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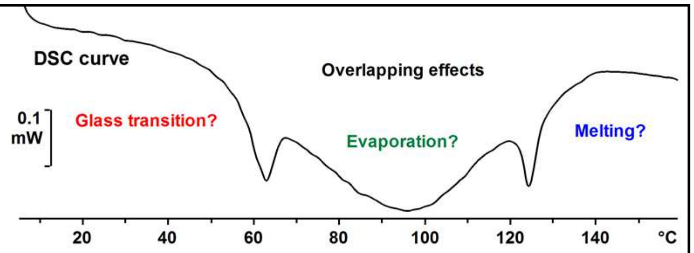

Strategies for Separating Overlapping effects by DSC and Flash DSC

METTLER TOLEDO

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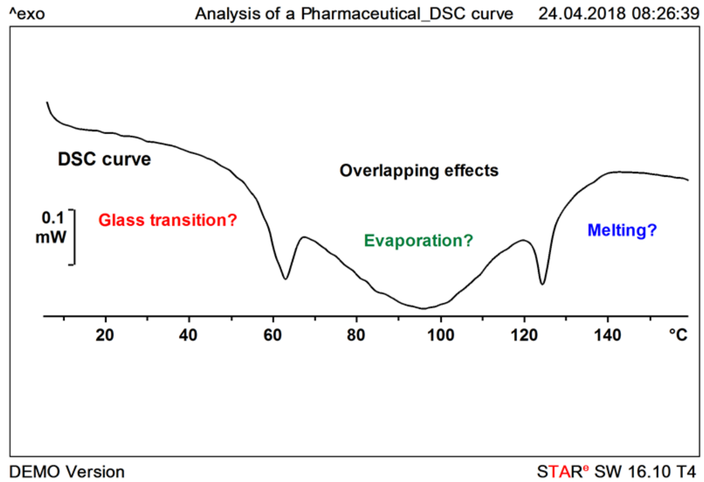
Agenda

- The interpretation and quantitative evaluation of thermal analysis measurement curves is difficult when several effects take place simultaneously. A number of methods are available that can be used to separate overlapping effects and analyze them individually afterwards. Here strategies for DSC curves are discussed



There are four main strategies that can be applied to separate overlapping effects:

- Changing the environmental conditions (using different gases or crucibles)
- Modulated techniques (TOPEM, ADSC, IsoStep)
- Combined techniques like DSC-microscopy
- **Variation of the temperature program . This includes the use of different heating and cooling rates as well as heating-cooling-heating cyles. Of special interest is the use of the Flash DSC with ultra fast scanning rates.**

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2 Flash DSC 2+
3 Applications
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Flash DSC 2+

The Flash DSC 2+ for analysis of reorganization processes.

- Sensor temperature range: –95 °C to 1000 °C
- Ultra-fast sensors
  - UFH 1 (high-temperature)
  - UFS 1 (standard temperatures)
- Cooling rates: **6 to 2'400'000 K/min**
- Heating rates: **6 to 3'000'000 K/min**
- Terminal with touch control
- Signal time constant (N_2):
  - Approximately **0.2 ms (UFH 1)**
  - Approximately 1 ms (UFS 1)

Flash DSC 2+: Features and Benefits

Features	Benefits
▪ **Ultra-high heating rates**	Suppress reorganization processes
▪ **Ultra-high cooling rates**	Allow the formation of materials with defined structural properties
▪ **High sensitivity**	Permits measurements at low heating rates that overlap with conventional DSC
▪ **Wide temperature range**	Perform measurements from **–95 to 1000 °C**
▪ **Simplicity of use**	From easy sample preparation and sensor change, to convenient result evaluation
▪ **Oxygen-free conditions**	**Protect your sample against oxidation**
▪ **Fast response sensor**	Enables the kinetics of extremely fast reactions or crystallization processes to be studied

