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In-Situ DSC-Humidity PXRD Analysis for Pharmaceuticals

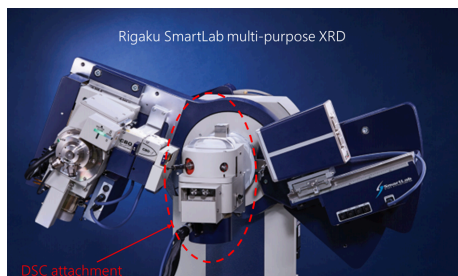
Simultaneous XRD-DSC-Humidity Analysis for Pharmaceutical Samples

For organic materials in general and active pharmaceuticals in particular, a combination of orthogonal analytical methods is often the optimal choice for meaningful characterization. While X-ray powder diffraction is the gold standard for structural characterization of polymorphs, thermal analysis provides critical insights into the thermodynamic relationships between the different solid forms.

In the XRD-DSC-Humidity system, Rigaku has combined simultaneous Dynamic Scanning Calorimetry (DSC) with reflection powder X-ray diffraction (XRD) for real-time analysis of solid form and thermodynamics. The in-situ DSC unit is able to scan temperature and/or humidity, providing a wealth of dynamic phase information.

The DSC-Humidity unit can be mounted on either the SmartLab XE or SmartLab SE and supports 0D, 1D, or 2D data collection (depending on the choice of detector). The DSC system is fully integrated into both the SmartLab electronics and SmartLab studio II data collection software allowing automated measurement setup and execution. The collection of thermal and powder diffraction data are monitored and tracked by the SmartLab Studio II ER/ES (electronic records and signatures) plugin and is 21 CFR part 11 compliant.

DSC attachment mounted on a Rigaku SmartLab XE



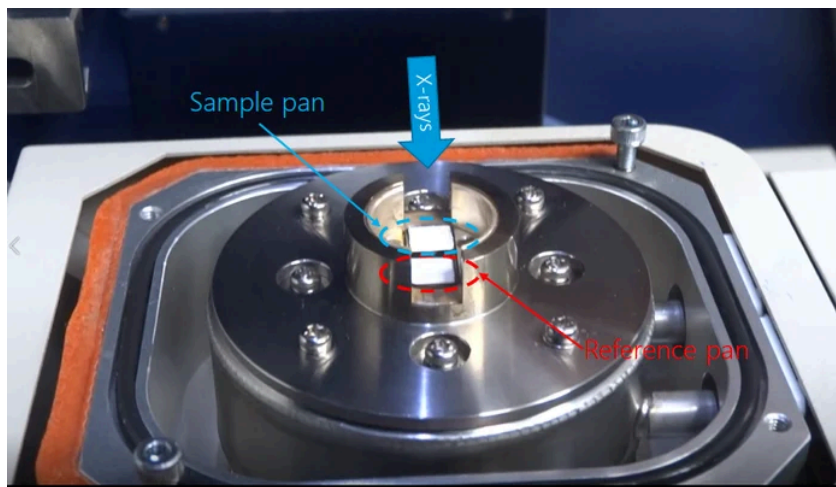
DSC attachment mounted on a Rigaku SmartLab XE

The DSC-Humidity Attachment

The DSC-Humidity unit has twin pans (reference and sample) with the sample heat capacity determined utilizing the heat flux method. The rectangular aluminum pans are flat and open and about 7 mm along each edge. Each pan has a 'U' shaped profile allowing the preparation of thin samples (with ~0.2 mm maximum thickness) while providing good thermal conductivity. Up to about 4 mg of organic powdered sample can be held in a full flat and leveled pan. The open pans are contained within a controlled environment which can be dry air or dry N₂ (max flow rate 200 mL/min). A controlled humid air environment is also possible with a humidity range between 5 to 95% RH with an appropriate generator.

For dynamic temperature measurements, the sample pans can be heated from ambient to 350 °C. In addition, active cooling down to -40 °C is also possible with the appropriate cooling option.

