
TEMPERATURE CALIBRATION

Common and Recommended Methods



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DSC-Workshop

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AGENDA

- temperature calibration step by step (recommended by GEFTA)
 - calibration substance
 - preparation
 - measurement
 - analysis
 - correction
- mistakes in common T-calibration
 - one calibration substance
 - one heating rate

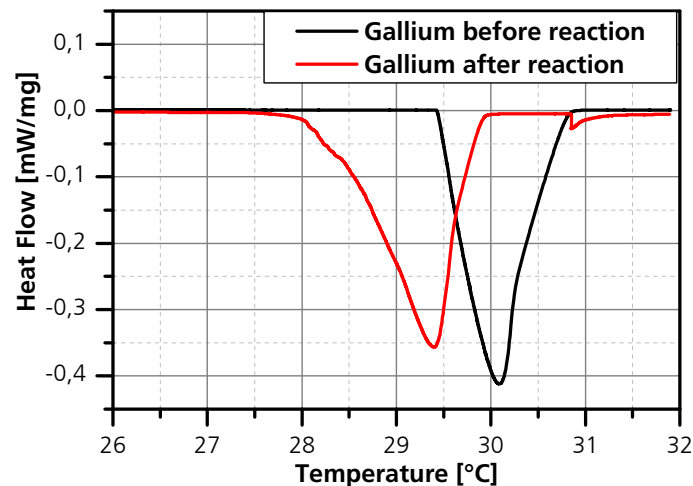
Temperature Calibration Step by Step

Selection of Calibration Substances

■ select 3 calibration substances

■ covering the desired temperature range

■ no reaction with crucible



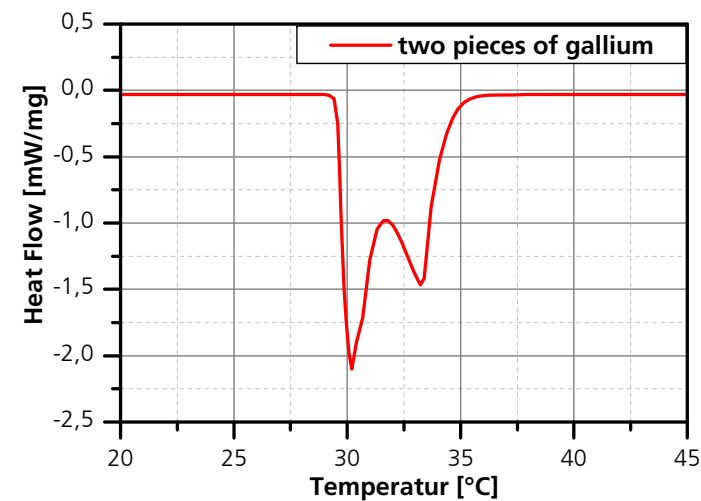
substance	transition temperature		uncertainty ΔT in mK	transition *
	T in K	t in °C		
Cyclopentane	122.38	-150.77	50	s/s
Cyclopentane	138.06	-135.09	50	s/s
Cyclopentane	179.72	- 93.43	50	s/l
Water	273.15	0.00	10	s/l
Gallium	302.9146	29.7646		s/l
Indium	429.7485	156.5985		s/l
Tin	505.078	231.928		s/l
Lead	600.61	327.46	10	s/l
Zinc	692.677	419.527		s/l
Lithium sulphate	851.43	578.28	250	s/s
Aluminium	933.473	660.323		s/l
Silver	1234.93	961.78		s/l
Gold	1337.33	1064.18		s/l

[1] Höhne, G.W.H., W. Hemminger, and H.J. Flammersheim, *Differential Scanning Calorimetry*. 1996: Springer Berlin Heidelberg.

Temperature Calibration Step by Step

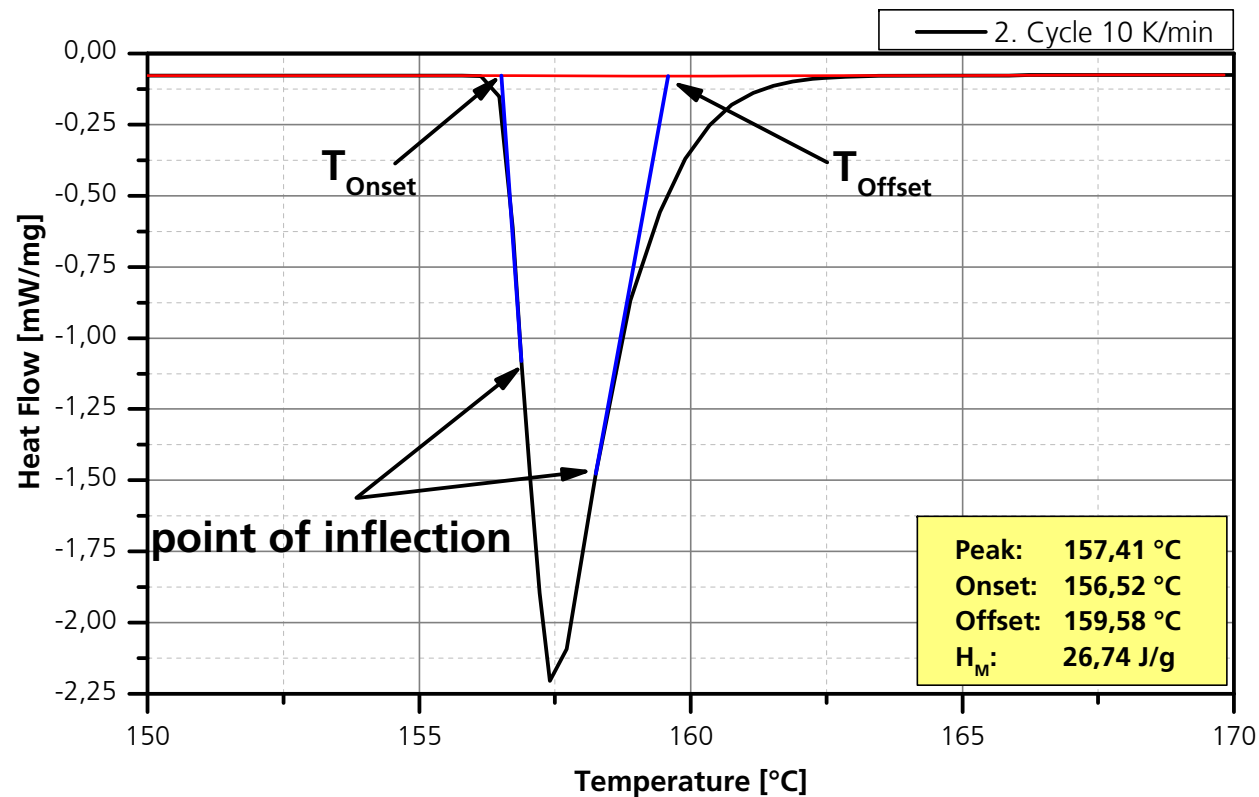
Preparation and Measurement of Calibration Substances

