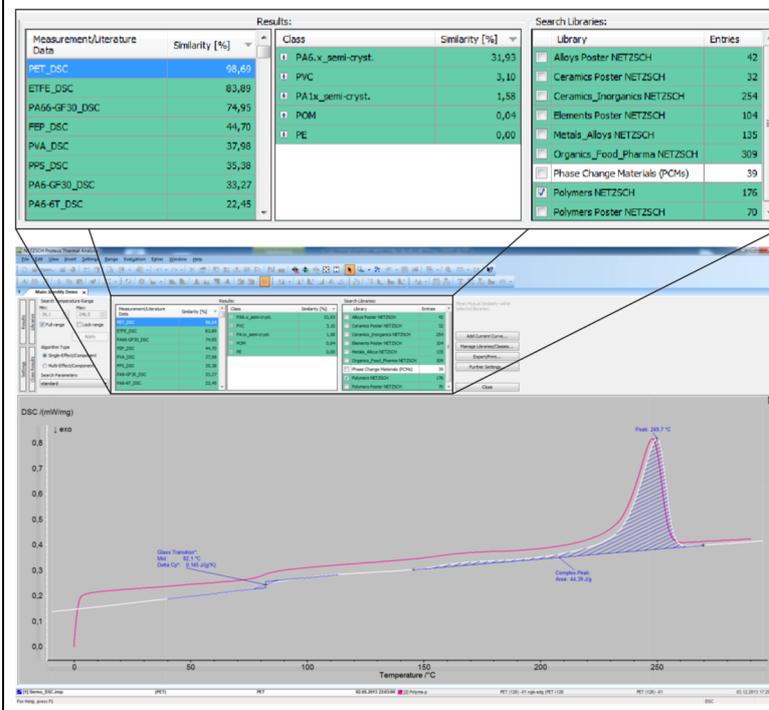
- Select Identify in the Extras menu after a curve segment was selected. Alternatively, click with right mouse button on a curve segment and select **Identify**.



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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