

DSC-Microscopy System

DSC Measurements with Visual Observation

DSC curves often exhibit effects that cannot immediately be understood. In such cases, it would be very helpful to actually see what is going on in the sample. This would enable us to identify solid-solid transitions, differentiate between overlapping effects such as melting and decomposition, observe the shrinkage of fibers or films, or simply identify the cause of an artifact in a DSC curve.

To obtain this visual information, we have developed a versatile optical accessory that can be used with any METTLER TOLEDO DSC. It consists of a dedicated optical system, a CCD camera and image capture and processing software that is synchronized with the DSC temperature program. The system provides images of samples at predefined temperature or time intervals. A further evaluation possibility is to quantify color differences by calculating an average brightness for each of the images in a selected temperature or time range. The brightness can then be displayed as a function of temperature or time and be directly compared with the DSC curve.

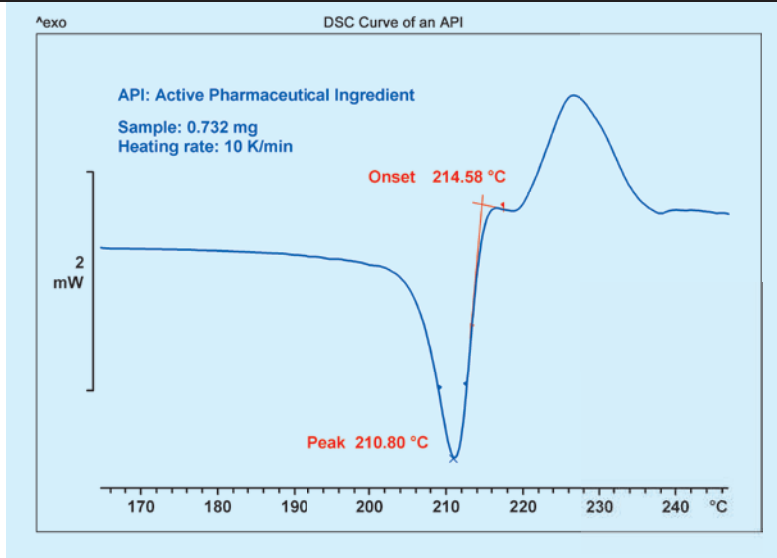


Features and benefits

- **Imaging possibilities** – microscopy yields information on processes that generate little or no enthalpy and cannot be detected by DSC
- **MultiSTAR® DSC sensor capability** – very weak effects can be measured with excellent sensitivity and temperature resolution
- **Simultaneous image analysis and DSC measurements** – provide a complete thermal picture of the sample
- **Possibility to upgrade from a conventional DSC to a DSC-Microscopy System within minutes** – a DSC 1 or DSC82x^e can be easily upgraded to include reflected-light microscopy measurement capability

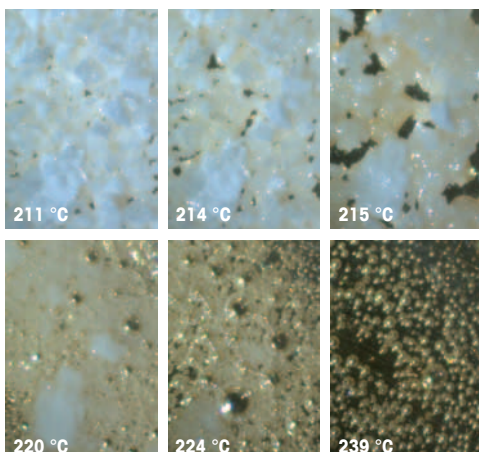
Industry	Applications
Paints, lacquers, adhesives, coatings	<ul style="list-style-type: none"> • Flowing (e.g. of powder coatings)
Packaging, films	<ul style="list-style-type: none"> • Visual observation of shrinkage
Chemicals (organic and inorganic materials), pharmaceutical products	<ul style="list-style-type: none"> • Observation of crystallization from solutions • Visualization of thermochromism • Evaporation and sublimation • Safety investigations
Food	<ul style="list-style-type: none"> • Oxidative stability of fats and oils • Reactions with reactive gases