- 2 samples for each calibration substance
- typical sample mass (e.g 10 mg)
- one piece in crucible
- at least 3 heating rates
 - including the smallest one
- one „premelting“ cycle



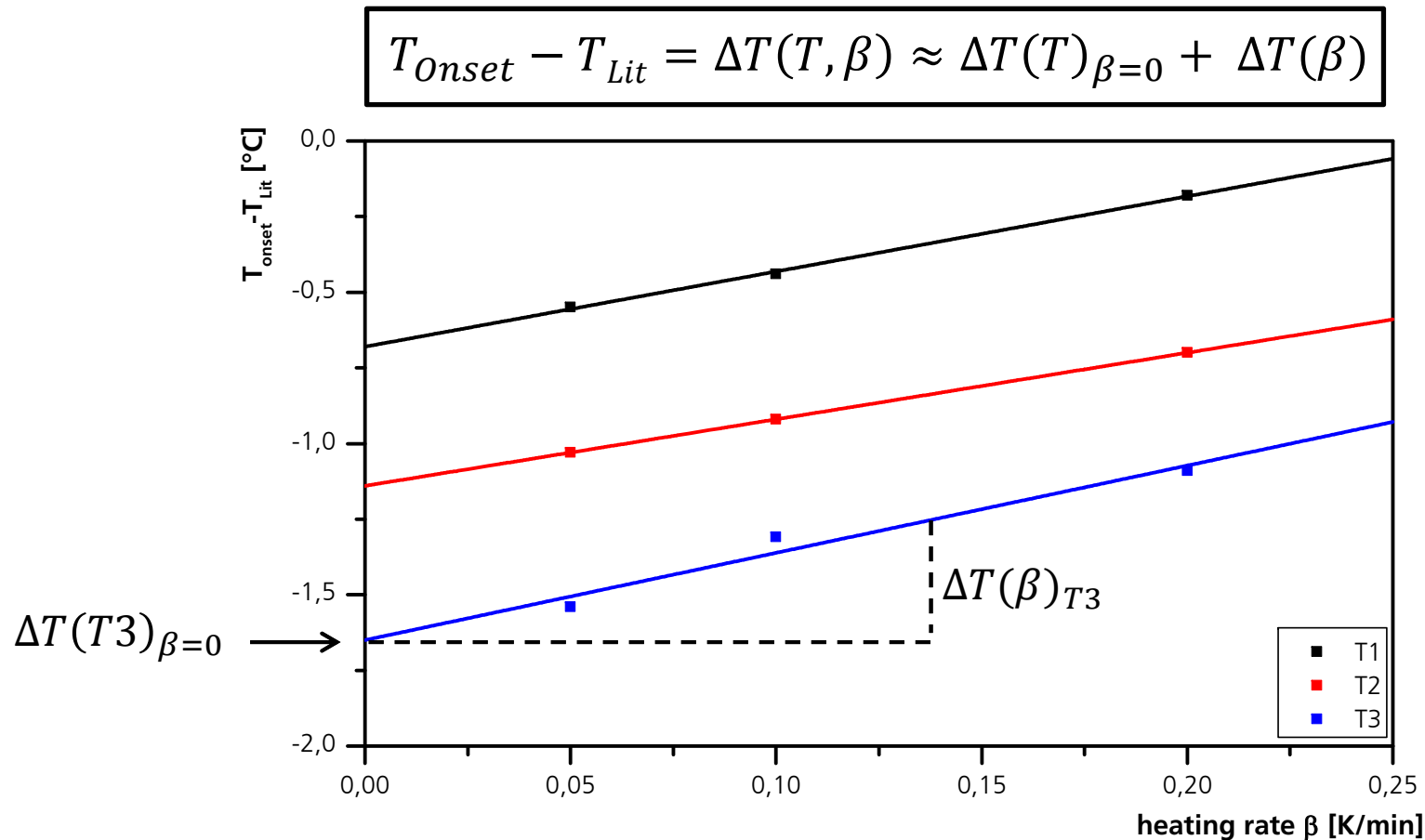
Temperature Calibration Step by Step

Analysis of Heat Flow



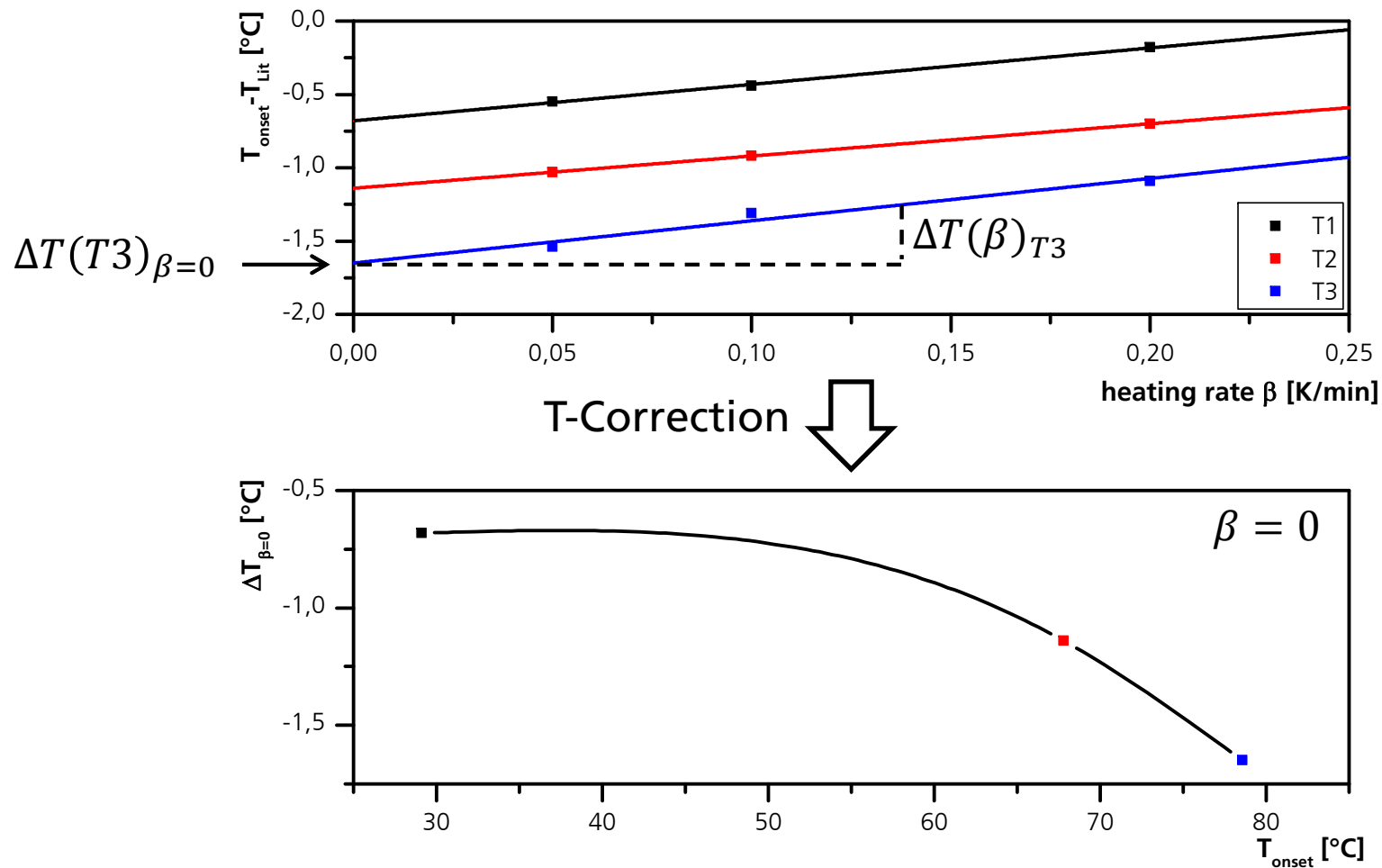
Temperature Calibration Step by Step

Comparison of T_{Onset} with Literature



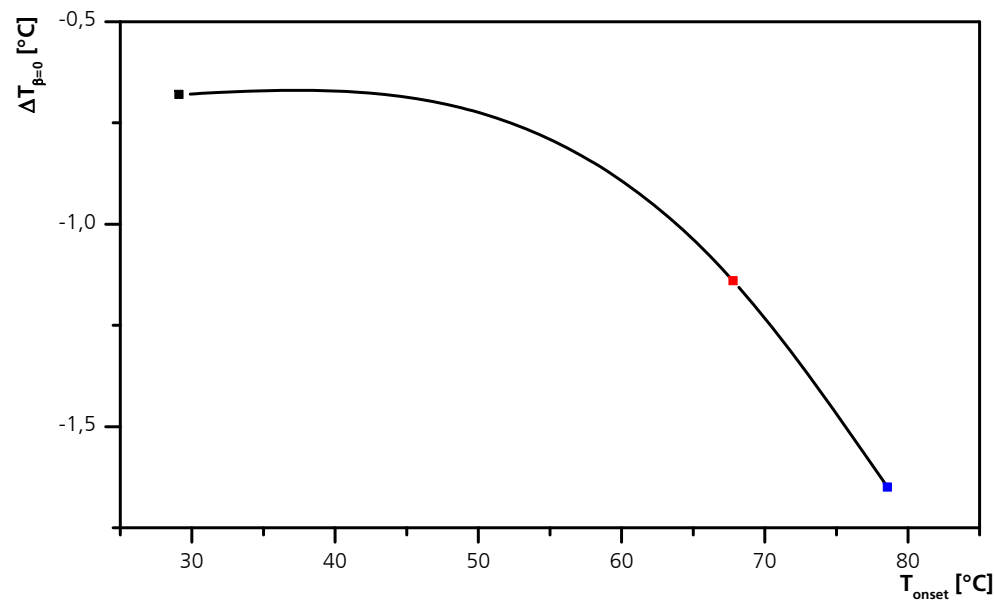
Temperature Calibration Step by Step

Temperature Dependence Correction



Temperature Calibration Step by Step

Set Temperature Correction



Multi-Calibration Sets

Calibration Set: 1)

Cell Constant (Wednesday, August 06, 2014, 13:43:30)

Cell Constant: 1.0600 Onset Slope: 0.0000 mW/ $^{\circ}\text{C}$

Temperature Table

	Observed Temperature $^{\circ}\text{C}$	Correct Temperature $^{\circ}\text{C}$
1	29.23	29.78
2	67.90	68.93
3	78.69	80.23
4		
5		

