NETZSCH

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# Software Manual

## Proteus 8.x

*Temperature- and Sensitivity Calibration*

*for DSC214/ DSC204F1/ DSC3500*

51491 / Version 1.1 / April 2019

DOCUMENTATION

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# Contents

## Introduction

Why do we need to calibrate the instrument?

- Calibration is used to determine the instrument-specific deviation. By determining this deviation, it is possible to adapt the instrument to a standard by creating a calibration file without intervening in the instrument. Information on calibrations can be found in the relevant, industry-specific standards.
- **Calibrations are essential for a quantitative evaluation of a measurement.**

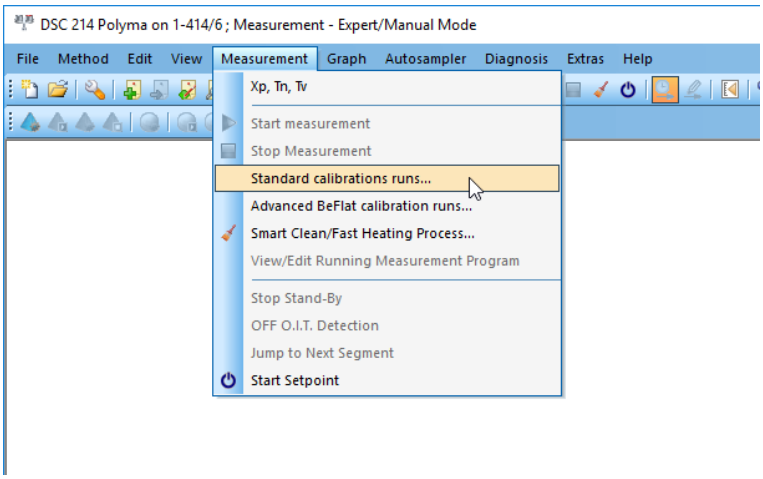
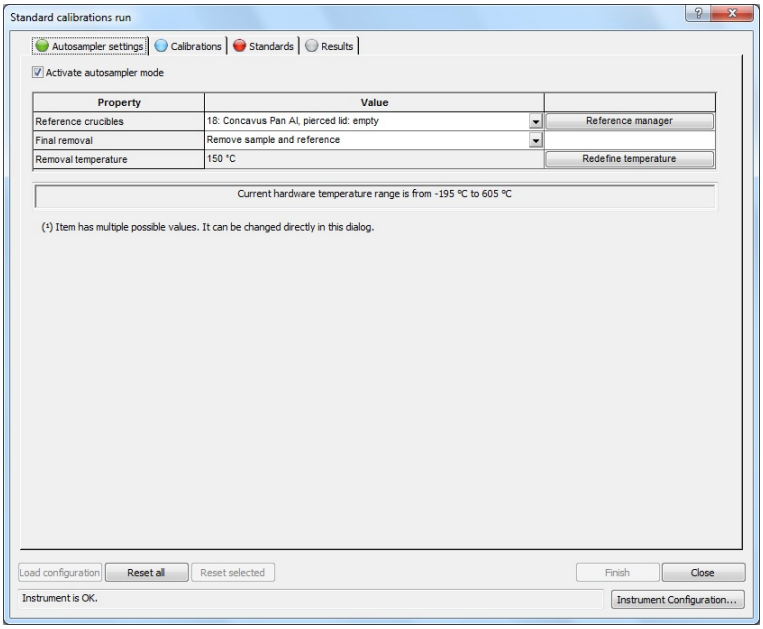
**NOTE!**

