Chip DSC- Miniaturized Calorimeter with integrated heater, temperature sensors and crucible

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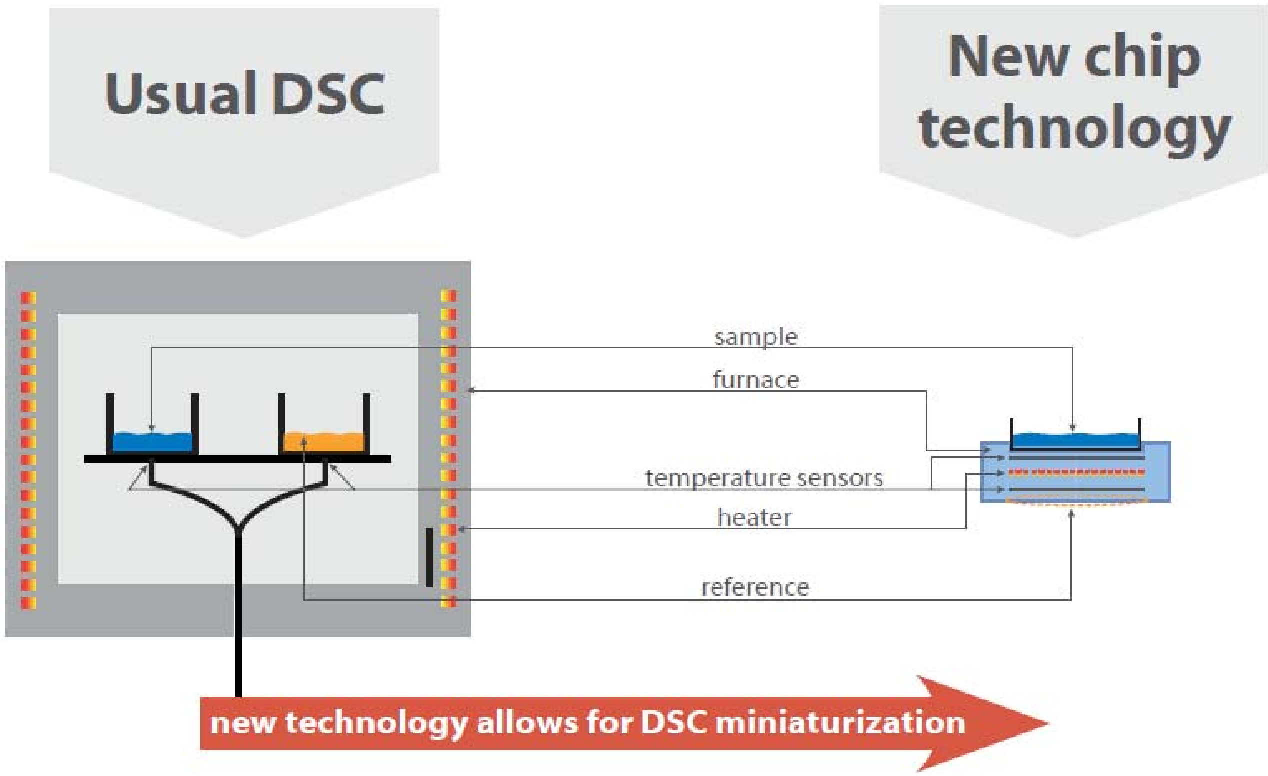
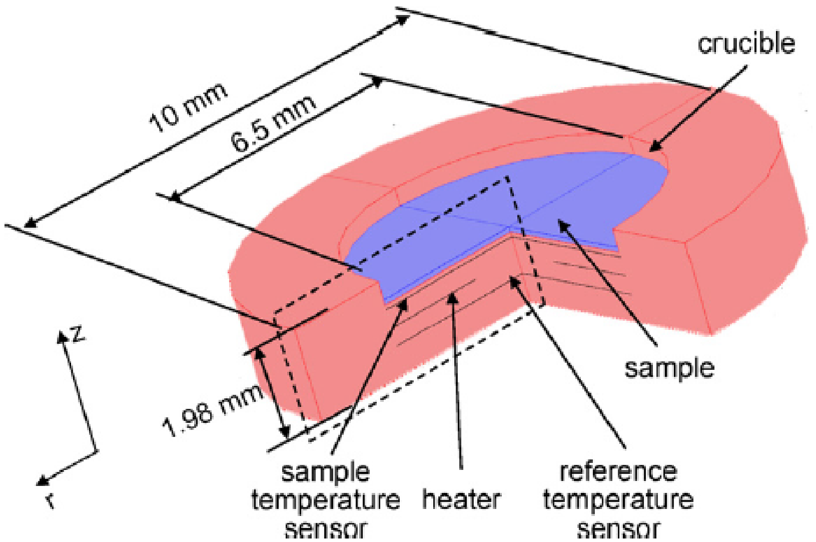
Basic Concept