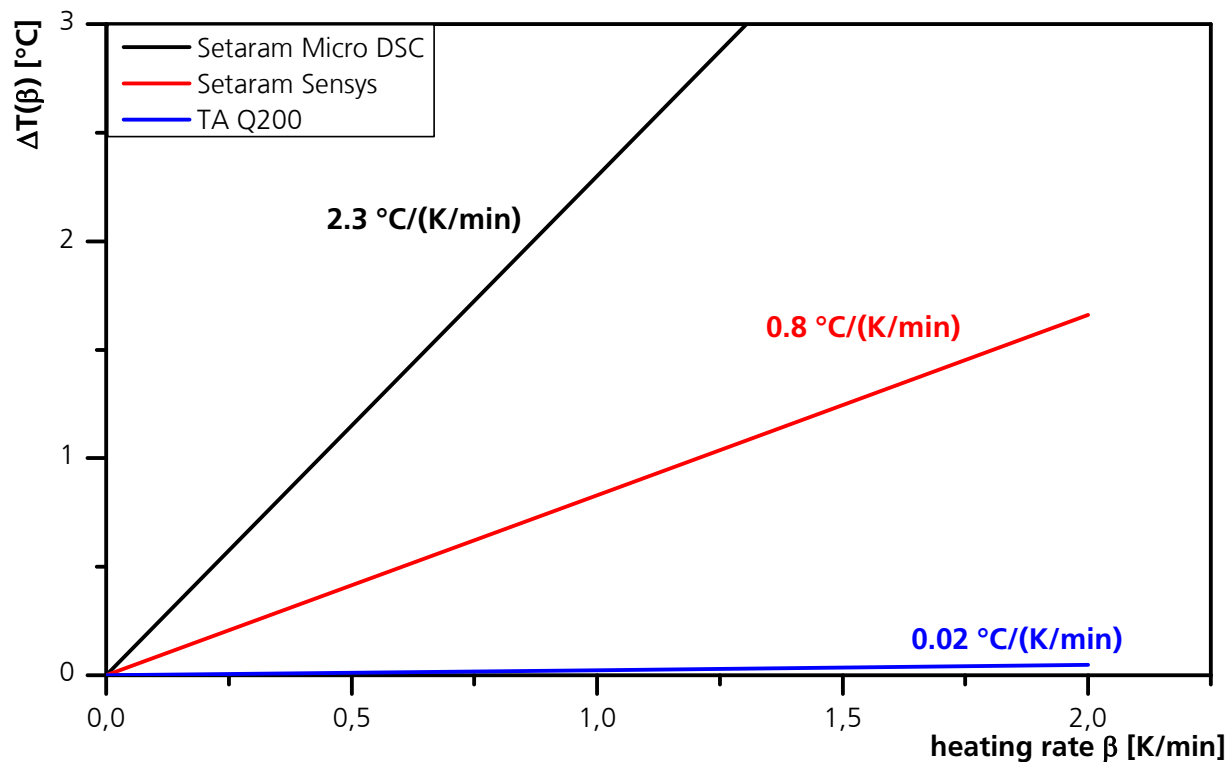
MDSC Cp Constant (Total) 1.000

MDSC Cp Constant (Reversing) 1.000

Reset this Set Reset All OK Close Help

Temperature Calibration Step by Step

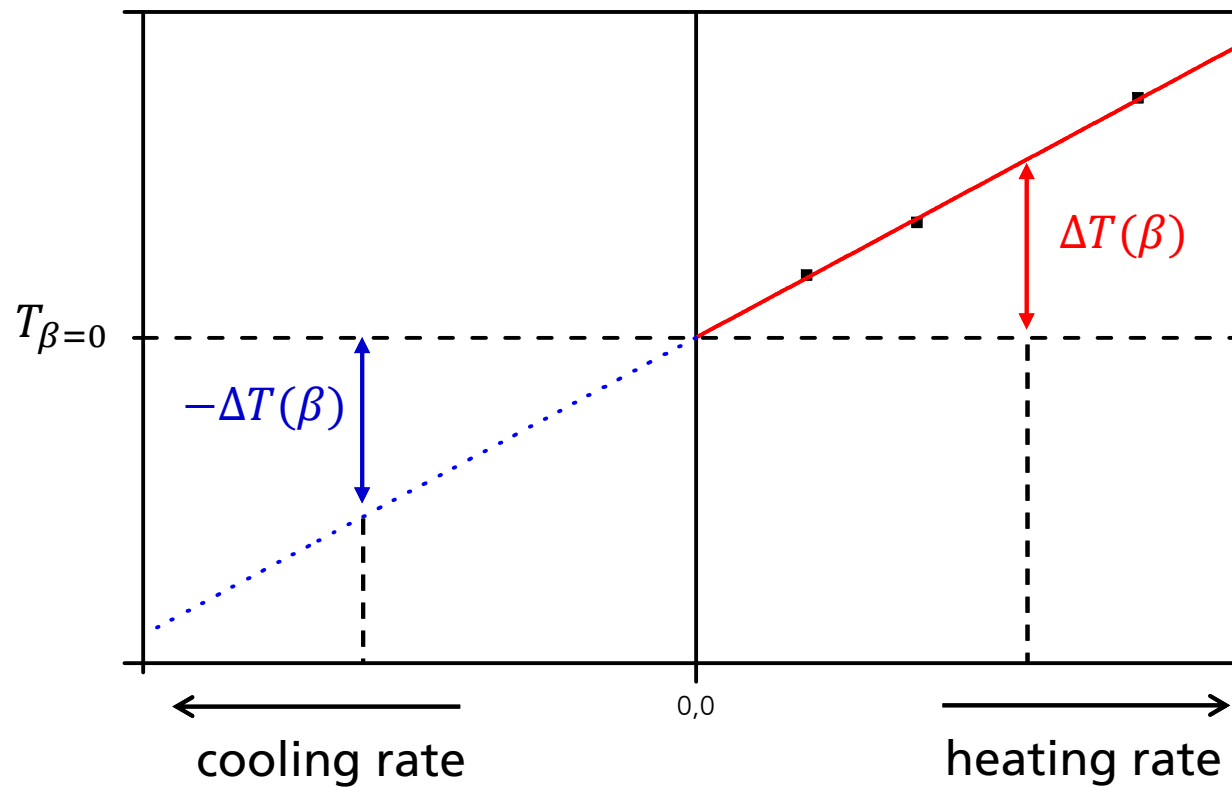
Dependence on Heating Rate, $\Delta T(\beta)$