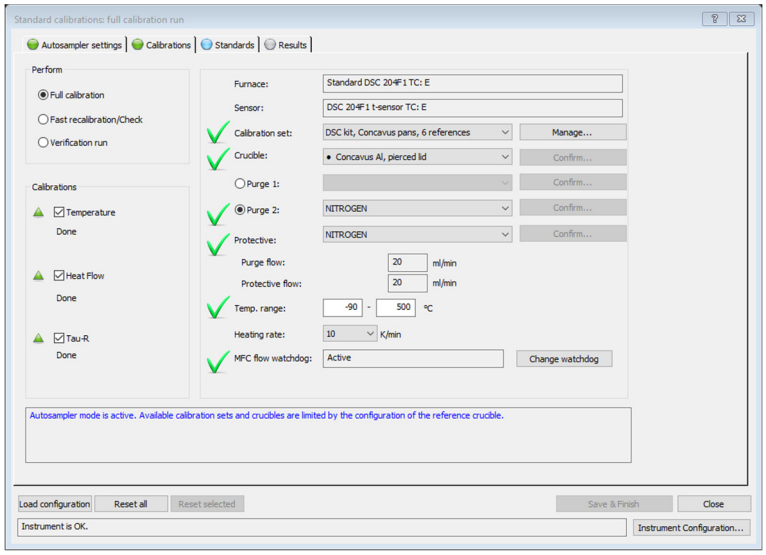
Observe below listed **important notes**:

- ⇨ The instrument must be calibrated at least once per year.
- ⇨ Measurements within limit ranges causes shorter calibration cycles.
- ⇨ It is Operators responsibility to define different calibration cycles/ verification cycles according to valid industry specific standards.
- ⇨ Calibration is required if other equipment (e.g. $LN_2$ cooling device) has been added or removed from the instrument.
- ⇨ Calibration is required after maintenance or repair (for example exchange of sensor, thermocouples or electronic components).
- ⇨ Verify calibration (using Indium) at least once per month.

## Execute a calibration

The calibration of a NETZSCH-DSC Instrument is described by means of a DSC 214 in this manual. The procedures are almost identical for all devices of the NETZSCH DSC series.

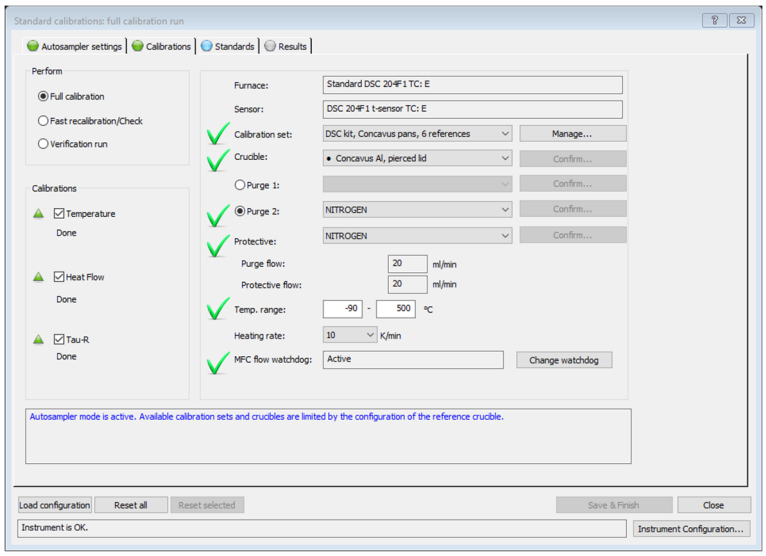
| | |
|---|---|
|  | Run the measurement software. |
|  | Select „Standard calibrations runs…“ under **Measurement**. |
|  | *Autosampler settings* header:<br>The calibration procedure can also be executed by using the ASC.<br>Set checkmark “Activate autosampler mode“ to enable calibration using the ASC.<br>Use the Reference Manager to define position, name, mass, crucible mass and crucible type of the reference. |




*Calibrations* header:

- Use „Full calibration“ for a complete calibration run. See section “Execute a “Full calibration”.
- “Fast recalibration/check“ is used to recalculate the calibration curve by means of a new measurement on Indium.
- “Verification Run” is to verify an existing calibration using one or more calibration standards. See section Perform a “Verification run”.

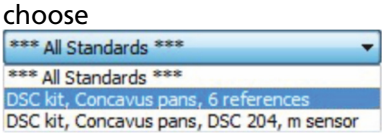
## *Execute a “Full calibration”*




*Calibrations* header:

Various settings can be made here to define the calibrations.

