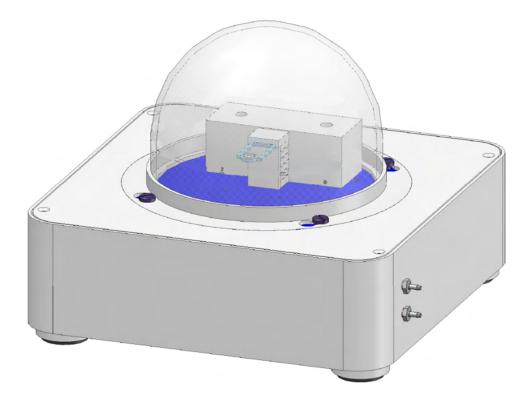


pushing boundaries

# How To calibrate with Chip-DSC



Linseis Messgeräte GmbH Gerlach Date: 18.12.2023



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## 1. General information

This manual is a short description for doing calibrations with the Chip-DSC. The presented features are the most common, that are usually used. However, there are much more features that cannot all be displayed here. For more Information, read the other available instructions about software or specific manuals for the Chip DSC.

# 2. Prepare the device and software

## 2.1 Requirements

- o Make sure your device is connected and complete
- Make sure LINSEIS Platinum software is installed and additional features (e.g. cryo mode) are unlocked by dongle
- o Make sure you have all calibration materials you need for the desired temperature range
- o Make sure all accessories for e.g. low temperature are available

#### 2.2 Setup your device and PC

- Turn on your PC and connect the Chip-DSC device
- o Turn on your Chip-DSC and start the measurement software
- o Select a new Sensor if needed or choose the right one from the dropdown menu

## 3. Run a high temperature calibration

#### 3.1 prepare a sample

- o Select the right crucible for your measurement
- o Select the right calibration standards for the desired temperature range
- o Cut the samples, weigh them and place them in your crucible

3.2 insert your



#### sample

- Place the crucible in the middle of your sensor and make sure the samples do not touch each other. Use tweezers to separate the calibration standards if necessary so they do not touch.
- Close the glass dome of your Chip-DSC



#### 3.3 setup your calibration in easy mode

. Setup	2. Current values	Start schedu	iler 📕 4. Stop Measurement 🕪 5. Evaluate	
		💭 General setting	is 🎦 Temperature profile	
Sample temperature Used calibration:	: -264.88 ×#	Crucible: Name:	Aluminium	•
dle		Weight:	1,000 mg	+
	olates 🕓 Scheduler	Sampling interval: Customer:	0,1s	•
Sensor: Sensor Sensor information	- + -	Folder: Order number:	Test\	•
Calibration: x#	- + - 📀	Type:	Sample measurement	+
		Material:	Sapphire	Ŧ
		Preheat temp	erature	
		40,00 ℃		*

 $\circ$   $\,$  Click on the "+" in the Acquisition software to create a new calibration  $\,$ 

	temperature temperatur		5,00 °C ation	Low temperature calibration
	Indium	•	0,000 mg 🜲	Cyclopentane 🔻 0,000 mg 🗘
$\checkmark$	Tin	•	0,000 mg 🗘	
$\square$	Lead	•	0,000 mg 🖨	
	Zinc	•	0,000 mg 🗘	
			0%	→ Start calibration

- $\circ$  Select the calibration standards you want to use for the calibration.
- o Measure the room temperature using a secondary laboratory thermometer at the DSC-Sensor and enter the temperature value in the software field.
  Enter the correct weight for your samples in the software fields.



## 3.4 setup your calibration in expert mode

.00m	temperature:	2	5,00 °C	
High	temperature o	alibra	ation	Lov
	Indium	•	0,000 mg 🗘	Cyclo
$\square$	Tin	•	0,000 mg 🗘	
$\square$	Lead	•	0,000 mg 🗘	
$\square$	Zinc	•	0,000 mg 🗘	
			0%	
			076	

