

Non-ambient Conditions X-ray Powder Diffraction in Pharmaceutical Industry

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One of the main applications of X-ray Powder Diffraction (XRPD) in pharmaceutical industry is the fingerprinting (qualitative analysis) of solid forms that have been crystallized prior analysis under various experimental conditions. In contrast to this, phase transitions are directly monitored during analysis using non-ambient conditions XRPD such as temperature controlled XRPD (TC-XRPD) and humidity controlled XRPD (HC-XRPD).

TC-XRPD is widely used for analyzing (pseudo)polymorphic phase transitions related to temperature changes. With high-resolution XRPD instrumentation fingerprints of high-temperature forms are obtained and potentially new forms are identified. The combination of TC-XRPD experimentation and Differential Scanning Calorimetry (DSC) gives fruitful information on the phase diagram of the substance under investigation.

Hydration/dehydration processes are analyzed by controlling the relative humidity of the sample environment during measurement. From HC-XRPD experimentations at various temperatures clues on the phase diagram are obtained. By correlating Dynamic Vapor Sorption (DVS) analysis and HC-XRPD weight changes are assigned to (pseudo)polymorphic phase transitions. HC-XRPD is applied both for analyzing the active pharmaceutical ingredient (API) and the excipients of the final drug product.