Under “Calibration set”, choose



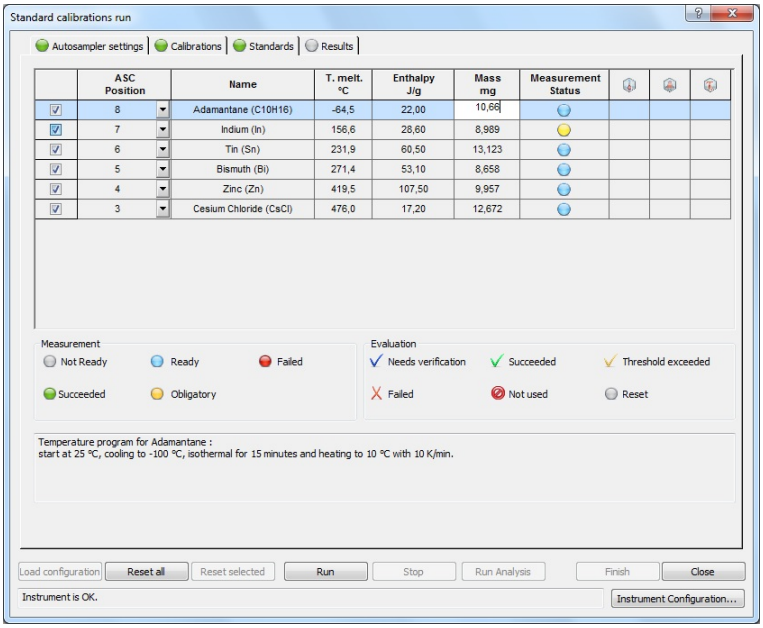

for calibration.

Choose the used crucibles under “Crucible”.

Define used gases and flow rates for the calibration.

The temperature range can be modified, if necessary.

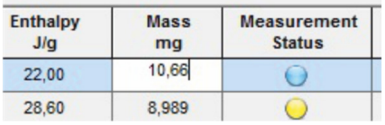
Choose the required heating rate.




*Standards* header:

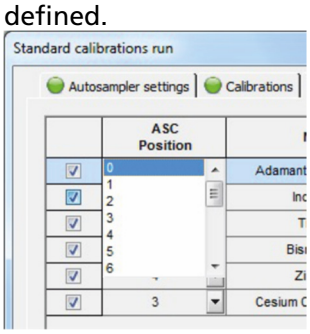
The standards, included in the calibration set are preselected, depending on the temperature range.

Enter the mass for each single standard material under mass:



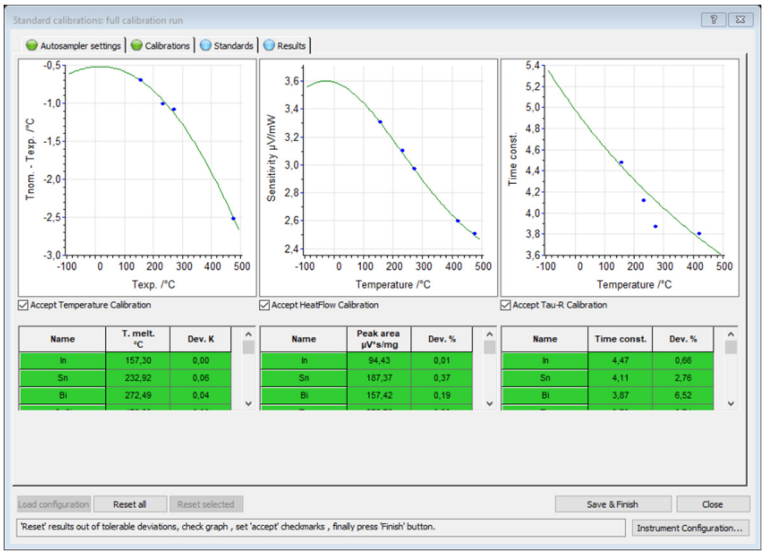

The mass of each standard is to be found inside the cover of the calibration set.

If an ASC-instrument is used, the position of each standard in the ASC must also be defined.




Press "**Run**" to start the calibration procedure.