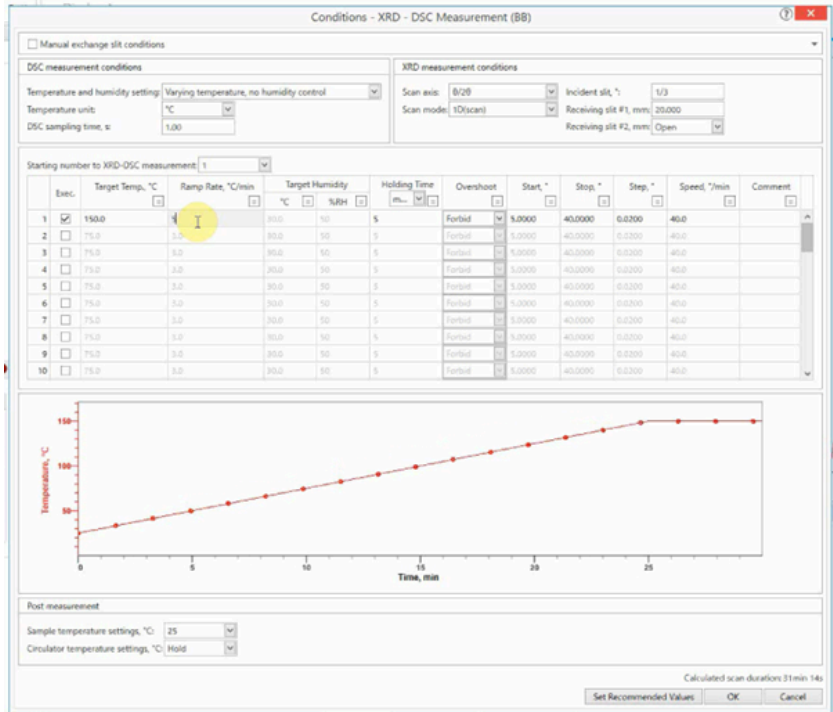
The in-situ DSC-Humidity stage has been optimized for collecting X-ray powder diffraction data on organic samples and supports a measurement range from 0.1 to 50.0 °2 θ .



View inside the DSC attachment showing sample and reference pans.

Control and Measurement Software

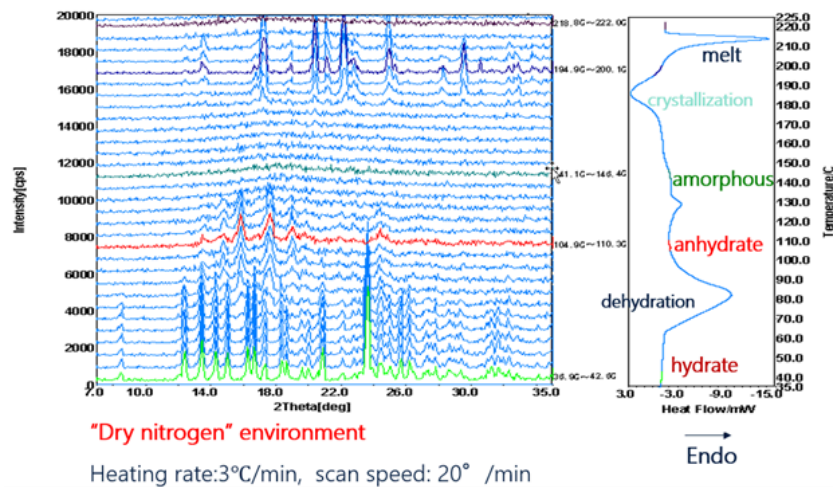
The control and monitoring of temperature and humidity for the DSC attachment are embedded within the SmartLab Studio II data collection and measurement plugin. This allows a transparent operation of the DSC unit and seamless synchronization of temperature and humidity with simultaneous powder diffraction measurements.