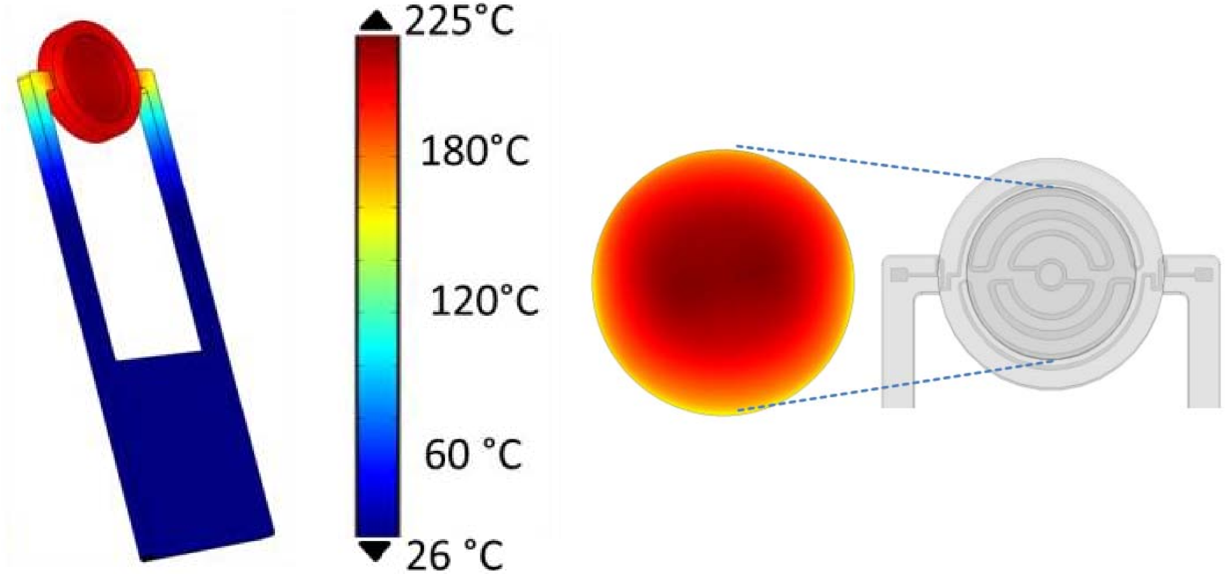
Chip DSC Sensor



Includes all components of a classical Heat Flux DSC:

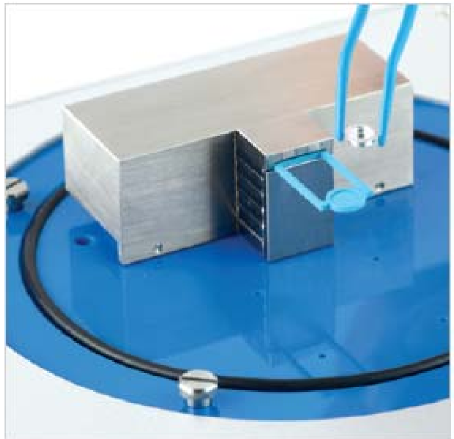
- Heater
- Sample and Reference temperature sensor
- Crucible

Optimized Chip Design



Uniform Temperature Distribution inside the Chip Crucible

Chip DSC Instrument



- Small Instrument, low costs
- Easy and cost effective exchange of the chip sensor -> ideal for critical samples (e.g. explosives, corrosive samples)
- Very Low Energy Consumption -> Control with Smart Phone/Laptop via USB port possible