Flash DSC 2+: MEMS Sensor Technology

Superior MEMS sensor technology

MultiSTAR UFH 1 / UFS 1 Sensors

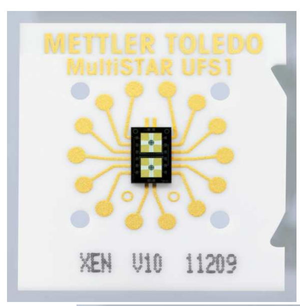
- Number of thermocouples: 4 (UFH 1); 16 (UFS 1)
- Excellent temperature resolution
- High sensitivity

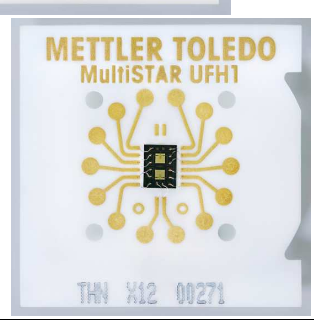
Temperature resolution

- Time constant of about 0.2 ms (UFH 1); 1 ms (UFS 1)
- Time constant of Flash DSC 1000x less than conventional DSC
- Perfect separation of close-lying effects

Handling of chip sensors

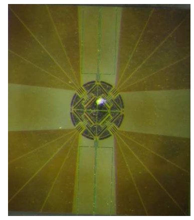
- Samples are positioned directly on the sensor
- Used sensors with samples can be stored for further measurements later on
- Change of chip sensor takes < 1 min



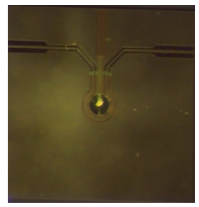


Flash DSC 2+: MEMS Sensor Technology

- Smaller sample area
- Thinner membrane
- Gold instead of aluminum
- Maximum temperature approx. 1000 °C
- Increased scanning rates