Follow instructions on screen.

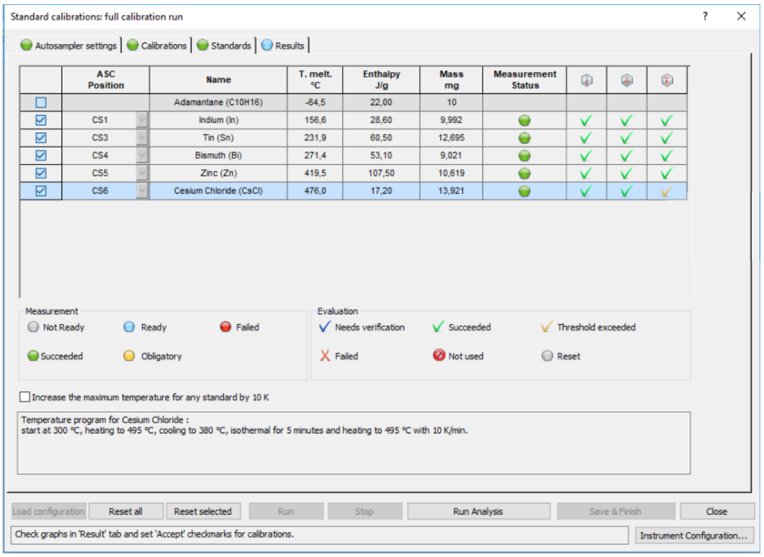



*Results* header:

The executed measurements are automatically evaluated via AutoEvaluation-software. Intermediate results of already measured standards will be displayed while the other calibration runs are still in progress.

The depicted graphs (on left side) for Temperature-, HeatFlux- and TauR Calibration are examples with a good curve shape.

Set the checkmark under the graph to accept the corresponding calibration. At least one of the three calibrations must be accepted to activate the "**Save & Finish**" button.




Rarely it might be necessary to optimize the executed calibration.

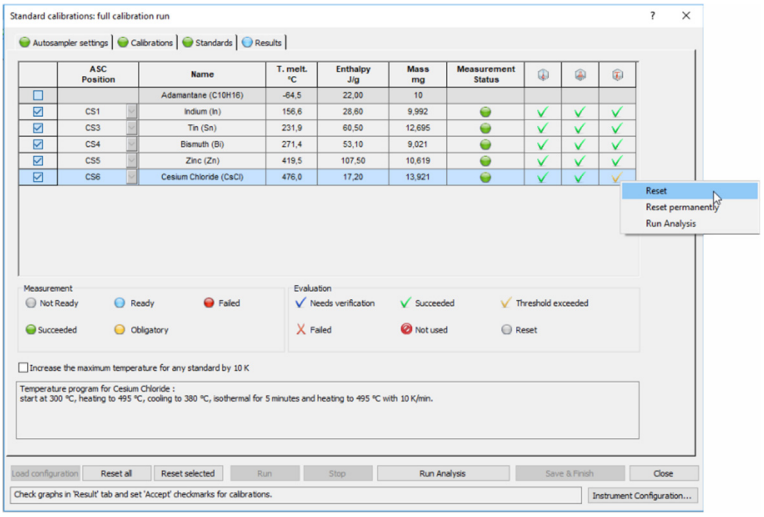
*Standards* header:

The last three columns will show the status of standard measurements.

The state of Cesium Chloride (CsCl) in column Tau-R shows "Threshold exceeded". Here it is necessary to edit the calibration file.

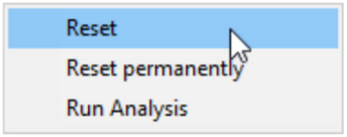
CsCl is yellow highlighted at Tau-R Calibration.

Due to its powder form, CsCl is not suitable for Temperature- and TauR calibrations and values should be removed manually.