◦ In easy calibration mode click on "expert mode" in the lower left corner □

SensorCalibration									?	$\times$
Room temperature: 25,1	00 °C			÷	Status					
Adjust Sensor offset	autoamtically before calib	oration			state: Id		and the second			
					Sample te DSC: 0 Power: 1	Vų 000.	::-273.1 ℃ ( 0.0 m	V)		
Sample 1 🔀 Samp	ole 2 🗵			+	Nominal	Temp.: -27	'3.1 ℃			
Name	Calibration sample 1				Log					
Maximum power	65,0 %			-						
Rate:	50,00 K/min			\$						
Wait before start	Stabilize before mea	surement								
Peak 1:	Indium	156,60 °C 🗘 -28,5	900 J/g 🖨 0,0	00 mg 韋						
Peak 2:	Tin	231,93 ℃ 🖨 -60,8	600 J/g 韋 0,0	00 mg 韋						
Peak 3:	Lead	327,46 ℃ 🗘 -24,8	100 J/g 🗘 0,0	00 mg 🜲						
Peak 4:	Zinc	419,53 ℃ 🗘 -112,	4600 J/g 🗘 0,0	00 mg ≑						
Sensor voltage					Start ca	01-15275	755.dat 036.dat 873.dat 983.dat 569.dat 653.dat 13458.dat 15051.dat	Creat	e repor	*
<b>X</b> 200	200 <b>Th</b>	400 60 ermometer volta		300	1.000	01_10_20 02_08_20 02_10_20 02_10_20 02_10_20 02_10_20	21080.dat 18 B12-1.dat 18 ind zinc.dat 18 B12-2.dat 18 B12-3 01.dat 18 TT acetone.dat 18 TRW 01.dat	cel		×

- Measure room temperature at the DSC-sensor and insert the right value in the field.
- Give your calibration run a name □
- Set the heating rate you want to use for the calibration. Note the same rate has to be used for the Sample run.
- $\circ$  Select the calibration standards you want to use  $\Box$
- Insert the right weight for your samples
- o Adjust other Settings if necessary



#### 3.5 Run and finish your high temperature calibration

o Click on "Start calibration" in your calibration window

SensorCalibratio	10		1 ×					
Room temperature:	25,00 °C		Status					
	set autoamtically before	e calbration	state: Idle Sample temperature: -273.1 *C ( 0.0 miV) DSC: 0.000 µV Power: 0.00 %					
Sample 1 🚺 S	Sample 2 🖸	•	Normal Temp.: -273.1 °C	SensorCalibrat	ion		?	×
Name	Calbration sample	1	Log					
Naximum power	65,0 %	\$		Room temperature:	25,00 °C	<b></b>		
Rote:	50,00 Kjimin	:		High temperature	calibration	Low temperatu	re calibration	
	art 🗹 Stabilize before			rightemperature	calbratori	Cow temperatu		
Peak 1:	Indum	156,60 °C 4 -28,5900 J/g 4 0,000 mg 4		Indium	▼ 0,000 mg 🗘	Cyclopentane	▼ 0,000 mg	•
Peak 2:	Tin	231,93 °C C 40,8600 J/g C 0,000 mg C		Tin	▼ 0,000 mg 🗘			
Peak 4:	Lead	327,46 °C 2 -24,8100 J/g 2 0,000 mg 2						
Peak 4	Zinc	419,53 °C 0 -112,4600 3/g 0 0,000 mg 0		Lead	▼ 0,000 mg ≑			
			Start calibration Stop calibration Greate report	Zinc Zinc	▼ 0,000 mg 🗘			
1.000 800			-1527517553.dat A -1527594755.dat -1527994756.dat -152769473.dat -1528721963.dat		0%	-	Start calibratic	n
sor voltage			- 1528123569.dat - 1528155553.dat .dat 01-1527513458.dat	Expert mode				
S 200	200	400 600 800 Thermometer voltage [mV]	01-132731501.dat 01-132731501.dat 01.10,2016 81-3.1.dat 02,00,2016 81-3.1.dat 02,00,2016 81-3.0.dat 02,10,2016 81-3.0.1.dat 02,00,2016 81-3.0.0.1.dat 02,00,2016 81-3.0.1.dat 02,00,2016 81-3.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0.0					

o Wait until the calibration finished

New Sensor	2	
Name:		

o In the first message popup insert the name for your calibration curve

Sample	Real temperature	Measured voltage	Calibration factor
2 🗹 Sample 1	231.9 °C	147.371 mV	575.513 mW/(mV*s)
3 🗹 Sample 1	327.46 °C	175.674 mV	756.961 mW/(mV*s)
4 Sample 1	419.53 °C	199.015 mV	704.278 mW/(mV*s)
4 <mark>1⊡</mark> ⊳ampie i	419.53 °C	199.015 mV	704.278 mw/(mv*s)

• Check the values of your calibration to see if everything worked well. If not try again without touching the calibration standards (the shape of your samples is better after first run since the sample makes better contact with the crucible.)