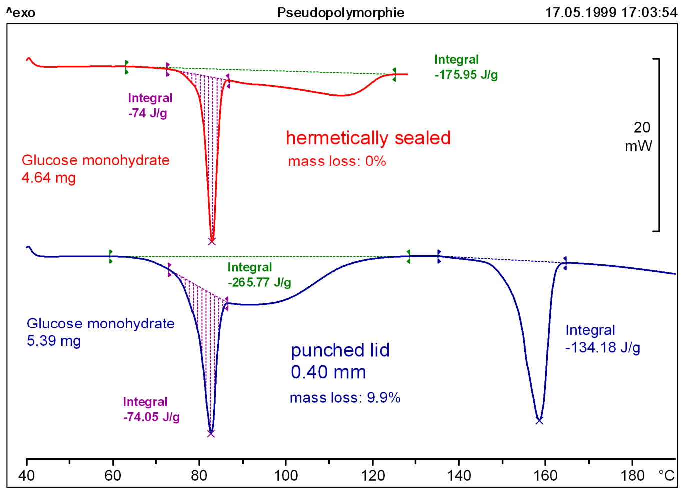


UFS1



UFH1

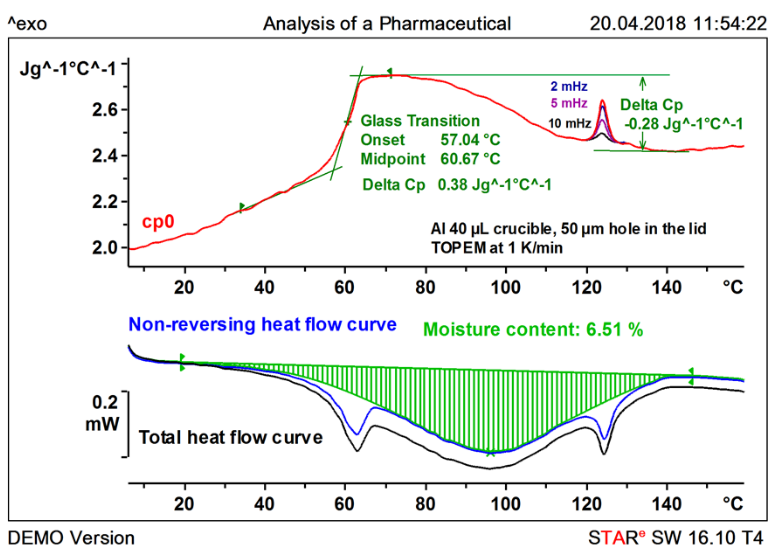
Agenda



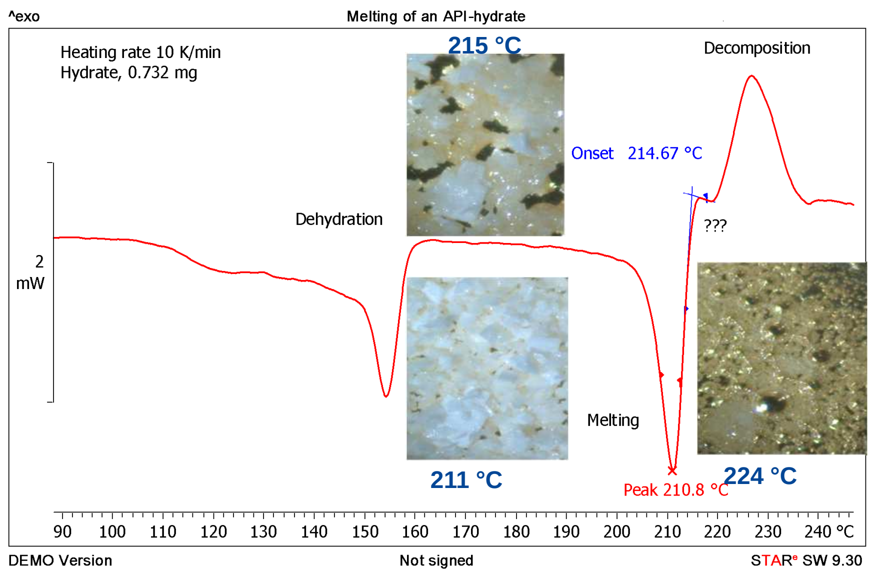
Glucose:
inactive ingredient,
filler for tablets and capsules

Pseudo-polymorphism: Incorporation of solvent molecules into the crystalline structure

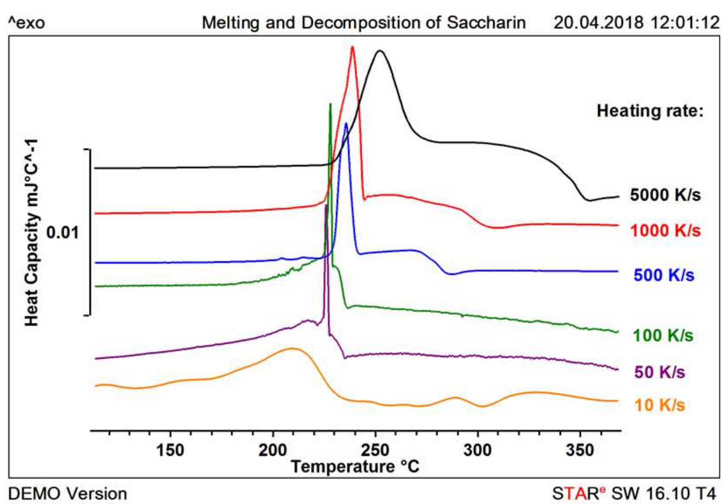
- Different physical properties
- Different stoichiometry