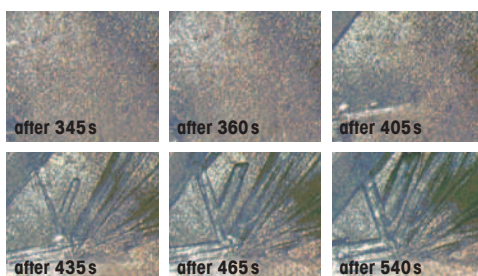
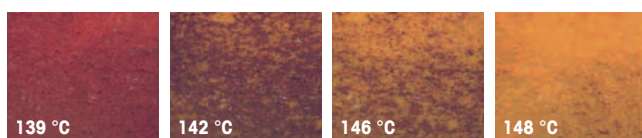
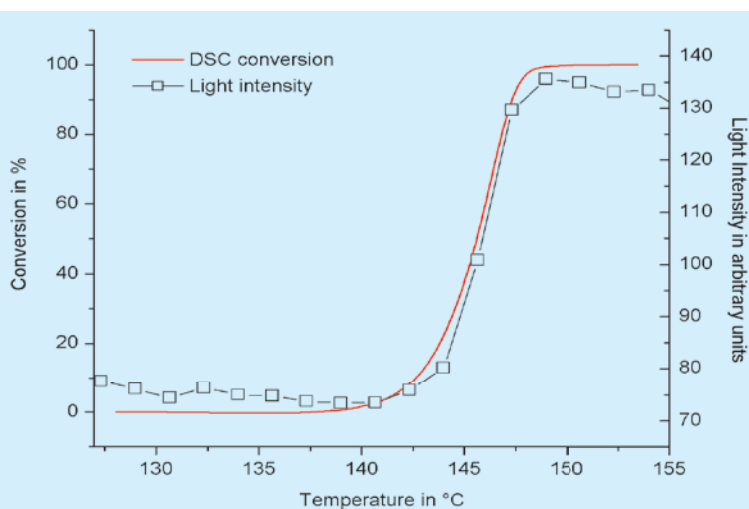
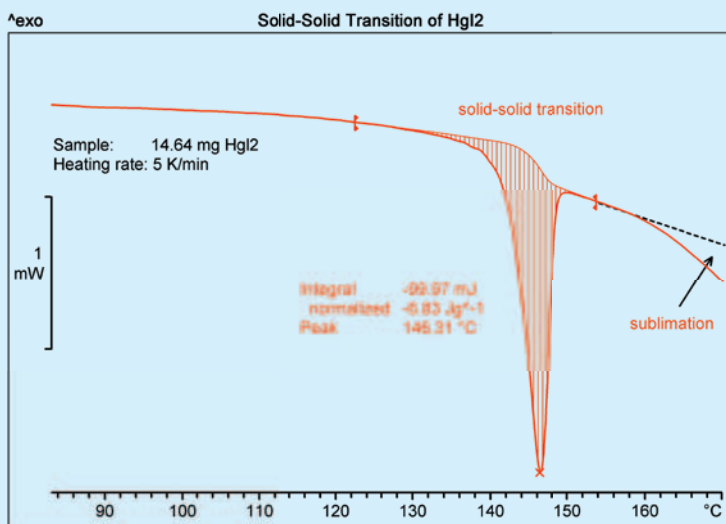
Application Examples



DSC-microscopy analysis of an active pharmaceutical ingredient

The melting point is an important characteristic property of a substance. The DSC measurement of an active pharmaceutical substance (API) yielded a curve with two main peaks – an endothermic peak with a maximum at 210.8 °C and an exothermic peak at about 228 °C. A smaller peak was also apparent between these two peaks at about 214 °C. The initial interpretation was that the endothermic peak at 210.8 °C is due to melting. The DSC-microscopy results, however, told a different story. No melting was observed at 211 °C – the first signs were detected at about 214 °C. Clearly, the endothermic DSC peak is not caused by a melting process. The color change of the molten substance leads one to conclude that it decomposes immediately on melting. The small DSC peak observed at about 214 °C is therefore the sum of two simultaneously occurring effects – endothermic melting and exothermic decomposition. Separate TGA-MS measurements showed that the endothermic DSC peak at 210.8 °C is caused by the evaporation of water of crystallization.