0	Click	"Ok"

o Insert the name for the saved calibration



## 4. Run a low temperature calibration

## 4.1 prepare a sample

- o Select the right crucible for your measurement
- o Select the right calibration standard for the wanted temperature range
- Fill in your sample with syringe (only if you use crimpable crucible, otherwise wait until 4.4 finished) and close crucible
- o Turn on Heating Pad in General Settings

Idle		
Instrument	Templates	🐹 Scheduler
Sensor: Sen		- + -
Calibration:	12042023 Dir	- +
Heating p	ad enabled	

## Tips and Tricks:

- The first sample is always active
- For enthalpy calibration minimum two melting points are necessary
- To calibrate samples one by one in separated crucibles you can repeat calibration runs or add samples by clicking "+" in expert menu
- In case you want to use the Linseis Calibration Disc check the specific HowTo



### 4.2 setup your device in easy mode

	💭 General setting	gs 🌇 Temperature profile	
Sample temperature: -264.88 Used calibration: x#	Crucible: Name:	Aluminium	
dle	Weight:	1,000 mg	
Instrument Templates 😔 Scheduler	Sampling interval: Customer:	0,15	-
Sensor: Sensor   Sensor information	Folder: Order number:	Test\	•
Calibration: 🗴 🗰 🖛 🌍	Type:	Sample measurement	
	Material:	Sapphire	

• Click on the "+" to create a new calibration

	temperature:		5,00 °C	
High	temperature c	alibra	ation	Low temperature calibration
	Indium	•	0,000 mg ≑	Cyclopentane 🔻 0,000 mg
	Tin	•	0,000 mg 🖨	
	Lead	•	0,000 mg 🖨	
$\square$	Zinc	•	0,000 mg 🖨	
				_
			0%	→ Start calibration

- $\circ$  Select the calibration standard you want to use for the calibration.
- $\circ$  Measure the room temperature at the DSC-Sensor and insert the right value  $\square$
- $\circ$  Insert the right weight for your sample  $\Box$



## 4.3 setup your device in expert mode

JOIL	temperature:	25,00 °C	
High	temperature c	alibration	Low
	Indium	▼ 0,000 mg	Cydop
$\square$	Tin	▼ 0,000 mg	3
$\square$	Lead	▼ 0,000 mg	3
	Zinc	▼ 0,000 mg	3

o In easy calibration mode click on "expert mode" in the lower left corner

temperature	: 25,00 °C	:					<b>+</b>	Status				
djust Sensor o alibrate entha		amtically befo	ore calibration					DSC: 0	emperature 1.000 µV	:-273.1 °C ( 0.0 m	nV)	
mple 1 🔀	Sample 2	×					+	Power: Nominal	0.00 % Temp.: -27	3.1 ℃		
me	Cal	bration samp	ole 1					Log				
		0 %					ŧ					
ximum power												
e:		00 K/min		54			-					
Wait before			ore measureme									
ak 1:	Ind	um		56,60 °C 😫		€ 0,000 mg						
Peak 2:	Tin		2	31,93 ℃ 🗘		¢ 0,000 mg	÷					
Peak 3:	Lea	d	3.	27,46 °C 🗘	-24,8100 J/g	\$ 0,000 mg	-					
Peak 4:	Zind		4	19,53 °C 🗘	-112,4600 J/g	\$ 0,000 mg	-					
800		·		n 00 ometer v		800	- 1	1.000	02_08_20 02_10_20 02_10_20 02_10_20	755.dat 373.dat 373.dat 383.dat 369.dat 3553.dat 13458.dat 15051.dat 21080.dat 8 B12-1.dat 8 B12-1.dat 8 B12-2.dat 8 B12-2.dat 8 B12-2.dat 8 B12-3 01.dat 8 TT acetone.dat 8 TRW 01.dat		
									01 00 00			
mode										Car	ncel	Ok
o Mea			-		DSC-sense	or and ins	sert the	e right	value			
	e vour	calibrat	tion curv	e a name	e 🗖							
o Give	,											
			ate you w	ant to c	alibrate witl	ר 🗖						