Chip DSC Instrument

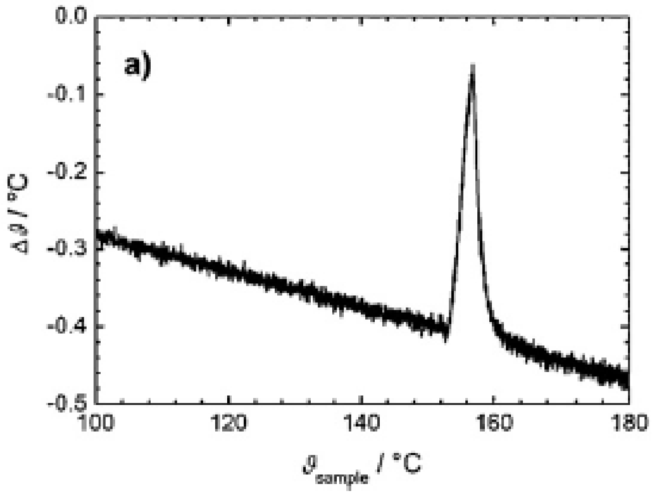
Available also with:

- Autosampler
- High Pressure Cell (e.g. OIT, Sorption)
- UV lamp (curing reactions)

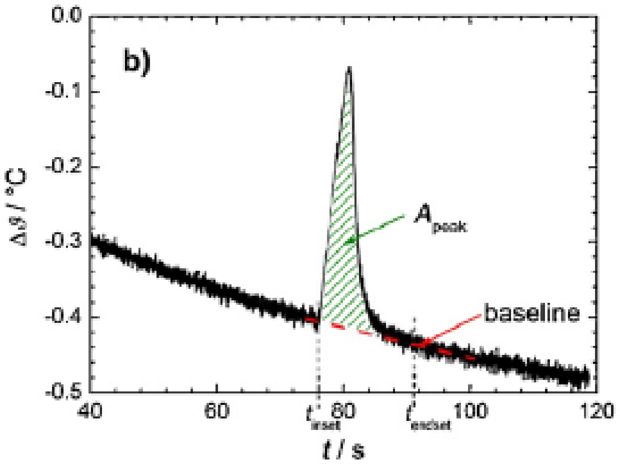
Performance Tests

Chip DSC

Repeatability of Melting Peak Area

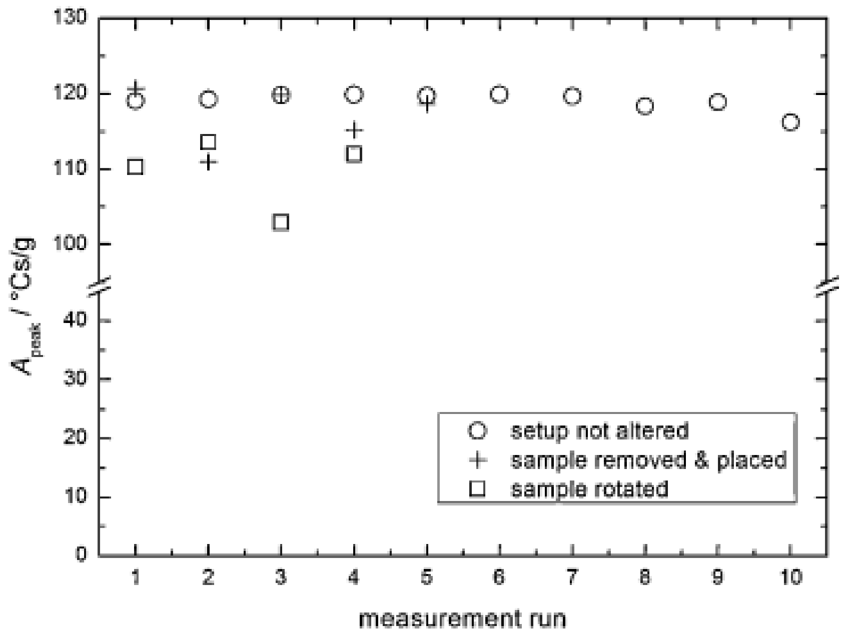


Indium Raw Signal



Calculation of Melt Peak Area

Repeatability of Melting Peak Area



10.993 mg Indium in Aluminum pan

$$R_{A,\text{peak}} = \pm\tilde{\delta}_{A,\text{peak}} = \frac{\tilde{\sigma}_{A,\text{peak}}}{\bar{A}_{\text{peak}}} \times 100\%$$

