

Software Manual

Proteus 8.x

Temperature- and Sensitivity Calibration for DSC214/ DSC204F1/ DSC3500

51491 / Version 1.1 / April 2019

DOCUMENTATION

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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.

$\mathbf{\Lambda}$	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.

NEUXSCHI Proiteus xx	Run the measurement software.
DSC 214 Polyma on 1-414/6; Measurement - Expert/Manual Mode File Method Edit View Measurement Graph Autosampler Diagnosis Extras Help Image: Imag	Select "Standard calibrations runs" under Measurement .
Standard calibrations rul Image: Calibrations rule	Autosampler settings header: The calibration procedure can also be executed by using the ASC. Set checkmark "Activate autosampler mode" to enable calibration using the ASC. Use the Reference Manager to define position, name, mass, crucible mass and crucible type of the reference.
Load configuration Reset all Reset all Close Instrument is OK. Instrument Configuration	

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Perform					• Us
Full calibration	Furnace:	Standard DSC 204F1 TC: E			
East recalibration/Check	Sensor:	DSC 204F1 t-sensor TC: E			60
	Calibration set:	DSC kit, Concavus pans, 6 references \sim	Manage		Se
Venication run	Crucible:	Concavus Al, pierced lid	Confirm		"E
Calibrations	OPurge 1:	~	Confirm		
🔺 🗹 Temperature	O Purge 2:	NITROGEN	Confirm		• "F
Done	Protective:	NITROGEN	Confirm		
Heat Flow	Purge flow: Protective flow:	20 ml/min 20 ml/min -90 - 500 °C			ca
Done	Heating rate:	10 × K/min Active	Change watchdog		In
Autosampler mode is active. Available	calbration sets and crucibles are limit	ed by the configuration of the reference crucible.			• "\ ve ca
oad configuration Reset all	Reset selected		Save	a Finish Close	m

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"

tandard calibrations: full calibration r	un				8 8	3	Calibrations header:
Autosampler settings Gerform	tions 🔵 Standards 🔘 Results					1	Various sattings can be made
Eul calibration	Furnace:	Standard DSC 204F1 TC: E					various settings can be made
Fast recalibration/Check	Sensor:	DSC 204F1 t-sensor TC: E					here to define the calibrations
O Verification run	Calibration set:	DSC kit, Concavus pans, 6 references	~	Manage			
	Crucible:	Concavus Al, pierced lid	~	Confirm			
Calibrations	O Purge 1:		\sim	Confirm			Under "Calibration set"
🔺 🗹 Temperature	O Purge 2:	NITROGEN	~	Confirm			
Done	Protective:	NITROGEN	~	Confirm			choose
Heat Flow	Purge flow:	20 ml/min					*** All Standards ***
Done	Protective flow:	20 ml/min					*** All Standards ***
	Temp. range:	-90 - 300 °C					DSC kit, Concavus pans, 6 references
A Tau-R Done	Heating rate:	10 V K/min					DSC kit, Concavus pans, DSC 204, m sensor
	V Mind now watchdog:	ALUVE	u	hange watchoog			for calibration.
Autosampler mode is active. Available	calibration sets and crucibles are lim	ted by the configuration of the reference on	ucible.				Choose the used crucibles under "Crucible".
ad configuration Reset all	Reset selected			Save &	Finish Close		Define used gases and flow rates for the calibration.
							The temperature range can be modified, if necessary.
							Choose the required heating rate.