- Select "Wait before start" []
- Unselect "Stabilize before measurement" and "Calibrate Enthalpy" (depends on your needs)
- Limit "maximum power" to 1 % 2 %



×

-

## 4.4 Run the calibration and go on with sample

	0 *C				Status										
Adjust Sensor offset a Calibrate enthalpy Sample 1 Sample		ration			Power	Ide temperature: -273.1 °C ( 0.0 mil) 0.000 yW : 0.00 % al Temp273.1 °C	Se Se	ensorCalibratio	on						?
	Calbration sample 1				Log		Room	temperature:	25	.00 °C		\$			
Maximum power	65,0 %			0											
Rote:	50,00 Kjimin						High	n temperature c	alibra	tion		Low temperat	ure c	alloration	
Wait before start	Stabilize before meas							Indium	-	0,000 mg	\$	Cyclopentane	-	0,000 mg	
Peak 1:	Indum			0.000 mg 0			-		_						
-	Tin			© 0.000 mg ©				Tin	•	0,000 mg	ŧ				
	Lead			0.000 mg				Lead	-	0,000 mg	\$				
Peak 4:	Znc	419,53 °C 🗘	-112,4600 3/g	© 0,000 mg ©					_						
					Start	calbration Stop calbration Greate report		Zinc	•	0,000 mg	÷				
1.000						-1527517553.det A				0%			$\rightarrow$	Start calibra	ation
voltage						- 1527599036.dat - 1527674873.dat - 1528121868.dat - 1528123598.dat - 152815553.dat dat	Expert	t mode							
400   1   1   1   1   1   1   1   1   1						07-1527513458.dat 07-1527515051.dat 07-1527515050.dat 07.10_2088 B12-1.dat 02_08_2085 ind ainc.dat 02_01_2088 B12-2.dat 02_10_2088 B12-3.dat									

o Wait until room temperature measurement and sensor adjust is done

Prepa	are sample and instrument	$\times$
?	Please continue when sample 1 is re	ady.
	ОК	

- o When window "continue when sample samplename is ready" appears start cooling
- o If the device is cold enough insert the sample in your crucible and put the crucible on the sensor.
- Make sure sample is frozen
- If Temperature is 20 K 30 K below onset temperature of your sample wait until LN2 is gone (if you use quenching option)
- In the "Continue when sample samplename is ready" window click Ok □



## 4.5 finish your low temperature calibration

 $\circ$   $\quad$  Wait until the calibration finished

🔛 New Sen	sor	?	×
Name:			
	c	Can	

- o In the first message popup insert the name for your calibration curve
- o Check the values of your calibration if everything worked well. If not try again
- o Click "Ok"

🔛 New Sensor	?	$\times$
Name:		
ОК	Can	cel

o Insert the name for the saved calibration

Tips a	nd Tricks:
0	Low temperature calibration starts always at room temperature without cooling
0	In case of not hermetically sealed crucible enthalpy calibration can be turned off
	due to inaccuracies caused by sample evaporation
0	For liquids its just possible to calibrate materials one by one
0	For enthalpy calibration minimum two melting points are necessary
0	During cooling it is highly recommended to enable Heating Pad to avoid
	condensation inside the device