$R_{A,\text{peak,nc}} = \pm 0.3\%$ (no change)

$R_{A,\text{peak,sc}} = \pm 1.5\%$ (sample change)

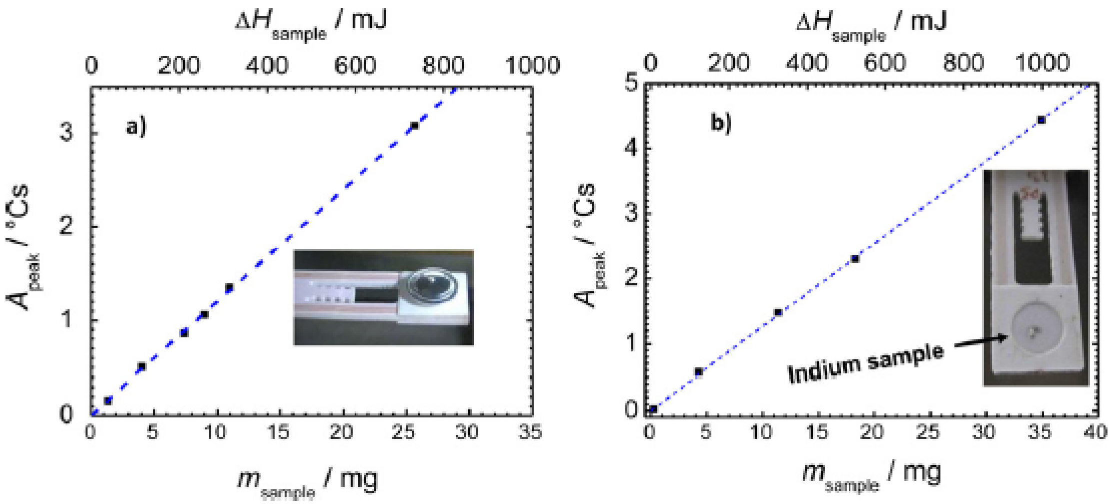
$R_{A,\text{peak,rt}} = \pm 2.2\%$ (sample position change)

-> Very good reproducability

Sample Mass - Linearity

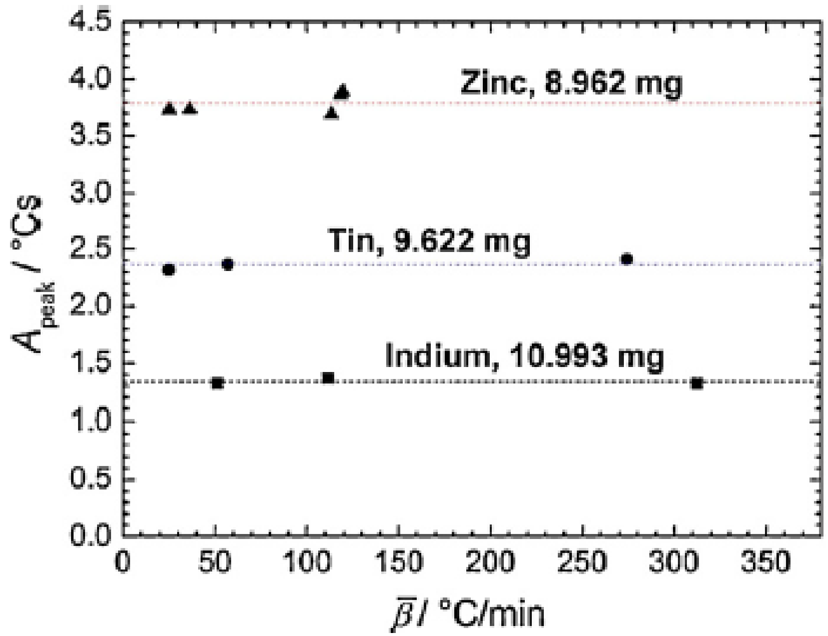
- Heat Transfer from the heater to the sample and reference ONLY through thermal conductivity
- Thermal conductivity of the chip material is balanced (glass ceramic material)-> nearly independent on temperature