Thermochromism of HgI₂

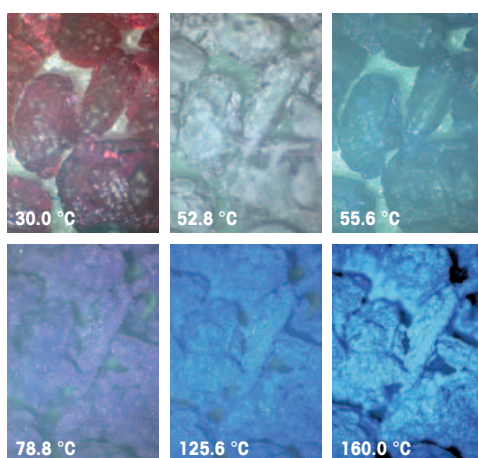
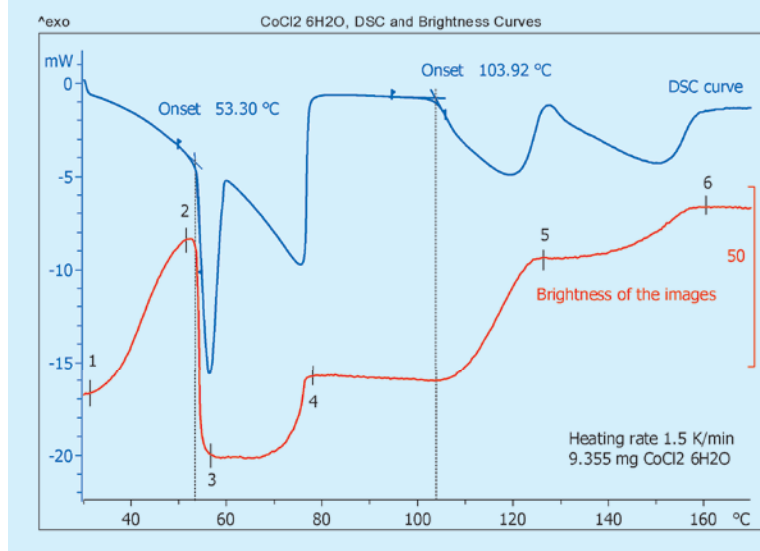
When HgI₂ is heated it undergoes an endothermic, solid-solid transition at about 140 °C. This polymorphic transition is accompanied by a change in color from red to yellow. DSC is an excellent technique to accurately measure the transition enthalpy of the sample but it cannot detect changes in color. When DSC is combined with microscopy, the change in color of the sample can be directly observed.

The images of a powdered HgI₂ sample show that the transition of each grain occurs separately during heating. This explains the relatively broad DSC peak. The bright spots which appear on the images are due to light directly reflected from individual grains of the sample.

Quantitative analysis of the increase in light intensity with temperature yields a curve that agrees well with the DSC conversion curve. This demonstrates that DSC combined with microscopy is an important technique for quantitative analysis.

Crystallization from an aqueous copper sulfate solution

This visual information helps you to interpret overlapping processes. During crystallization from solution, the DSC peak due to solvent evaporation may partially, or even completely, dominate the measurement of the crystallization process. This depends on the enthalpy of evaporation of the solvent and the temperature at which the crystallization process occurs. In such cases, the DSC curve would just show a broad endothermic peak due to the evaporation of the solvent. The combination of DSC and microscopy, however, allows the crystallization process to be observed and recorded visually.



Dehydration of CoCl₂ hexahydrate

The images illustrate the dehydration process of cobalt (II) chloride hexahydrate when it is heated from 30 °C at 1.5 K/min. The initial ruby-red color becomes lighter and lighter until it suddenly changes at about 55 °C. Further color changes are observed between 100 °C and 120 °C and at 160 °C. The differences can be quantified by calculating a curve showing image brightness as a function of temperature. The DSC curve shows a broad endothermic peak up to 80 °C with a sharp peak at about 55 °C superimposed on it. Two further endothermic peaks follow at about 104 and 130 °C. The brightness first increases and then suddenly decreases at about 55 °C. Afterward, it increases again in several steps.

The peak at about 55 °C is due to a change in crystalline structure. The images around 55 °C show small droplets of water on the surface of the crystals. This indicates that part of the water of crystallization eliminated from the crystal lattice during the solid-solid transition collects on the surface of the crystals and then evaporates. This is completed by about 80 °C. The two broad endothermic peaks on the DSC curve and the step-like changes in the brightness are due to further loss of water of crystallization.

Image capture

The images shown in these examples were captured using analySIS[®] software from Olympus Soft Imaging Solutions GmbH. Packages including analySIS[®] software, zoom optics and camera are available from METTLER TOLEDO.



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