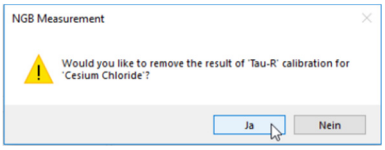


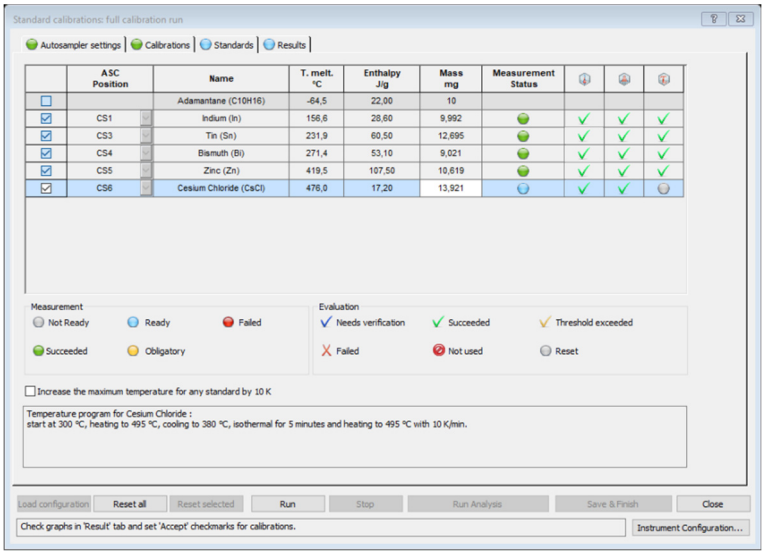

**Green** checkmarks indicates a good value.
**Yellow** checkmarks (and red dot in the graph in *Results* header) indicates a value exceeding the threshold.
A **red** checkmark (and red dot in the graph in *Results* header) indicates a value out of range.

Click on the yellow highlighted checkmark to access the operations:

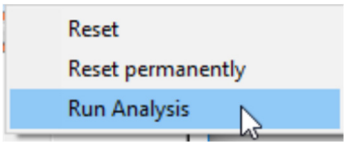


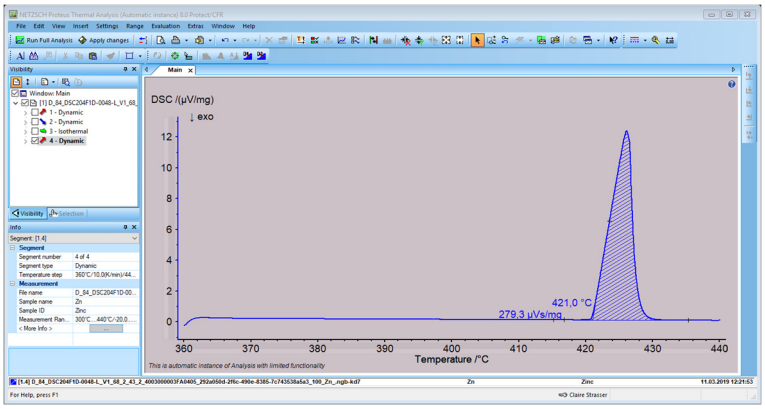

And confirm with "Yes":



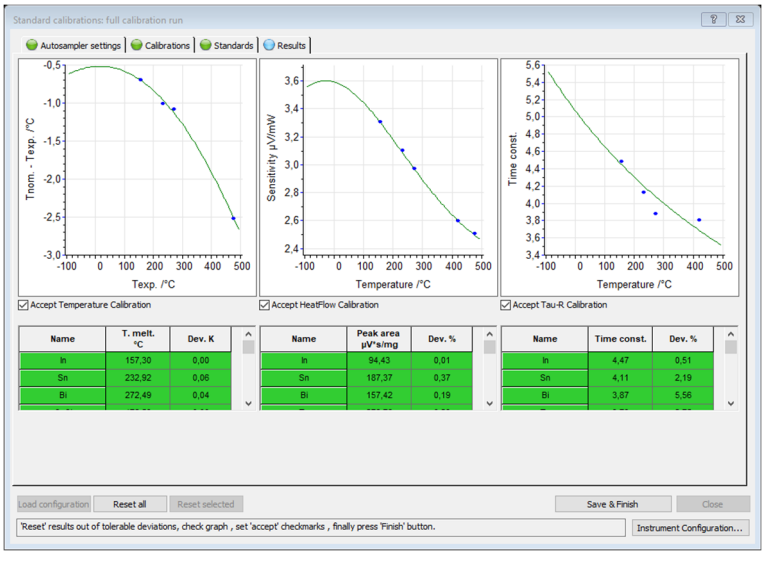



Additionally, the user has the opportunity to open the file in analysis software.




