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Advances in Synchrotron XRPD for the characterization of pharmaceuticals

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X-Ray Powder Diffraction (XRPD) directly provides structural and microstructural characterization of materials. Considered the gold standard method in the field of pharmaceutical powders for the identification of solid forms (i.e. polymorphs, solvates, hydrates, salts, co-crystals, amorphous), when combined with a synchrotron X-ray beam, XRPD becomes a truly mighty analytical tool for the characterization of pharmaceuticals. Ultra-high FWHM and d-spacing resolutions, accurate 2theta angle assignment, high signal-to-background and signal-to-noise ratios distinguish synchrotron XRPD patterns from conventional XRPD, whereas the combination of the synchrotron properties with new outstanding single-photon-counting detection systems drastically reduces the measurements times to milliseconds allowing in-situ study of the kinetic of transformations and radiation-damage-free high-resolution diffraction patterns. Advances in instrumentation, calibration and data collection procedures leading to detection limits of contaminating crystalline phases better than 0.05% wt% as well as subtle structural details are described.

Keywords: Synchrotron X-Ray Powder Diffraction, Pharmaceuticals, Structural analyses