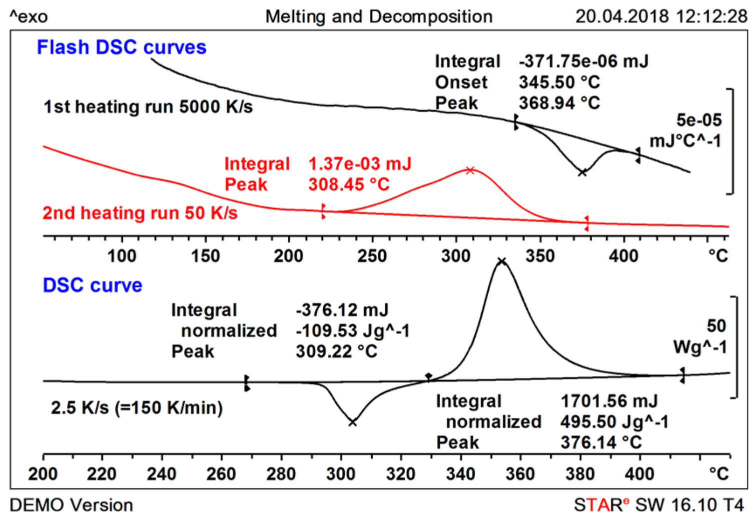
Application 3: Combined technique



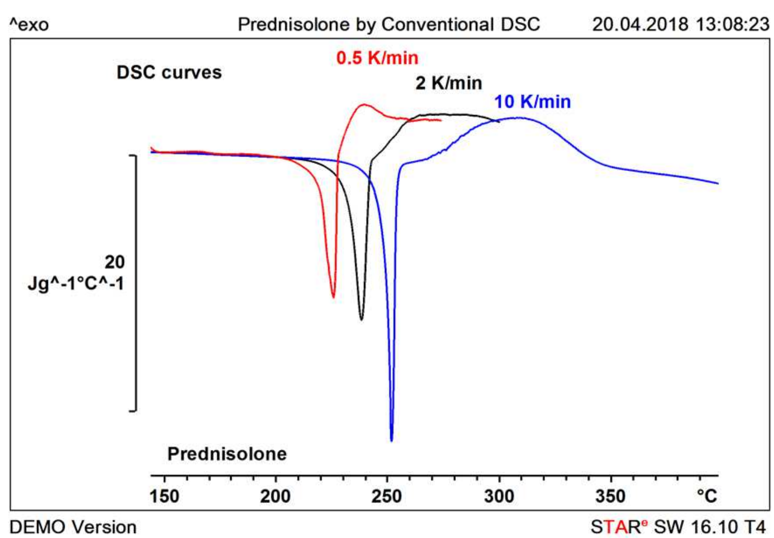
Application 4: Variation of the heating rate



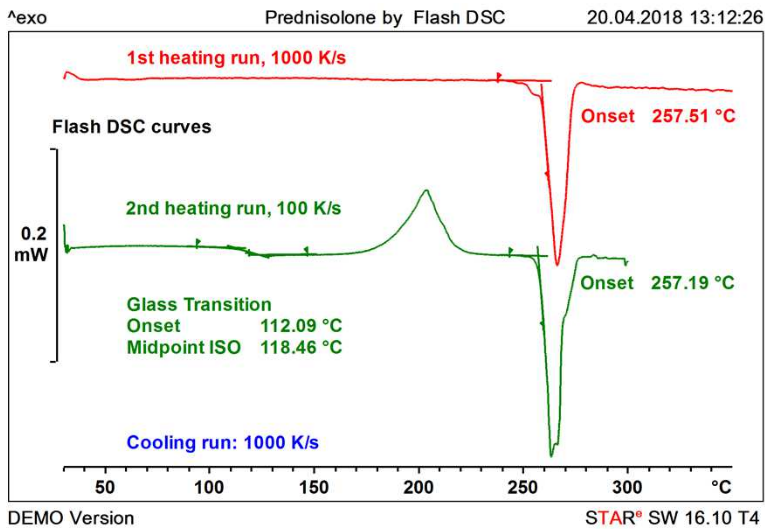
Application 5: Variation of the heating rate, pigment



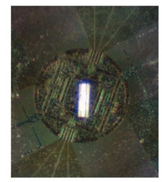
Application 6: Variation of the heating rate

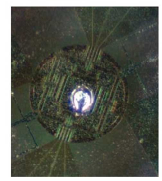


Application 6: Variation of the heating rate



Flash DSC sensor





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4 strategies for separating overlapping effects by DSC

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Thank you