Example for a measurement file, opened in Analysis software.




Press "**Save & Finish**" to complete the procedure. Accepted calibrations are written together with the measurements of the standards as files.

"**Close**" will close the dialog without saving.



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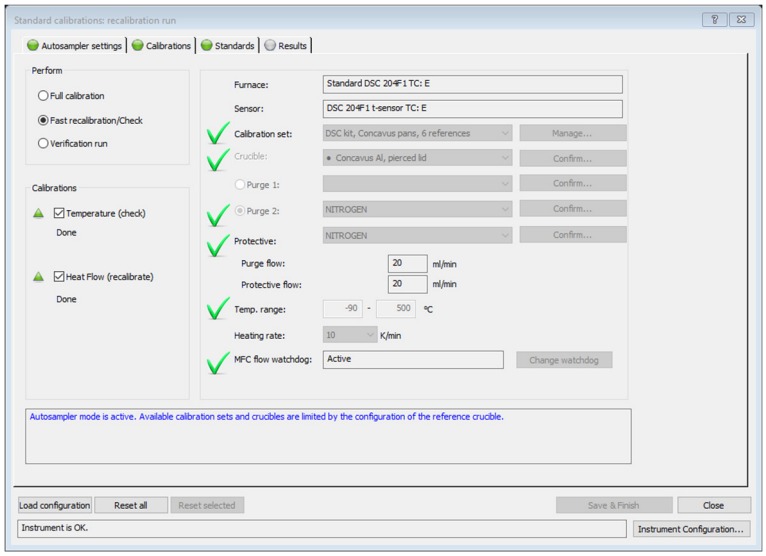
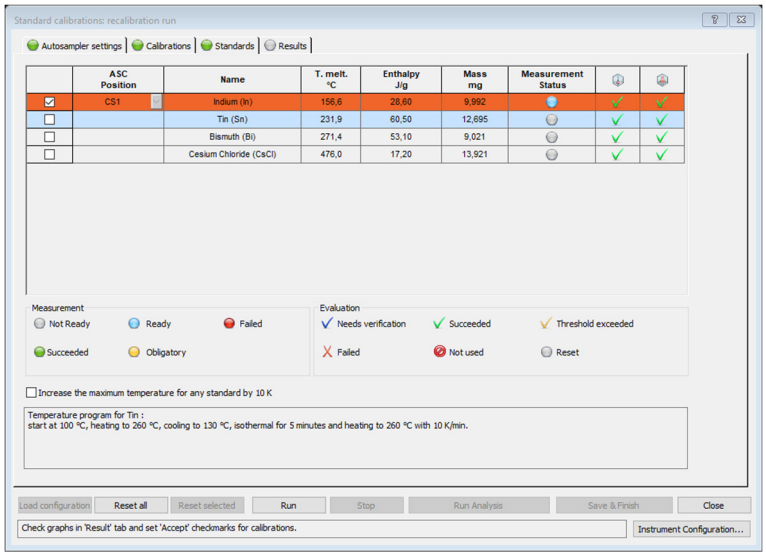
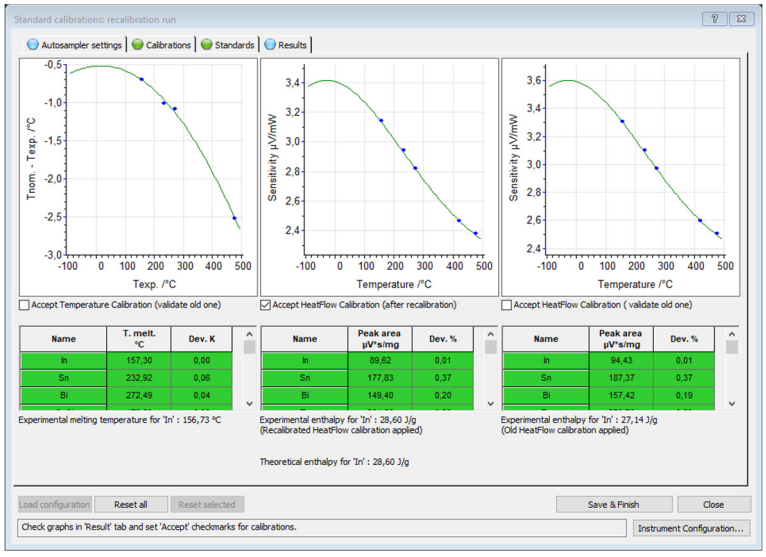
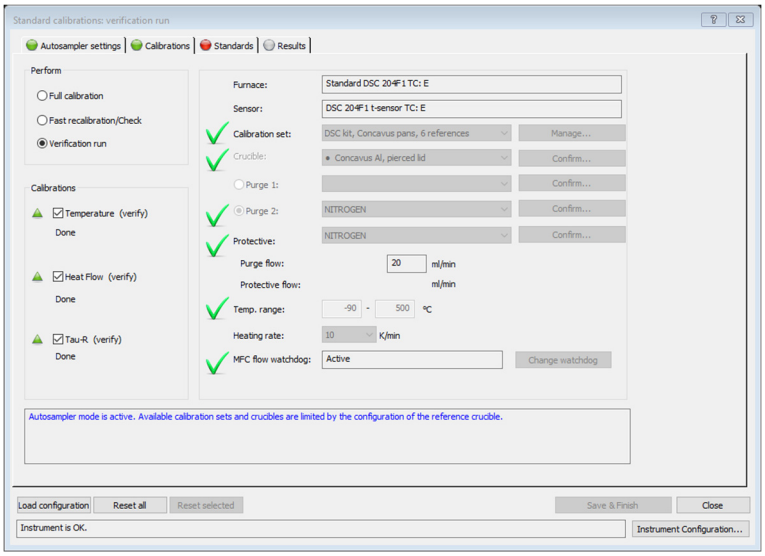
## *Carry out a "Fast recalibration/check"*