-> Linearity regarding the sample mass



Melting Peak Areas vs. Sample mass

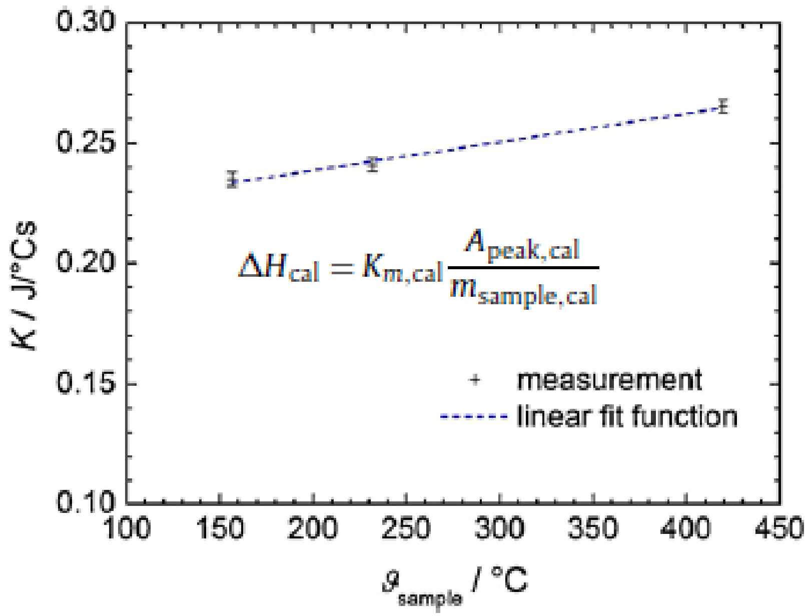
Heating Rate



Melting Peak Areas vs. Heating Rate

Melting Peak Areas independent on Heating Rate (up to 320 K/min)

Calorimetric Calibration



Enthalpy Conversion Factor vs. Temperature

- Indium, Tin and Zinc measurements
- Linear behavior

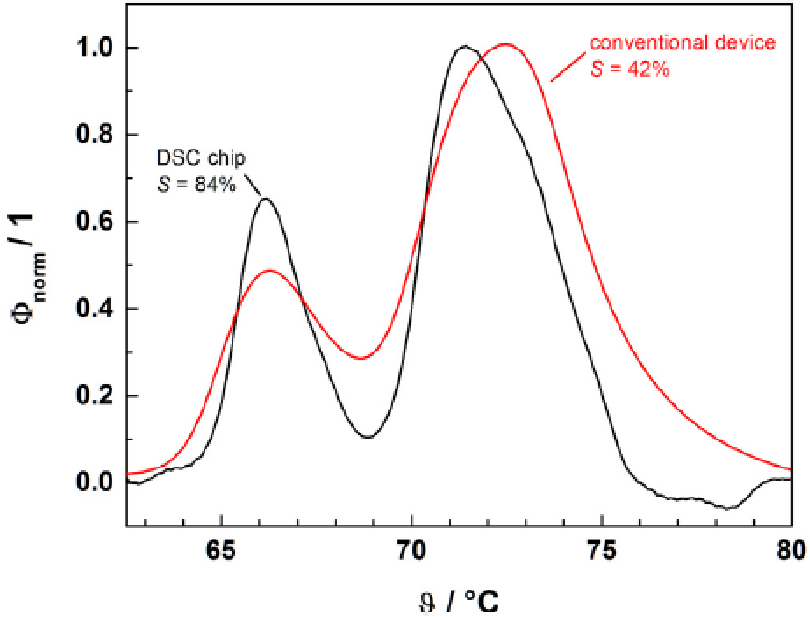
$$K(\vartheta_{sample}) = 0.215\,\mathrm{J/s\,°C} + 1.2 \times 10^{-4}\,\mathrm{J/s\,°C^2}\,\vartheta_{sample}$$

$$\Phi(\vartheta_{sample}) = K(\vartheta_{sample})\Delta\vartheta(\vartheta_{sample})$$