DSC	typ. β [K/min]	$\Delta T(\beta)$ [°C]
μ DSC	~ 0.05	~ 0.1
Sensys	~ 1.0	~ 0.8
Q200	~ 1.0	~ 0.02

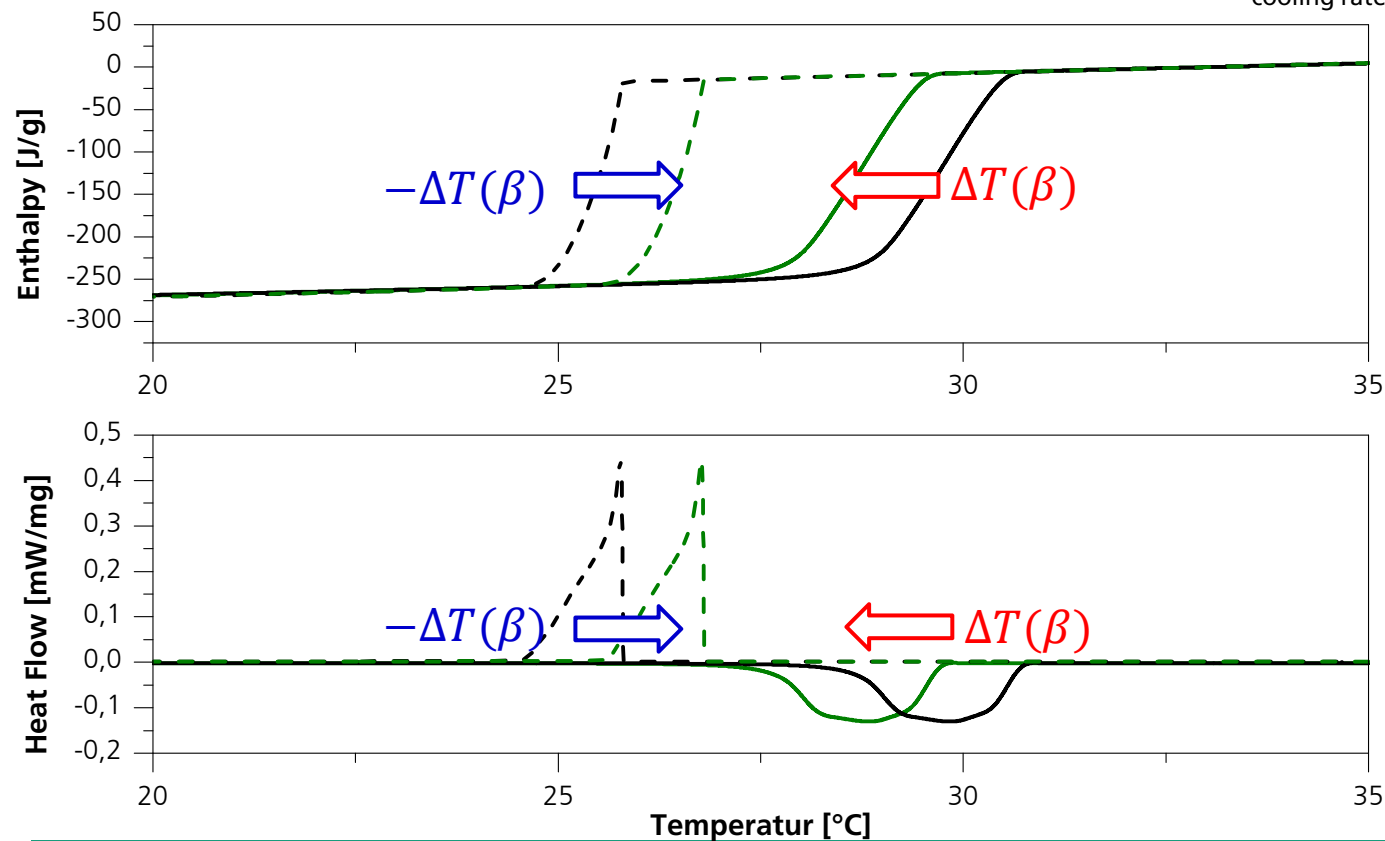
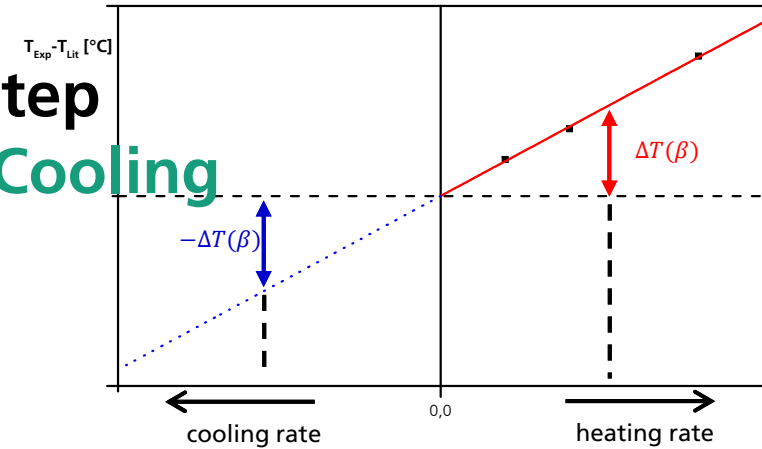
Temperature Calibration Step by Step

$\Delta T(\beta)$ -Correction for Heating and Cooling



Temperature Calibration Step by Step

$\Delta T(\beta)$ -Correction for Heating and Cooling



Mistakes in Common Temperature Calibration

calibration:

- with one substance
 - e.g. Indium @ 156.6 °C
- at one heating rate
 - e.g. 10 K/min

problem: measurements

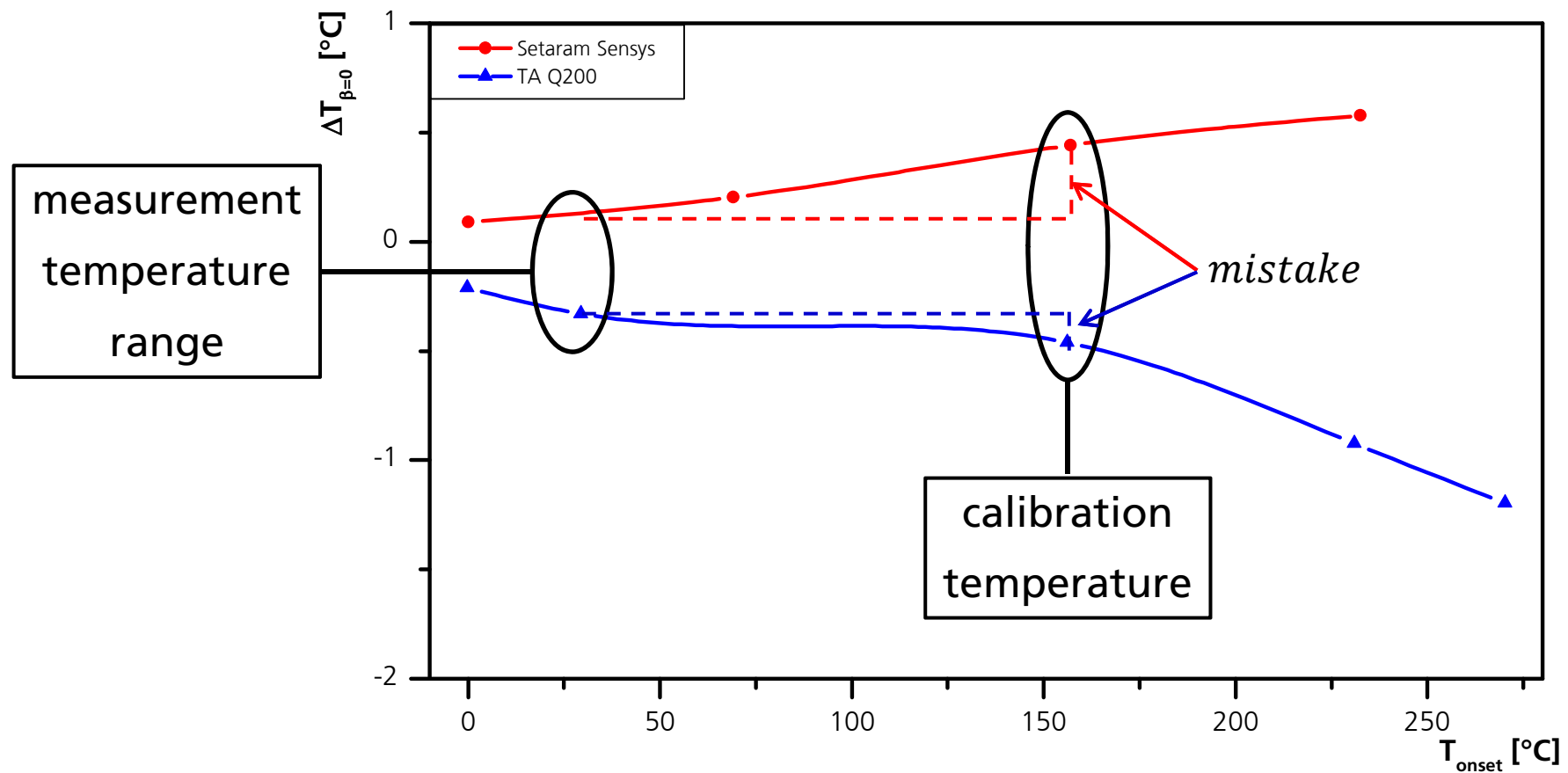
- at different temperature range
 - e.g. room temperature
- at different heating rates
 - e.g. 1 K/min

mistake: ignoring temperature and heating rate dependencies

$$T_{Onset} - T_{Lit} = \Delta T(T, \beta) \approx \Delta T(T)_{\beta=0} + \Delta T(\beta)$$

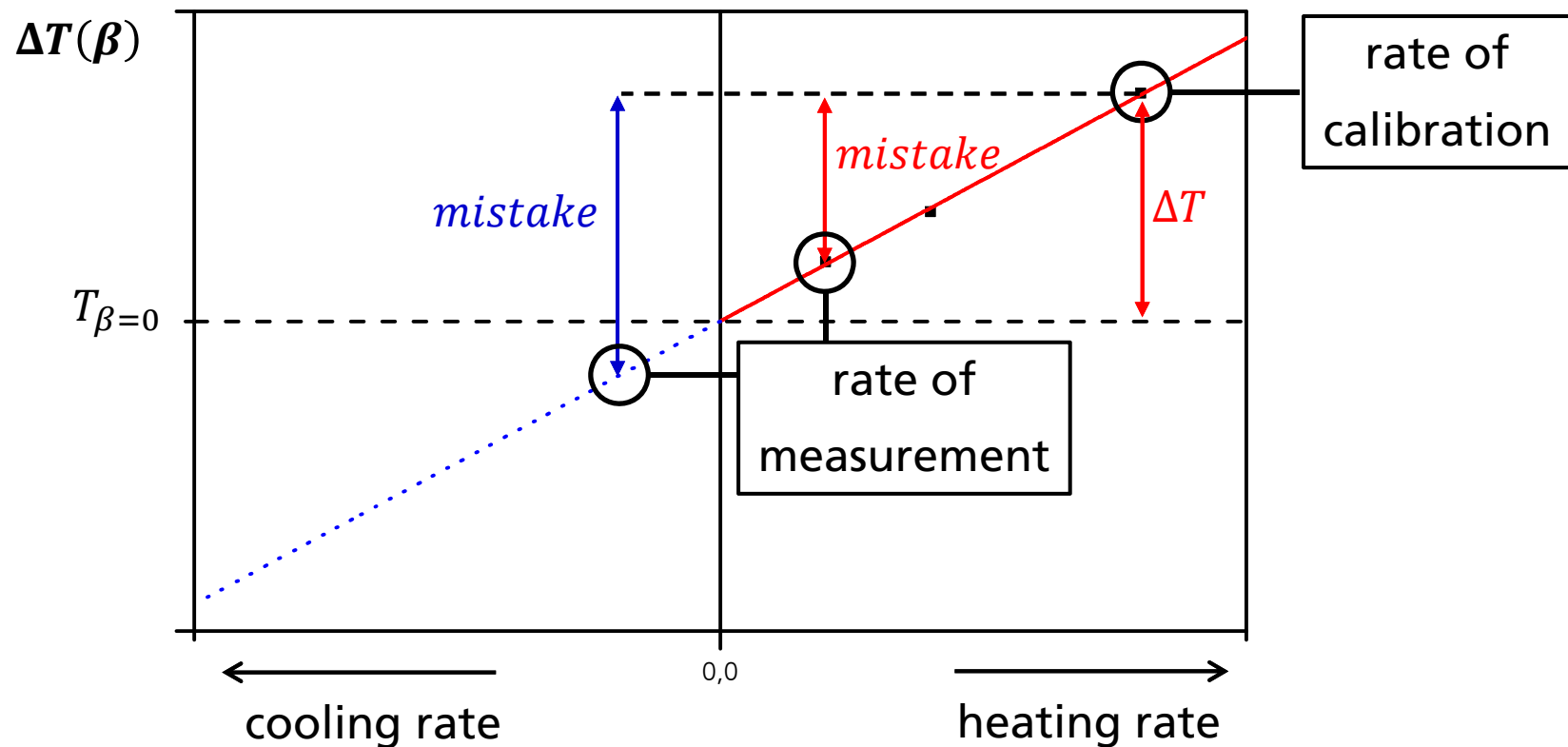
Mistakes in Common Temperature Calibration