# 5. Find and select your calibrations

### 5.1 Select your (multiple) calibrations

• Click on "+" in the acquisition software to create a new calibration

				?	× SensorCalibration	
Room temperature: 25	5.00 °C		÷	Status	Room temperature: 25,00 °C	
	t autoamtically before cal	ibration		state: Idle	High temperature calibration	Low
				Sample temperature: -273.1 ℃ ( 0.0 mV ) DSC: 0.000 µV	Indium 🔻 0,000 mg 🖨	Cydop
				Power: 0.00 % Nominal Temp.: -273.1 °C	☑ Tin ▼ 0,000 mg 🐳	
Sample 1 🔀 🛛 Sa	mple 2 🖾		+	Nominal Temp.: -275.1 *C	✓ Lead ▼ 0,000 mg ♀	
Name	Calibration sample 1			Log	Zinc • 0,000 mg ÷	
Maximum power	65,0 %		\$			
Rate:	50,00 K/min		÷		0%	
Wait before start	Stabilize before me	asurement				
Peak 1:	Indium	156,60 ℃ 🗘 -28,5900 J/g	g 🗘 0,000 mg 🗘		Expert mode	
Peak 2:	Tin	231,93 ℃ 🖨 -60,8600 J/g				
Peak 3:	Lead	327,46 °C  + -24,8100 J/g				
Peak 4:			Land Land			
M Peak 4:	Zinc	419,53 °C 🗘 -112,4600 J	/g 🗘 0,000 mg 🗘			
				Start calibration Stop calibration Create report		
1.000				-1527517553.dat	^	
-				-1527594755.dat -1527599036.dat		
800				-1527674873.dat		
8 1				-1528121983.dat		
ro -				-1528123569.dat		
+ 600 -				-1528185653.dat		
100 - Oft				.dat		
r volt						
100 tolt				01-1527513458.dat		
6000 to 1000 t				01-1527513458.dat 01-1527515051.dat		
Sensor vo				01-1527513458.dat 01-1527515051.dat 01-1527521080.dat		
Sensor volt 400				01-1527513458.dat 01-1527515051.dat 01-1527521080.dat 01_10_2018 B12-1.dat		
				01-1527513458.dat 01-1527515051.dat 01-1527521080.dat 01_10_2018 B12-1.dat 0_0_08_2018 ind zinc.dat		
				01-1527513458.dat 01-152751005.ldat 01-1527521000.dat 01_10_2018 B12-1.dat 02_08_2018 ind zinc.dat 02_10_2018 B12-2.dat		
200 -				01-1527513458.dat 01-1527515051.dat 01-1527521080.dat 01_10_2018 B12-1.dat 02_08_2018 ind zinc.dat 02_10_2018 B12-3.01.dat 02_10_2018 B12-3.01.dat		
200 -	200	400 600 rermometer voltage [1	800	01-1527513458.dat 01-152751005.ldat 01-1527521000.dat 01_10_2018 B12-1.dat 02_08_2018 ind zinc.dat 02_10_2018 B12-2.dat		

- o Click on "Expert mode" in the lower left corner
- o Select the calibration you want to use in the right window
- If you want to use more than one calibration curve select multiple calibrations by pressing "Ctrl" and select the calibrations you want
- o Right click on one of your wanted calibrations and select load



the second s		÷		A	djust sen	sor offset		
ust Sensor <mark>offs</mark> et autoa	ntically before calibration		Status					
brata onthalow					-			
Peak selection				?	×			
Sample	Real temperature 156.6 °C	Measured vo 125.573 mV	10. <b>1</b> . 10. 10. 10. 10. 10. 10. 10. 10. 10. 10	oration facto	ər			
2 🗹 Sample 1	231.9 °C	147.371 mV	575.513 m	W/(mV*s)				
3 🗹 Sample 1	327.46 °C	175.674 mV	756.961 m	IW/(mV*s)	۱۸	i		
4 🗹 Sample 1	419.53 °C	199.015 mV	704.278 m	W/(mV*s)				
<		1			>			
			ОК	Cance	-	1.		
						Ston	calibration	
0,1 -		J.		-	152950	4343.dat		
0,1 ,05 ,05 ,05 ,01					152950 152991 153001 153002 153008 153008 29_06_ 30_07_ 31_07_ 31_07_ 31_07_ 31_07_			

- Select the onset temperatures you want to use
- o Click "Ok" 🔲
- o Select the enthalpies you want to use
- o Click "Ok"
- $\circ$  Click "Ok" in the calibration window  $\square$



o Insert a name for the saved calibration



## 5.2 Find your calibration on your PC

• Open your Data explorer (WIN + E)

ChipDSC	14.01.2020 11:22	Dateiordner
driver	04.12.2019 10:57	Dateiordner
EDSC	04.12.2019 10:57	Dateiordner
	11.12.2019 16:14	Dateiordner
🔄 Log	04.12.2019 10:57	Dateiordner
📑 manuals	04.12.2019 10:54	Dateiordner
ReportTemplates	04.12.2019 10:54	Dateiordner
SetupFiles	14.01.2020 11:47	Dateiordner
	15.01.2020 12:08	Dateiordner
	04.12.2019 10:57	Dateiordner

- Go to your Linseis data directory (normally C:/Linseis)
- Go to C:/Linseis/EDSC or C:/Linseis/ChipDSC (depends on version) □
- $\circ$   $\$  Here you find the .dat-files of your calibration curves