-> Time saving 2 point Calibration enough

-> valid for all heating rates

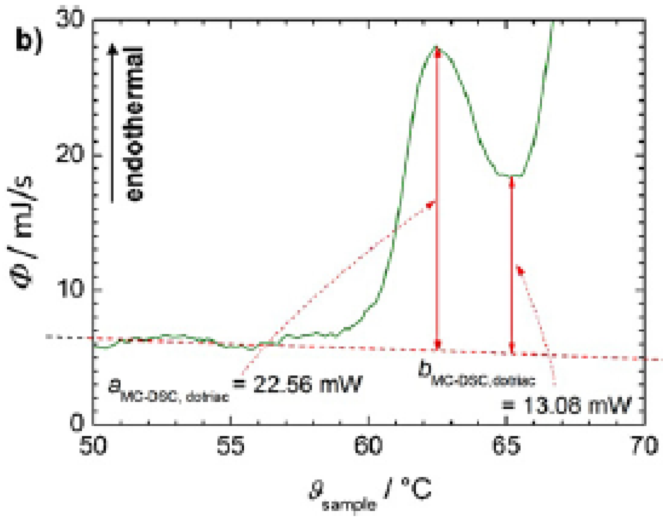
Resolution



1,5 mg Dotriacontane ($C_{32}H_{66}$) – Paraffin in Aluminium Crucible

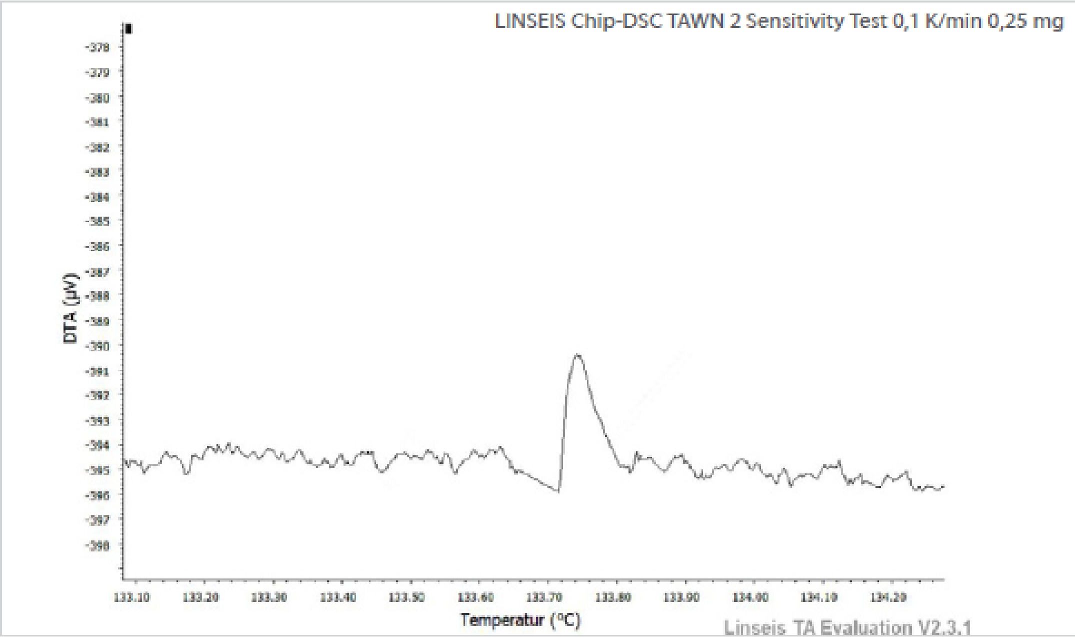
Calculation of the Resolution Factor:

$$S = \left(1 - \frac{b}{a}\right)$$



-> Better Resolution compared to conventional DSC instruments

Sensitivity



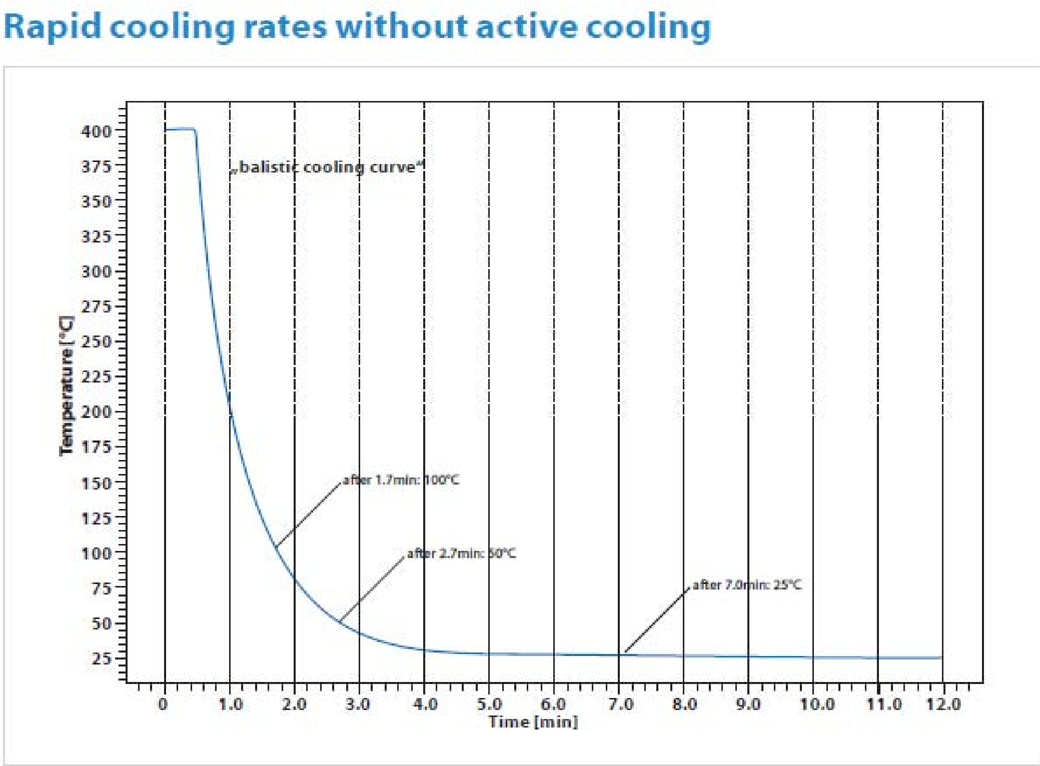
4,4´- Azoxyanisole – TAWN Test

Sensitivity Factor (Peak/Noise Ratio):

5.034

- Compareable to high end conventional DSC instruments
- Measurements at high heating rates up to 300 K/min can improve the sensitivity significantly

Cooling


Rapid cooling rates without active cooling
„balistic cooling curve“
after 1.7min: 100°C
after 2.7min: 50°C
after 7.0min: 25°C
Temperature [°C]
400
375
350
325
300
275
250
225
200
175
150
125
100
75
50
25
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
Time [min]

Specifications

	Chip-DSC 10	Chip-DSC 100
Temperature range	–150°C (with appropriate cooling option) up to +600°C (Peltier cooling system, LN_2 cooling system)	–180°C up to +600°C (Peltier cooling system, Closed-loop Intracooler, LN_2 cooling system)
Heating and cooling rates	0,001 up to 200°C/min	0,001 up to 1000°C/min
Temperature accuracy	+/- 0.2K	+/- 0.2K
Temperature precision	+/- 0.02K	+/- 0.02K
Digital resolution	16.8 million points	16.8 million points
Resolution	0.03 µW	0.03 µW
Atmospheres	inert, oxidizing (static, dynamic)	inert, oxidizing (static, dynamic)

Conclusions

- Better Resolution than conventional DSC´s
- Same sensitivity than conventional DSC´s
- Caloric Measdurements independent on heating rates
- Caloric sensitivity depends linear on sample mass
- High heating and cooling rates
- Measurements under pressure and UV- light easy to realize
- Much lower costs
- Easy and cost effective exchange of the chip (ideal for critical samples)

Thank you for your attention!

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