DSC and XRD measurement programs inside SmartLab Studio II software.

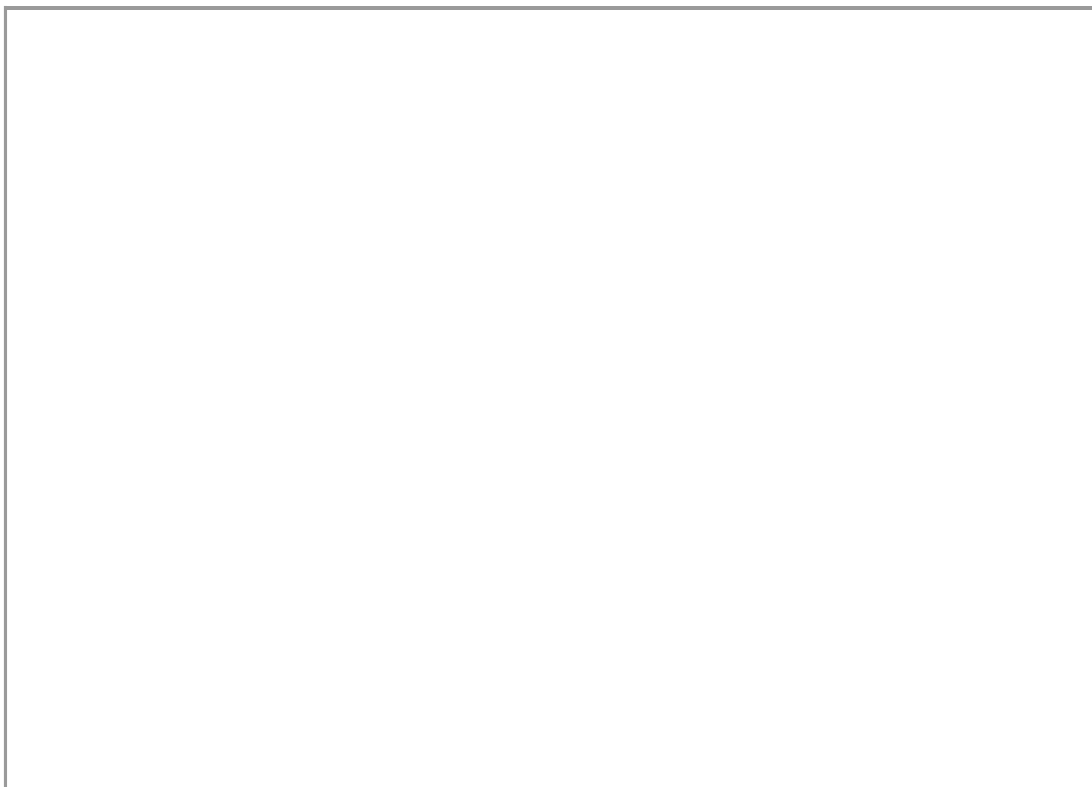
Data Presentation and Analysis Software

Thermal and diffraction data can be interactively viewed and processed together as a single data ensemble or individually for a more in-depth analysis. The ensemble viewing and visual analysis allow for a broad range of presentation styles emphasizing change of solid form with the associated thermodynamic events.



Visual analysis and presentation of DSC and XRD data ensemble

The chosen visual representation of the diffraction and thermal data can be replayed as an interactive 'movie' to follow the induced solid form changes as a function of time or thermodynamic variable (temperature/humidity).



Interactive analysis and replay of simultaneous DSC / XRD measurements

A full range of powder diffraction and thermal analysis methodologies are supported for individual XRD and DSC data sets.

Related products



SmartLab

Advanced state-of-the-art high-resolution XRD system powered by Guidance expert system software



SmartLab SE

Highly versatile multipurpose X-ray diffractometer with built-in intelligent guidance



SmartLab Studio II

Windows-based software suite for Rigaku's X-ray diffractometers