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ndard calibrations: full calibratio							
	in run prations 🛛 🍚 Standards 🕽 🔵 R	(esuits				? ×	Rarely it might be necessary to
ASC Position	Name	T. melt. Ent	halpy Mass J/g mg	Measurement Status	(i)	T	calibration.
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CS3 CS4	Tin (Sn) Bismuth (Bi)	231,9 60 271,4 5:	0,50 12,695 3,10 9,021	<u> </u>			Standards fieader.
CS5 CS6	Zinc (Zn) Cesium Chloride (CsCl)	419,5 10 476,0 17	7,50 10,619 7,20 13,921	e			The last three columns will show the status of standard measurements.
							The state of Cesium Chloride
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ucceeded 🤤 Oblig	atory	X Failed	🙆 Not use	i ⊜ R	eset		"Threshold exceeded". Here it
ease the maximum temperatu	ure for any standard by 10 K						is necessary to edit the
ture program for Cesium Cl 300 °C, heating to 495 °C,	hloride : cooling to 380 °C, isothermal for	r 5 minutes and heating t	o 495 °C with 10 K/min.				calibration file.
guration Reset all	Reset selected	Run Stop	Run Ar	alysis	Save & Finish	Close	CsCl is yellow highlighted at Tau-R Calibration.
graphs in 'Result' tab and set 'A	Accept' checkmarks for calibratio	xns.			Instrument	Configuration	\wedge
) E
							Due to its powder form, CsCl is
							not suitable for Temperature-
							and TauR calibrations and
							values should be removed
							manually.
alibrations: full calibration r	un				? 2	×	Click on the vellow
ampler settings 📔 🍚 Calibrat	ions 📔 🝚 Standards 📔 🔵 Results	i]				1	highlighted checkmark to
ASC Position	Adamantane (C10H16)	-64,5 22,00	Mass Measing St	atus 🕼	(i)		access the operations:
CS1 CS3	Indium (In) Tin (Sn)	156,6 28,60 231,9 60,50	9,992 12,695	 ✓ ✓ ✓ 	\checkmark \checkmark \checkmark		Dent
CS4	Bismuth (Bi) Zinc (Zn)	271,4 53,10 419,5 107,50	9,021 10,619				Reset
CS6	Cesium Chloride (CsCl)	476,0 17,20	13,921	●	Reset	1	Reset permanently
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		Evaluation	V Gurundud	Threshold av			And confirm with "Yes":
urement	Exied	V Needs vermesedri		Reset			
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urement tot Ready Ready ucceeded Oblgat rease the maximum temperature relates program for Cestan Chico ti 300 °C, heating to 495 °C, coo	Failed ry for any standard by 10 K ide : ing to 380 °C, isothermal for 5 min	X Failed	With 10 K/min.				NGB Measurement × Would you like to remove the result of 'Tau-R' calibration for Cesium Chloride ?
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Standard calibrations: full calibration supervised in the set of any standards in Results Image: Calibration supervised in the set of any standards in the set of any standards in the set of any standards by 10 K Passion Name T. metic Enthalpy Mass Measurement Image: Calibration supervised in the set of any standards in the set of any standards in the set of any standards by 10 K Measurement Image: Calibration supervised in the set of any standards by 10 K Evaluation Image: Calibration supervised in the set of any standard by 10 K Image: Calibration Reset Reset Reset Reset Reset in the set of the set of any standards by 20 K Image: Calibration Reset Reset Reset Reset Reset Image: Calibration Reset Reset Reset Reset Reset Image: Calibration Reset Reset Reset Reset Reset Image: Calibration Reset Reset Reset Reset Reset	Additionally, the user has the opportunity to open the file in analysis software. Reset Reset permanently Run Analysis
	Example for a measurement file, opened in Analysis software.
Sended calibrations full calibration un <pre></pre>	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.



Carry out a "Fast recalibration/check"





Perform a "Verification run"



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.

최희 DSC 214 Polyma on 1-414	/6 ; Measurement - Exp	pert/Manual N	lode				Open "Watchdog settings"
File Method Edit View	Measurement Grap	ph Autosamp	ler Diagnosis → I ← 理由 → →	Ettras Help Run analysis program Log Options View Log File Change Folders Watchriggs settings Activate All Messages / I E-mail Settings Manager of Materials Manager of Gases Security Settings	Reset Positions		under Extras .
Watchdogs settings Temperature calibration: Heat flow calibration: Tau-R calibration: Advanced BeFlat calibration: Baseline measurement:	Refresh require 180 days 180 days 180 days 180 days is8 days Refresh require 3 days OK	ed after: 100 100 500 ed after:	measurements measurements measurements Cancel	Expired after: 360 days 360 days 360 days 360 days 360 days We prived after: 30 days Restore defaults	200 meas 200 meas 200 meas 1000 meas	? × urements urements urements	Define a validity period for calibrations and/or baselines. This period can be defined in days and numbers of measurements. Each measurement will be counted independent of the measurement type

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