

# Pharmaceutical Solid-State Characterization by Simultaneous Small- and Wide-Angle X-Ray Scattering

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The structure of pharmaceutical solids at the nanoscale i.e. between 1 and 100 nm, is a critical factor for many technological properties, such as stability, reactivity, solubility, water- and chemisorption, active compound release and bioavailability. While classical XRPD is mainly restricted to the molecular structure within crystalline domains, small-angle X-ray scattering (SAXS) extends the scope to the characterization of the nano-domains. A combination of the two techniques within one instrument, therefore, is a powerful tool to characterize pharmaceutical solids.

The specific inner surface is the key determinant for the stability or reactivity of solid-state materials. SAXS allows to evaluate rapidly precisely this important material property. Combining this with simultaneous wide-angle powder diffraction opens a complete view on the nanostructure of solids.

An instrument is described, that allows this combination in the transmission-optical mode and is designed for investigations of microcrystalline or microparticle materials. An essential component is the SpinCap system which allows to rotate the sample during measurement under close thermal control. This avoids texture artifacts, ensures high-resolution quasi-powder patterns, and allows in-situ, time-resolved studies on reacting or sedimenting systems, e.g. liquid suspensions. It also facilitates time-resolved studies on the crystallization of amorphous solids.

Application examples will be described for pharmaceutically important systems, such as lactose-based inhaler powders, mesoporous silica, amorphous sucrose, and lyotropic LC-phases.