| | |
|---|---|
|  | Select this option to recalculate the calibration curve. |
|  | Indium is preselected, click "Run" to start the measurement after insertion of Indium. |
|  | Press "**Save & Finish**" to complete the procedure. |

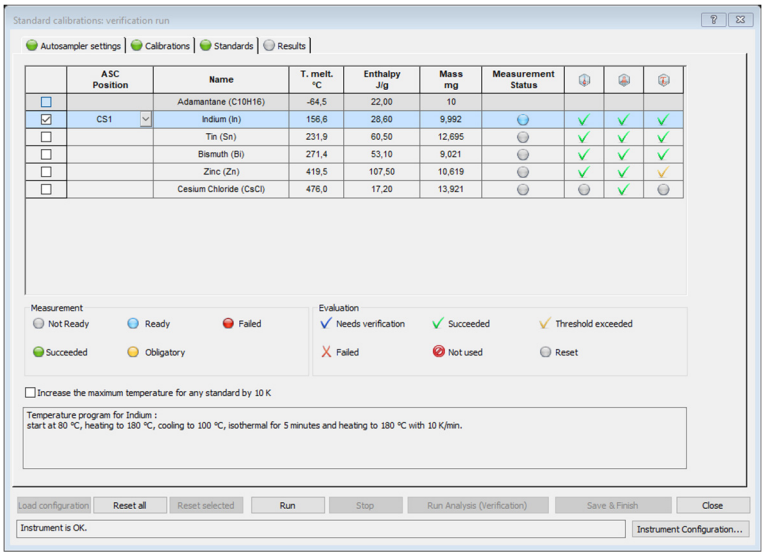
## *Perform a "Verification run"*




*Calibrations* header:

Once "Verification run" is selected, all necessary information of the current active calibration is already loaded.

Press "Load configuration" to choose a different calibration file for verifying.

