

PXRD and SSNMR spectroscopy: Complimentary techniques for polymorph identification

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Advanced analytical techniques can provide unique insight into the composition and properties of pharmaceutical formulations. Powder X-ray diffraction and solid-state NMR spectroscopy are two complementary techniques for the characterization of pharmaceuticals. In this presentation, the ability of each technique to identify crystalline forms and quantify polymorphs in both the drug itself as well as in formulation will be discussed. Aspirin is a classic example of where a unique peak was identified in the solid-state NMR that was different than the known aspirin form, but the powder X-ray diffraction pattern was not unique enough to confirm that this was a new form. Since that time, the new polymorph has been confirmed, although it took almost a decade to demonstrate that the new form and the form observed via solid-state NMR were the same. Neotame is another example of where the identification of form transitions could be observed using solid-state NMR, but could not be observed using powder X-ray diffraction. Finally, the quantitative ability of solid-state NMR was compared to both traditional and synchrotron X-ray diffraction techniques for the conversion of chlorpropamide in formulations was studied. The sensitivity of solid-state NMR was comparable to that of synchrotron X-ray diffraction, and about an order of magnitude more sensitive than a traditional powder X-ray diffractometer. However, the acquisition times for the solid-state NMR spectra were close to 48 hours, compared to minutes for the diffraction techniques. Finally, an example of the comparison of powder X-ray diffraction and solid-state NMR spectroscopy for examining particle size in crystalline drugs will be presented.

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