Ignoring Temperature Dependency, $\Delta T(T)$



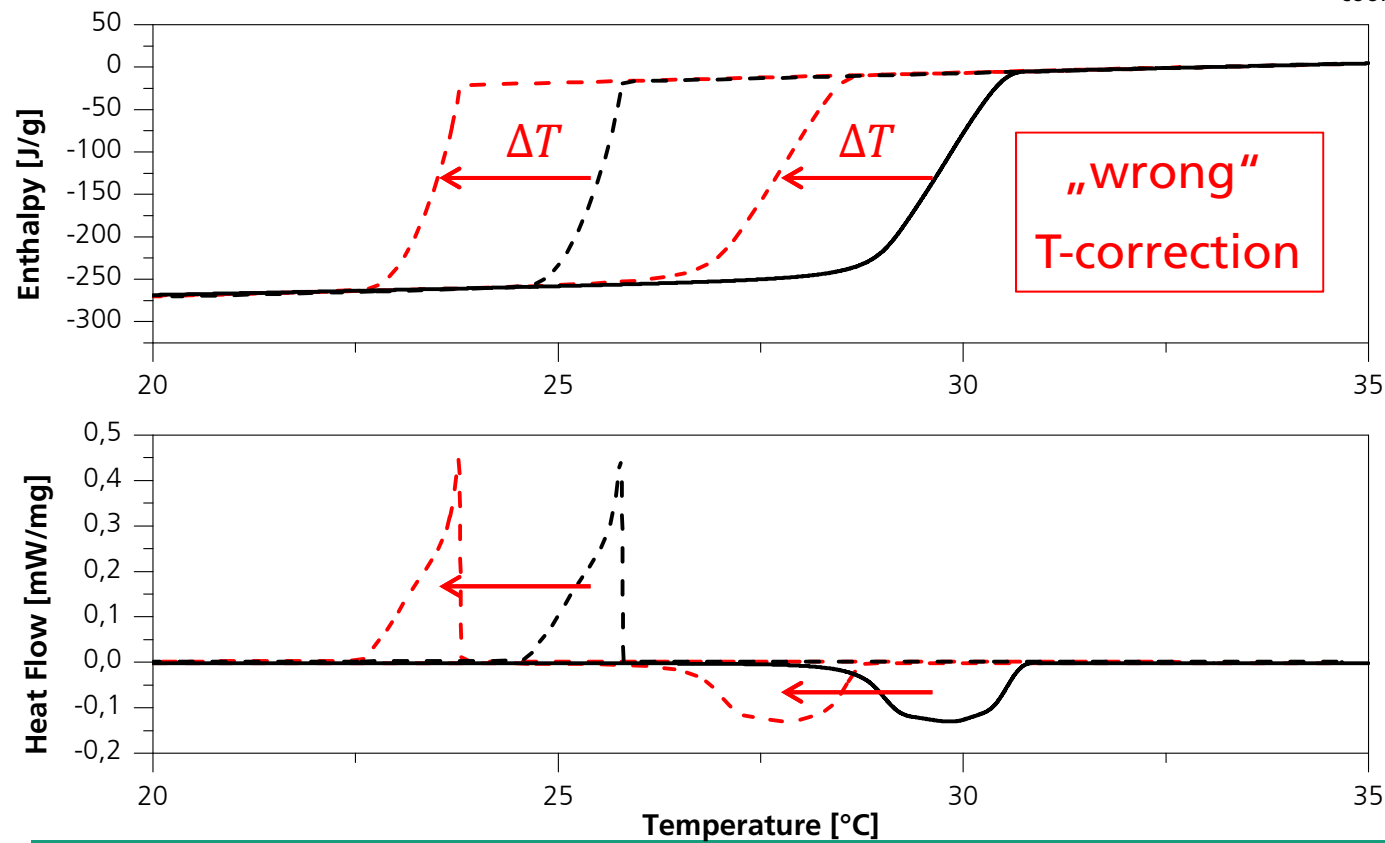
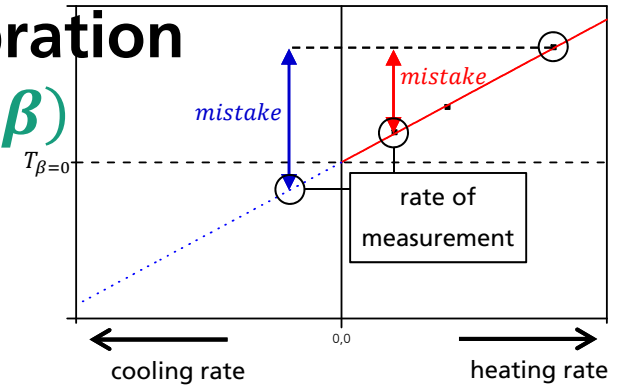
Mistakes in Common Temperature Calibration