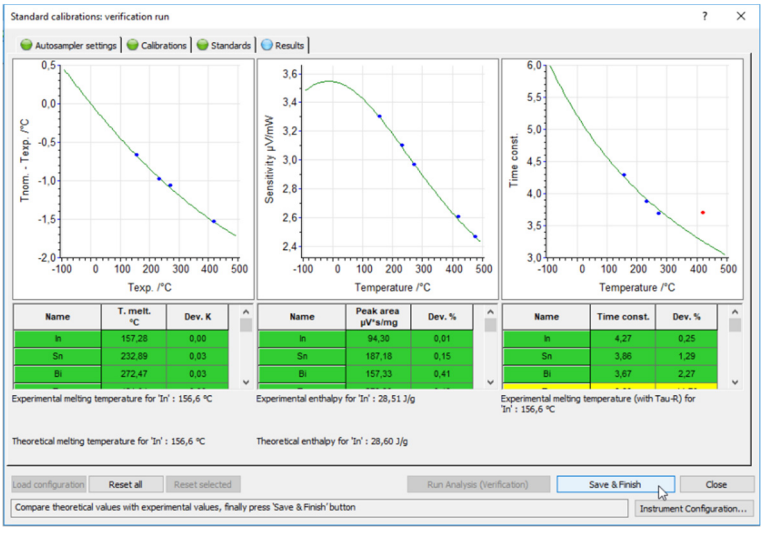



*Standards* header:

Select the standard material(s), which have to be used for verifying the calibration.

Press "Run" to start the procedure.

One standard material must be selected, more standard materials can be selected.



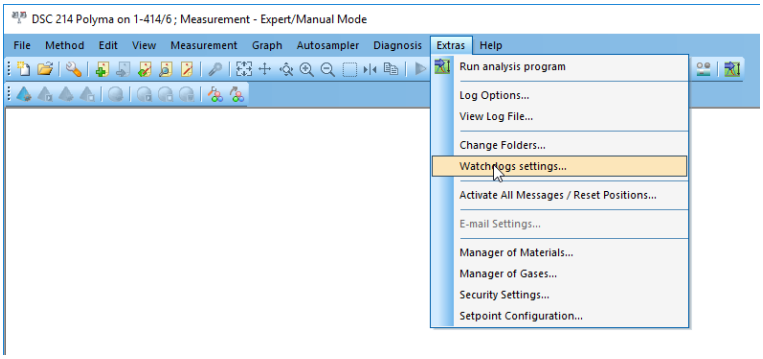
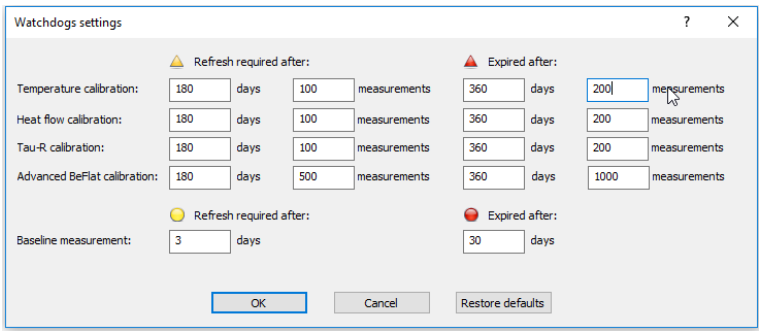

*Results* header:

When the verification run is finished and the result is ok, press "Save & Finish" to complete the procedure.

## Watchdog for calibrations

Since Software version 7.0 Dec.2013 there is also a watchdog for calibrations available.

This watchdog for calibrations option allows the user to define a limited validity of the calibration files and base lines to ensure a periodic refresh or renewal of the files.

<table>
<tr>
<td> 
</td>
<td>Open “Watchdog settings” under Extras.</td>
</tr>
<tr>
<td> 
</td>
<td>Define a validity period for calibrations and/or baselines.<br><br>This period can be defined in days and numbers of measurements.<br><br>  <br>Each measurement will be counted independent of the measurement type.</td>
</tr>
</table>

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