Ignoring Heating Rate Dependency, $\Delta T(\beta)$



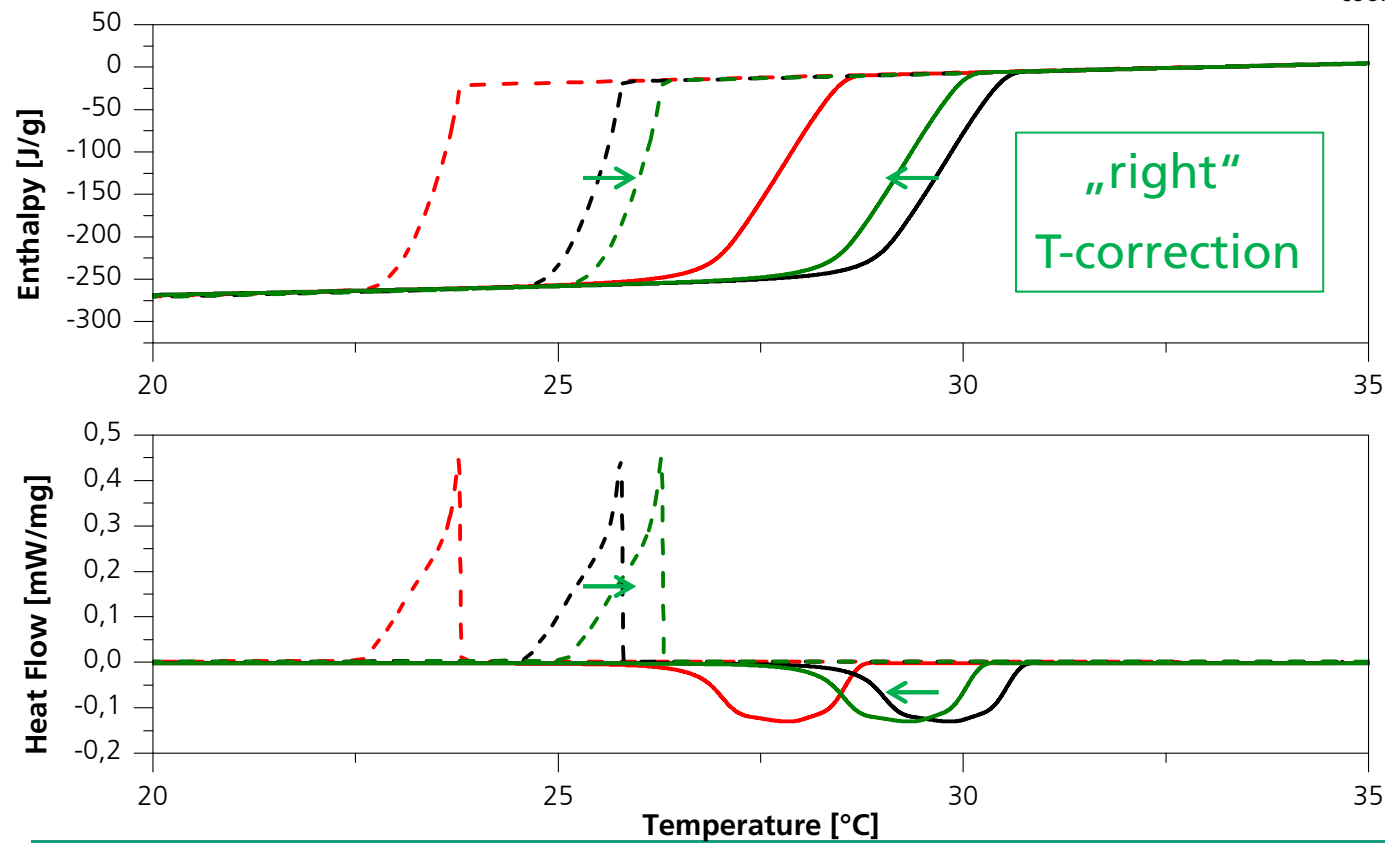
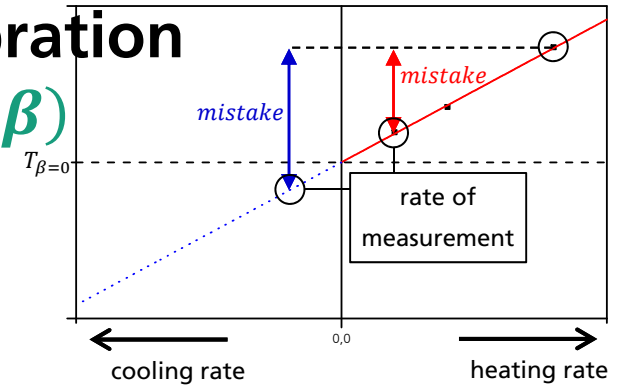
Mistakes in Common Temperature Calibration

Ignoring Heating Rate Dependency, $\Delta T(\beta)$



Mistakes in Common Temperature Calibration

Ignoring Heating Rate Dependency, $\Delta T(\beta)$



Conclusion

- calibration substance
 - 3, covering temperature range
 - inert against crucible material
 - 2 samples for each substance
- preparation
 - typical sample mass
 - one piece in crucible
 - 3 heating rates
 - „premelting“ cycle
- analysis of heat flow
 - onset-temperature
- comparison with literature values
 - dependence of temperature
 - dependence of heating rate
- separate correction
 - temperature range
 - heating/cooling rate

Thank you for your attention!



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